

Key Words:

Hanford River Protection Project
Waste Feed Evaporation
Pretreatment
Envelope C

Retention:

Permanent

Key WTP R&T References:

Test Specification
24590-PTF-TSP-RT-02-004, Rev. 0
Test Plan **WSRC-RP-2002-00158**
SRT-RPP-2001-00243, Rev. 0
Test Exception 24590-WTP-TEF-RT-02-045
R&T Focus Area **Pretreatment**
Test Scoping Statement(s) **S-68 & S-113**

**EVAPORATION OF HANFORD TANK SAMPLE
AN-107 MIXED WITH RECYCLES (U)**

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DECEMBER 2003

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This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

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LIST OF ACRONYMS

BSE	Backscattered Electron
mg/L	milligram per liter
mCi/L	millicurie per liter
ntu	nephelometric turbidity unit
orp	oxidation reduction potential
RPP	River Protection Project
rsd	relative standard deviation
SCF	Shielded cells facility
SE	secondary electron
SRS	Savannah River Site
SRTC	Savannah River Technology Center
WTP	Hanford Tank Waste Treatment and Immobilization Plant

ABSTRACT

A proof-of-technology demonstration for the Hanford River Protection Project (RPP) Hanford Tank Waste Treatment and Immobilization Plant (WTP) was performed by the Savannah River Technology Center (SRTC) at the Savannah River Site (SRS). As part of this demonstration, a sample of as-received Tank AN-107 waste was mixed with surrogate recycle and then evaporated to concentrate the mixture. A second test was conducted in which surrogate recycle was initially concentrated, then mixed with as-received AN-107 waste. Both of these tests were the first studies conducted that investigated the potential impact of secondary-waste recycle streams on the evaporation process using actual radioactive waste feed. The first test using recycle to dilute the AN-107 feed represented a scenario in which waste feed is received below the specific gravity of 1.22 or 5 molar sodium. The second test using concentrated recycle added to 8.6M Na⁺ AN-107 represents a scenario in which waste feed is received that exceeds the stated requirements.

Various analytical measurements on the two product solutions from these two scenarios indicate that either product will be sufficient as feed material to the Sr/TRU precipitation and filtration pretreatment step for Envelope C supernatants. Neither product solution contained excessive amounts of insoluble solids and no troublesome solids such as alumino-silicates or gels were detected. The product solutions appeared similar to previous solutions of mixed recycle blended with actual AN-107 examined in stream blending testing at SRTC. No observations of de gassing or extreme temperature changes were found on mixing of the recycle and AN-107 samples for these evaporation tests. Both the evaporation of AN-107 mixed with recycle and the evaporation of recycle alone did not require the use of antifoam. No significant foaming was observed in either test. Evaporation was carried out under prototypical WTP evaporation conditions of approximately 27 inches mercury vacuum at nominally 50 °C. Flux rates were about 2 mL/min in the first test evaporation of recycle mixed with AN-107 and about 5 mL/min in the evaporation of recycle only. These fluxes approach about 10% of design basis flux.

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1.0 SUMMARY OF TESTING

The tasks addressed in this report were originally described in scoping statement S-68 for “Demonstrate contract requirement to validate the LAW Feed Evaporator design basis calculations, flow sheet and material balance and operating requirements by: Performing integrated small-scale radioactive evaporation using Hanford tank sample AN107 with radioactive recycle or inactive simulants (Integrated C) to evaluate recommended antifoam agents and determine LAW solubility.”¹ After the S-68 Test Specification² was issued by WTP personnel, it was decided³ to capture the S-68 scope under the Task Plan⁴ guidance of similar work being performed under S-113 for “Recycle Stream Blending for High and Low Level Waste.”⁵ While previous S-113 testing has examined the mixing of surrogate recycle with either actual radioactive AW-101 or with actual radioactive AN-107 (See Section 3.1.2, Radioactive Testing, Phase 3B⁶), the tasks described in this current report examine the influence of evaporation on the mixing of recycles with actual waste.⁷

One task described in this report investigates the potential impact of secondary-waste recycle stream influence on the waste feed evaporation process. In a related task the recycle was evaporated separately and then subsequently used to dilute the AN-107 feed solution to a target nominal 6M Na concentration. Both of these product solutions of AN-107 mixed with recycle were analyzed for cations, limited anions, and limited radionuclides. The product solutions were also tested for certain physical properties for comparison to similar data obtained for the as-received AN-107 tank sample. Solids from both AN-107 mixed with recycle solutions were separated, washed, and characterized by x-ray diffraction and scanning electron microscopy. The product solutions were also measured for pH, turbidity and redox potential for comparison to similar testing that involved mixing recycle solutions (without concentration) with AN-107 feed.⁶

1.1 OBJECTIVES

The goals of this task were to determine what effect plant recycles have on the evaporation process. Data obtained from the evaporation process samples along with modeling of the evaporation process provide solubility data for the evaporator concentrate solutions. Another objective of this task was to investigate the effectiveness of an antifoam reagent on actual waste feed blended with recycle. This latter goal was contingent upon actual observation of any foaming during the evaporation of AN-107 waste mixed with recycle.

1.2 CONDUCT OF TESTING

Testing included two main tasks that involved two different scenarios. Scenario #1 was to dilute the as-received AN-107 feed with a simulant of mixed recycle solution in the flowsheet volumetric ratio of waste feed to recycle, followed by a bench-scale evaporation test. This first scenario was designed to demonstrate the case of as-received waste delivered to WTP at the contract-minimum sodium molarity of 4. The evaporation process of the diluted sample targeted approximately 6M Na concentration as the concentration endpoint. This is the reference concentration for Envelope C feed to the Sr/TRU precipitation and filtration process.

Scenario #2 involved diluting the as-received AN-107 sample with a surrogate of concentrated plant recycle solutions. The surrogate plant recycle solution was evaporated before mixing, in a manner similar to previous recycle evaporation testing conducted at SRTC.⁸ The target final concentration of the AN-107 mixed with concentrated recycle for this test was also the approximately 6M Na concentration representative of the Envelope C feed to the Sr/TRU precipitation and filtration process.

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

Results from these evaporation, mixing and product characterization tasks met the task objectives by demonstrating the following:

- Evaporations of both waste feed mixed with recycle and evaporation of neat recycle were accomplished without the formation of any potentially troublesome solids.
- Data for solids measured in the product solutions as well as modeling results provided solubility data for the evaporator concentrate solutions.
- Evaporation of both waste feed mixed with recycle and evaporation of neat recycle was accomplished without the need for antifoam additions, indicating that under these condensate flux conditions tested in the range of 2 mL/min to 5 mL/min, the foaming of these systems was not significant.

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO M0SRLE60. SRTC has provided matrices to WTP demonstrating compliance of the SRTC QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRTC QA program with RW-0333P, Revision 10, NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices. A Task Plan describing the QA requirements applicable to the tasks described in this report has previously been issued.⁴

1.5 ISSUES

- Application of centrifuge, decant, and drying methods to determine the amount of undissolved solids in the product solutions from these tests were unsuccessful. This is likely due to the fact that these product solutions have relatively low amounts of undissolved solids and the AN-107 waste feed mixed with recycle solution matrix is a very dark, opaque liquid.
- These evaporation tests did not raise issues for the design and operation of the RPP-WTP. However experimental vapor flux was only approached approximately 10% of design basis and the evaporation testing did not concentrate the AN-107 plus recycles to the endpoint target of 6M Na⁺. The actual endpoint was determined to be 4.6M Na⁺. Because higher flux rates were not tested, a foaming issue during evaporation of this material in the RPP-WTP cannot be completely ruled out. Because the target 6M endpoint was not achieved in the concentration, the worst case scenario that would generate more solids and have a higher potential for developing foam was not completed, which would indicate at what concentration foaminess or excessive bumping becomes a problem.

2.0 CD-ROM ENCLOSURES

A CD-ROM, entitled S68: LAW Feed Evaporator, containing a video of the recycle-only evaporation testing is attached.

The recommended minimum computer system is as follows:

- Pentium II running at 233 MHz
- 32 MB ram
- Windows 95 or later.

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3.0 DISCUSSION

The goals of this task were to determine what effect plant recycles have on the evaporation process using surrogate recycle streams and actual radioactive Hanford tank samples. The first task involved diluting the as-received AN-107 sample with recycle, followed by evaporation of the product. The characterization of as-received AN-107 has been reported previously.⁹ A summary table of the methods used in the AN-107 analysis is shown in Table 1. Summary characterization data of the AN-107 filtrate and solids composition are presented in Table 2 through Table 5. Radionuclide data shown in Table 2 indicate that the as-received AN-107 sample is highly radioactive with total specific activity level approaching 1 Curie/L. This radioactivity results mostly from the Cs-137 (0.335 Ci/L gamma) and the Sr-90 (0.084 Ci/L from Sr-90 and Y-90 and ~ 0.335 Ci/L from Ba-137). The recycle stream used in this work has also been described previously. Summary tables of the recycle stream filtrate and solids composition are presented in Table 6 and Table 7. It should be noted that even though both the AN-107 as-received sample and the recycle stream used in this work both contained insoluble solids, the amount of solids present in these samples was relatively low. The amount of insoluble solids in the AN-107 as-received sample was measured in the range of 0.2 to 0.9 wt%.⁹ The amount of insoluble solids in the recycle stream was measured to be 0.12 wt%.⁶

Section 3.1 describes the experimental setup and details of the first scenario tested involving dilution of the as-received AN-107 sample, followed by evaporation. The second scenario was to evaporate recycle prior to mixing with as-received AN-107 waste. Section 3.2 describes the experimental details of this task. These product samples were measured for pH, turbidity, and redox potential for comparison to previous recycle stream blending work reported by Barnes.⁶ Both of the product solutions from these two tasks were analyzed for metals, anions, and certain radionuclides. The condensate from the first task of evaporation was also analyzed. These data are presented in Section 3.3.

The product solutions from the two tasks were also analyzed for certain physical properties to compare with similar data reported for the original AN-107 as-received sample.⁹ These physical properties data, determined from centrifuging product solutions and separating the supernatant liquid remaining above the solids, are presented in Section 3.4. Solids from both product solutions from the two tasks were also analyzed after filtration and rinsing, for crystalline identification and microscopy. These data, determined from additional centrifuging followed by solids separation using filtration, are presented in Section 3.5.

Table 1. Summary Table of Methods Used in AN-107 Analysis

Analytical Method	Abbreviation in Tables	ADS Procedure No.
Ion Chromatography	IC	ADS-2306
Ammonia Purge and Trap	PT	ADS-2306
Titration	T	ADS-1206 Rev. 1
ICP-AES	IE	ADS-1564
ICP-MS	IM	ADS-1543
AA	AA	ADS-1554 Rev. 3
Calc. By Difference	Diff	NA
Acidification	A	ADS-1206 Rev. 1
Gamma Spec.	GS	ADS-2420
Separation/Gamma Spec.	SG	ADS-2420
Separation/Alpha Spec.	SA	ADS-2453 ADS-2449
Separation/Liquid Scintillation	SL	ADS-2447 ADS-2444 ADS-2407
Alpha Counting	AC	ADS-2402
HPLC	HL	ADS-2660
GC-MS	GM	ADS-2661

Table 2. As-Received 241-AN-107 Supernate Analyses

Analyte	Average (mg/L)	Analyte	Average (mg/L)	Radionuclides	Average (mCi/L)
Ag (IE)	2	¹³³ Cs (IM)	11	Gross Beta* (SL)	592
Al (IE)	306	¹³⁵ Cs (IM)	2	²³⁸ Pu (SA)	1.26E-02
B (IE)	13	¹³⁷ Cs (IM)	5	^{239/240} Pu (SA)	4.99E-02
Ba (IE)	10	La (IM)	35	²⁴¹ Pu (SA)	8.66E-02
Be (IE)	<0.199	W (IM)	165	¹³⁷ Cs (GS)	335
Ca (IE)	566	HEDTA (HL)	1123	⁹⁰ Sr (SL)	84.2
Cd (IE)	72	EDTA (HL)	3627	⁹⁹ Tc (total) (SL)	1.04E-01
Cr (IE)	191	IDA (GM)	4054	⁵⁹ Ni (SL)	2.47E-02
Cu (IE)	38	TIC (A)	26600	⁶³ Ni (SL)	1.66
Fe (IE)	1865	TOC (Diff)	33300	⁶⁰ Co (GS)	8.16E-02
Li (IE)	<8.55	Total Base (T)	1.32	¹²⁶ Sb/ ¹²⁰ Sn (GS)	7.28E-04
Mg ~ (IE)	0.879	Free OH (T)	<0.02	¹²⁵ Sb (GS)	2.42E-03
Mn (IE)	664	F (IC)	3319	¹⁵² Eu (GS)	7.37E-03
Na (IE)	207,793	CHO ₂ (IC)	11112	¹⁵⁴ Eu (GS)	4.33E-01
Ni (IE)	581	Cl (IC)	1392	¹⁵⁵ Eu (GS)	2.52E-01
P (IE)	560	NO ₂ (IC)	60760	²³¹ Pa (GS)	<1.25E-02
Pb (IE)	460	NO ₃ (IC)	224869	¹²⁹ I (SG)	1.49E-04
S (IE)	3763	PO ₄ (IC)	946	¹⁴ C* (SL)	6.97E-04
Si (IE)	15	SO ₄ (IC)	8480	⁹⁹ Tc Pertech. (SL)	<5.11E-02
Sr (IE)	121	C ₂ O ₄ (IC)	663	⁷⁹ Se (SL)	<4.76E-03
Ti (IE)	<1.53	NH ₃ (PT)	415	²⁴¹ Am (SG)	8.06E-01
U (IE)	169	Citrate (IC)	12193	²⁴³ Am (SA)	1.30E-03
V (IE)	0.785	Glycolate (IC)	24801	²⁴⁴ Cm (SA)	3.70E-02
Zn (IE)	29	Formate (IC)	18515	²⁴² Cm (SA)	2.98E-03
As (AA)	<0.937	Acetate (IC)	1440		
K (AA)	1556	Carbonate (IC)	17824		
Na (AA)	188,321				
Se (AA)	<0.937				
Hg (AA)	<2.29				
Th (IM)	18				
²³⁴ U (IM)	<0.0327				
²³⁵ U (IM)	0.78				
²³⁶ U ~ (IM)	0.04				
²³⁷ Np (IM)	0.09				
²³⁸ U (IM)	125				
²³⁹ Pu (IM)	0.71				
²⁴⁰ Pu (IM)	0.05				
Total U (IM)	126				
Rb (IM)	8				

* ~5% contribution from gamma

Table 3. As-Received 241-AN-107 Centrifuged Solids Microwave-Dissolved

Analyte	Average (mg/kg)	Analyte	Average (mg/kg)	Analyte	Average (mg/kg)	Radionuclides	Average (mCi/kg)
Ag (IE)	<11.63	As (AA)	<19.6	¹³³ Cs (IM)	121	⁶⁰ Co (GS)	4.74E-02
Al (IE)	264	K (AA)	995	¹³⁵ Cs (IM)	5	¹²⁶ Sb/ ¹²⁶ Sn (GS)	<1.38E-03
Ba (IE)	<27.63	Na (AA)	154667	¹³⁷ Cs (IM)	10	¹²⁵ Sb (GS)	<4.01E-03
Be (IE)	<5.61	Se (AA)	<19.6	Ba (IM)	13	¹⁵² Eu (GS)	<1.17E-02
Ca (IE)	649	Hg (AA)	<48.0	Ce (IM)	197	¹⁵⁴ Eu (GS)	2.20E-01
Cd (IE)	40	Th (IM)	29	Pr (IM)	<300	¹⁵⁵ Eu (GS)	1.22E-01
Cr (IE)	117	²³³ U (IM)	<1.39	Ta (IM)	12	²³¹ Pa (GS)	<3.47E-02
Cu (IE)	25	²³⁴ U (IM)	<1.39	W (IM)	124	⁷⁹ Se (SL)	<1.67E-03
Fe (IE)	1263	²³⁵ U (IM)	<1.39	Pt (IM)	<3	²⁴¹ Am (SG)	5.59E-01
La (IE)	34	²³⁶ U (IM)	<1.39	Tl (IM)	<5	²⁴³ Am (SA)	<3.33E-03
Li (IE)	<62.57	²³⁷ Np (IM)	<1.39	Rh (IM)	36	²⁴⁴ Cm (SA)	3.84E-02
Mg (IE)	<7.71	²³⁸ U (IM)	88	Pu (IM)	<0.14	²⁴² Cm (SA)	3.03E-03
Mn (IE)	407	²³⁹ Pu (IM)	<1.39	Y (IM)	55	⁵⁹ Ni (SL)	2.90E-03
Na (IE)	155000	²⁴⁰ Pu (IM)	<1.39	Bi (IM)	<0.89	⁶³ Ni (SL)	8.41E-01
Ni (IE)	385	Total U (IM)	88	Nd (IM)	405	Gross Alpha (AC)	1.10E+00
P (IE)	182	V (IM)	170			³ H (SL)	<1.30E-02
Pb (IE)	242	Co (IM)	118			¹³⁷ Cs (GS)	1.94E+02
S (IE)	3997	As (IM)	<20			⁹⁰ Sr (SL)	7.96E+01
Si (IE)	442	Se (IM)	<300			^{239/240} Pu (SA)	8.48E-03
Sr (IE)	141	Rb (IM)	48			²³⁸ Pu (SA)	8.95E-03
Ti (IE)	<11	Ru (IM)	48			²⁴¹ Pu (SA)	3.48E-02
U (IE)	<330	Mo (IM)	24			¹⁴ C (SL)	3.25E-04
V (IE)	14	Pd (IM)	13				
Zn (IE)	<5	Sb (IM)	<12				
Zr (IE)	47	Te (IM)	<6				

Table 4. As-Received 241-An-107 Centrifuged Solids Aqua-Regia Dissolution

Analyte	Average (mg/kg)	Analyte	Average (mg/kg)	Analyte	Average (mg/kg)	Radio-nuclides	Average (mCi/kg)
Ag (IE)	<1.40	As (AA)	<5.58	Ba (IM)	1.6	¹³⁷ Cs (GS)	5.60E+01
Al (IE)	121	K (AA)	337	Ce (IM)	7.8	³ H* (SL)	<6.15E-03
B (IE)	<4.90	Na (AA)	128,667	Pr (IM)	4.5	Gross Alpha (AC)	5.45E-01
Ba (IE)	<3.32	Se (AA)	<5.58	Ta (IM)	<0.03	⁹⁰ Sr (SL)	2.32E+01
Be* (IE)	0.19	Hg (AA)	<13.67	W (IM)	25.6	²⁴¹ Am (SG)	1.28E-01
Ca (IE)	208	Th (IM)	3.3	Pt (IM)	<0.3	²⁴³ Am (SA)	5.60E-04
Cd (IE)	11	²³³ U (IM)	0.014	Tl (IM)	<0.2	²⁴⁴ Cm (SA)	9.74E-02
Cr (IE)	33	²³⁴ U (IM)	<0.013	Rh (IM)	1.7	²⁴² Cm (SA)	6.00E-04
Cu (IE)	9	²³⁵ U (IM)	0.29	Pu (IM)	0.2	²³⁸ Pu (SA)	2.46E-02
Fe (IE)	450	²³⁶ U* (IM)	0.03	Y (IM)	2.1	^{239/240} Pu (SA)	8.23E-03
Li (IE)	<7.52	²³⁷ Np (IM)	0.04	Bi (IM)	<0.3	²⁴¹ Pu (SA)	3.63E-02
Mg ~ (IE)	14	²³⁸ U (IM)	46.7	Nd (IM)	21.1	¹²⁹ I (SG)	<6.67E-04
Mn (IE)	119	Total U (IM)	47.0				
Ni (IE)	102	V (IM)	11.8				
P (IE)	97	Co (IM)	1.3				
Pb (IE)	69	As (IM)	0.9				
S (IE)	536	Se (IM)	<0.12				
Si ~ (IE)	4	Rb (IM)	2.5				
Sr (IE)	44	Ru (IM)	5.7				
Ti (IE)	<1.35	Mo (IM)	4.9				
U~ (IE)	80	Pd (IM)	1.7				
V (IE)	<0.350	Sb (IM)	0.1				
Zn (IE)	6	Te (IM)	<0.08				
Zr* (IE)	12	¹³³ Cs (IM)	2.0				
		¹³⁵ Cs (IM)	0.4				
		¹³⁷ Cs (IM)	1.2				

Table 5. Composition of the Water Leach Digested As-Received 241-AN-107 Centrifuged Solids

Analyte	Average (mg/kg)
TIC (A)	18933
TOC (Diff)	29600
F (IC)	1803
CHO ₂ (IC)	4801
Cl (IC)	<2662
NO ₂ (IC)	30532
NO ₃ (IC)	113775
PO ₄ (IC)	<13308
SO ₄ (IC)	4006
C ₂ O ₄ (IC)	3846
Br (IC)	<13308

Table 6. Composition of Filtrate Obtained from Recycle Sample

Analyte	Method	Units	Recycle Solution	Analyte	Method	Units	Recycle Solution
Na	AA	Molar	0.81	Ni	ICP-ES	mg/L	< 7
Na	ICP-ES	Molar	0.79	P	ICP-ES	mg/L	67
OH ⁻	Titration	Molar	0.293	Pb	ICP-ES	mg/L	< 16
total base	Titration	Molar	0.225	S	ICP-ES	mg/L	133
CO ₃ ²⁻	Titration	Molar	< 1.0	Si	ICP-ES	mg/L	< 9
NO ₃ ⁻	IC	Molar	0.337	Sn	ICP-ES	mg/L	< 23
NO ₂ ⁻	IC	Molar	0.111	Sr	ICP-ES	mg/L	< 4
SO ₄ ²⁻	IC	Molar	0.00424	Ti	ICP-ES	mg/L	< 4
Cl ⁻	IC	Molar	0.0101	U	ICP-ES	mg/L	< 116
F ⁻	IC	Molar	0.011	Zn	ICP-ES	mg/L	< 2
HCO ₂ ⁻	IC	Molar	0.011	Zr	ICP-ES	mg/L	< 12
C ₂ O ₄ ²⁻	IC	Molar	0.00174				
PO ₄ ³⁻	IC	Molar	0.0043				
Ag	ICP-ES	mg/L	< 4				
Al	ICP-ES	mg/L	1437				
B	ICP-ES	mg/L	63				
Ba	ICP-ES	mg/L	< 10				
Ca	ICP-ES	mg/L	< 12				
Cd	ICP-ES	mg/L	< 2				
Ce	ICP-ES	mg/L	< 14				
Cr	ICP-ES	mg/L	4				
Cu	ICP-ES	mg/L	7				
Fe	ICP-ES	mg/L	< 2				
Hg	AA	mg/L	22				
K	AA	mg/L	89				
K	ICP-ES	mg/L	< 481				
La	ICP-ES	mg/L	< 4				
Li	ICP-ES	mg/L	< 22				
Mg	ICP-ES	mg/L	< 3				
Mn	ICP-ES	mg/L	< 0.4				
Mo	ICP-ES	mg/L	< 27				

Table 7. Chemical Composition of Solids Obtained from Recycle Solution

Analyte	Method	Units	Recycle Solution
Ag	ICP-ES	ug/g	< 301
Al	ICP-ES	ug/g	63590
B	ICP-ES	ug/g	502
Ba	ICP-ES	ug/g	138
Ca	ICP-ES	ug/g	2501
Cd	ICP-ES	ug/g	13823
Ce	ICP-ES	ug/g	27852
Cr	ICP-ES	ug/g	892
Cu	ICP-ES	ug/g	< 50
Fe	ICP-ES	ug/g	62648
La	ICP-ES	ug/g	< 702
Li	ICP-ES	ug/g	6588
Mg	ICP-ES	ug/g	1156
Mn	ICP-ES	ug/g	2518
Mo	ICP-ES	ug/g	< 100
Na	ICP-ES	ug/g	53185
Ni	ICP-ES	ug/g	7119
P	ICP-ES	ug/g	< 682
Pb	ICP-ES	ug/g	844
Si	ICP-ES	ug/g	6408
Sn	ICP-ES	ug/g	464
Sr	ICP-ES	ug/g	6803
Ti	ICP-ES	ug/g	< 140
U	ICP-ES	ug/g	NA*
V	ICP-ES	ug/g	2369
Zn	ICP-ES	ug/g	5354

*NA = not present or measured in non-radioactive solution.

3.1 EVAPORATION OF AN-107 MIXED WITH RECYCLE

The AN-107 feed sample was mixed with recycle solution and a Hanford process water stream. This process water from Hanford was trimmed with NaOH and NaNO₂ to target 0.01M OH⁻ and 0.011 M NO₂⁻ concentrations. The ratio of streams for this test is given as:

- 1) 358 mL of recycle solution (0.79M Na⁺)
- 2) 116.7 mL of Hanford process water (with 0.01M NaOH and 0.011M NaNO₂ added)
- 3) 100 mL of as-received AN-107 (8.6 +/- 0.6 M Na⁺)

Figure 1 shows the actual samples before they were mixed as they appeared in the remote Shielded Cells Facility (SCF) at SRTC. The combination of the above streams resulted in 575 mL of evaporator feed that was approximately 2M Na⁺. Using the uncertainty in the as-received AN-107 sodium concentration of about 7 wt%, this evaporator feed material blend was in the range of 1.86 to 2.14 M Na⁺. The target for evaporation was to concentrate the 575 mL of evaporator feed by 3X to give approximately 192 mL of concentrate at 6M Na⁺. Assuming the 7 wt% uncertainty in the AN-107 sodium concentration, the expected sodium molarity in this final concentrate was in the range of 5.58 – 6.42M Na⁺.

Evaporation was performed in a low temperature vacuum evaporator that was mixed and heated by a heater/stirrer plate located below the evaporator pot. Figure 2 shows the experimental rig as it was located inside of the remote SCF at SRTC. Preliminary mockup testing with the evaporator indicated obtainable condensate flux rates of about 2 mL/min. Figure 3 is a close-up view of Figure 2. Figure 4 shows the experimental conditions towards the latter part of testing and Figure 5 shows the final concentrate and condensate samples collected in poly bottles.

Evaporation testing used the entire 575 mL blend sample as a single batch in the evaporator. Details of the evaporation testing that lasted from one afternoon, followed by shutdown and restart the next morning, are given in Table 8. Condensate collection volumes divided by evaporation times for both the first afternoon segment and the following morning segment indicate a condensate flux rate for this system of approximately 2 mL/min.

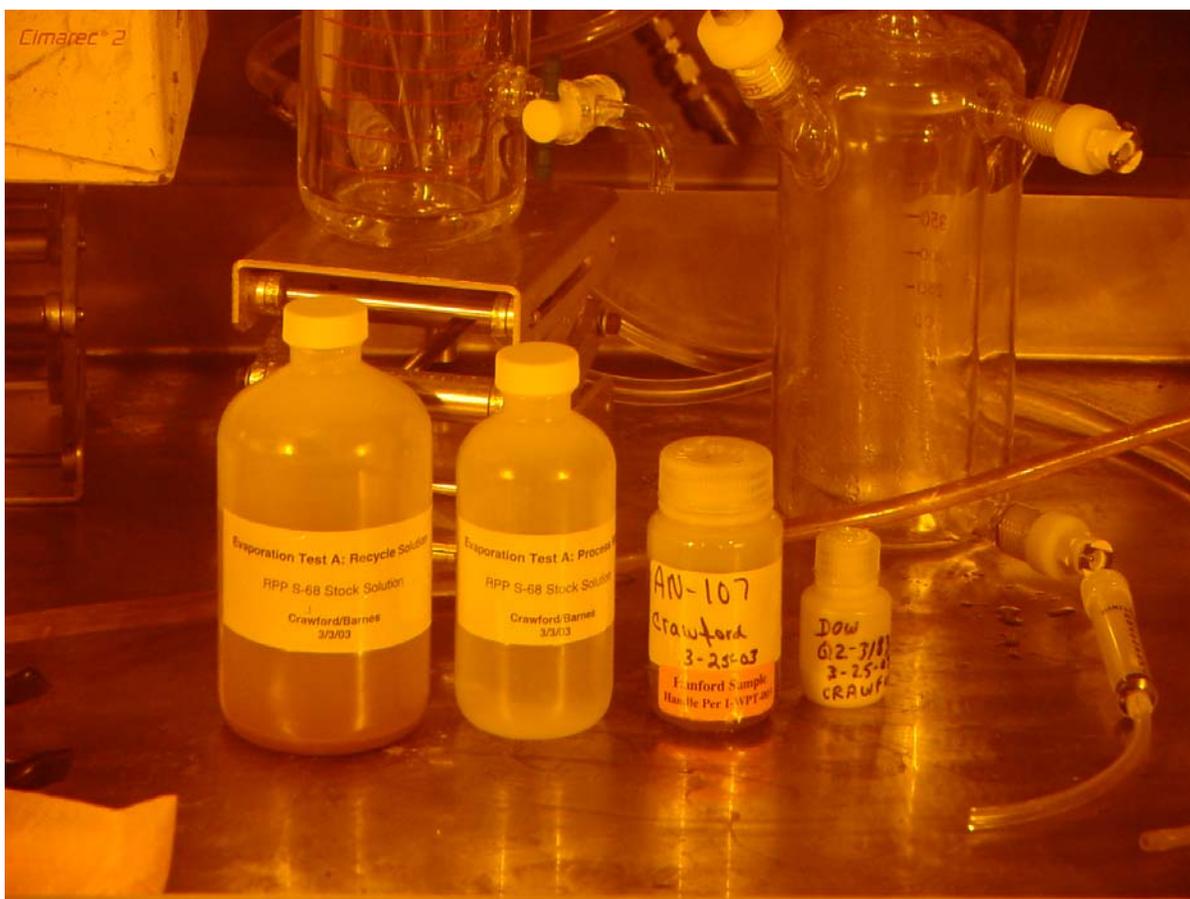


Figure 1. Evaporation Samples

Left sample is 358 mL of recycle solution (0.79M Na^+).

Second from left sample is 116.7 mL of Hanford process water (with 0.01M NaOH and 0.011M NaNO_2 added).

Third from left sample is 100 mL of as-received AN-107 ($8.6 \pm 0.6 \text{M Na}^+$).

Far right sample is antifoam that was not required in evaporation testing.

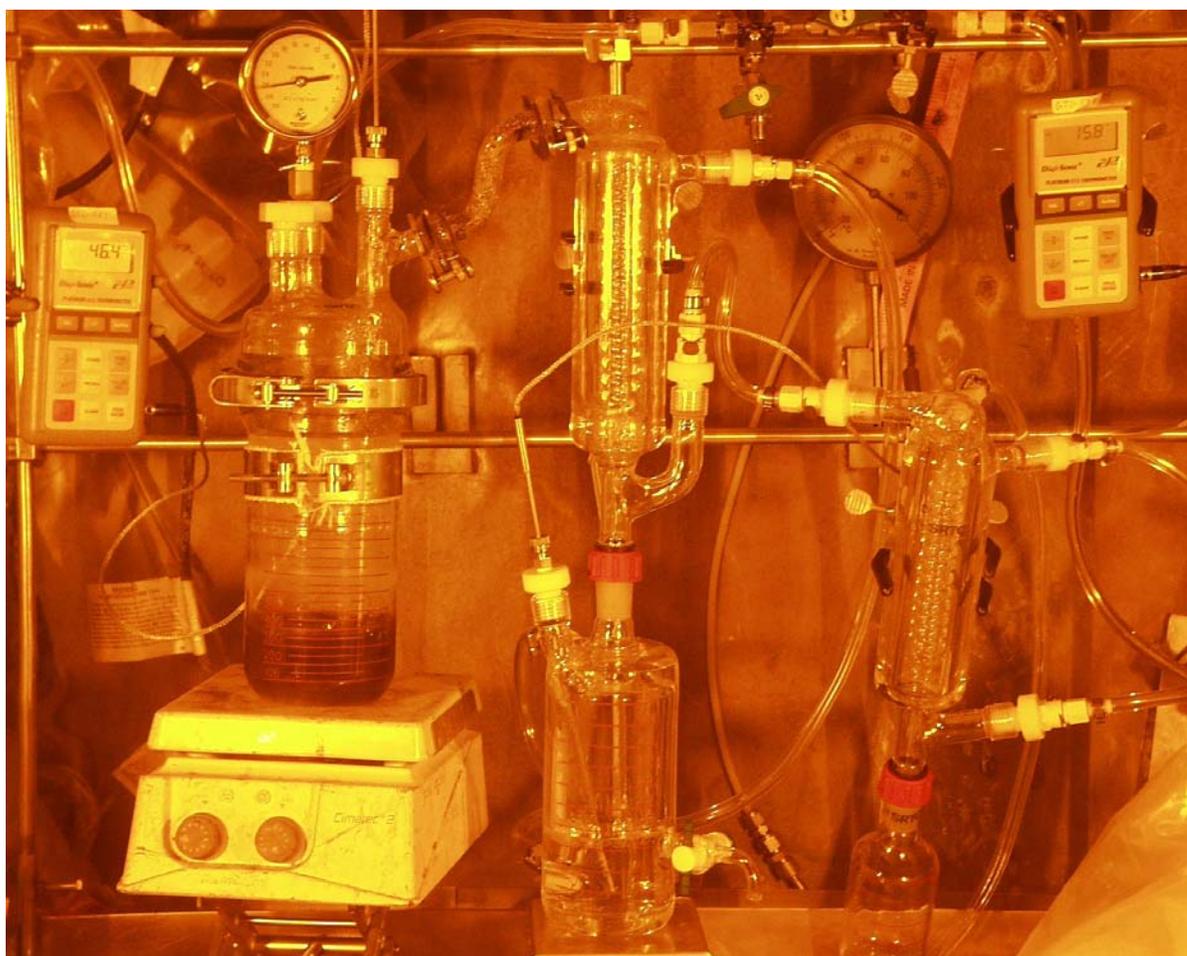


Figure 2. Evaporation Experimental Setup

This photo was taken in the 16:15 to 16:20 timeframe of testing on 3/26/03 with approximately 400 mL of concentrate in the evaporator pot (left with dark liquid) and approximately 160 mL of condensate in the condensate collection pot (bottom-middle). See Table 8.

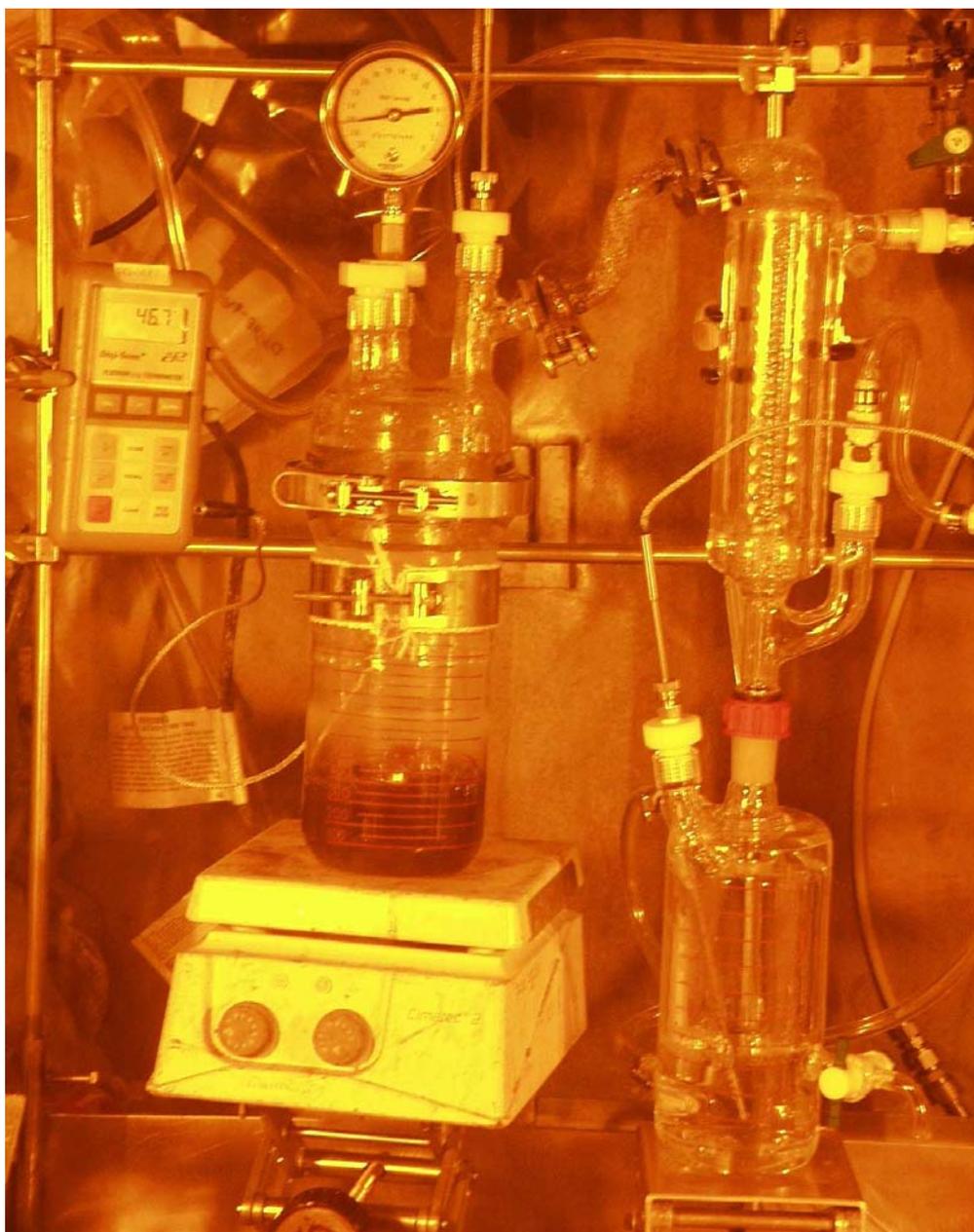


Figure 3. Evaporation Experimental Setup – Close-up

This is a close-up view of Figure 2 taken in the 16:15 to 16:20 timeframe of testing on 3/26/03. – See Table 8. Note slight boiling of concentrate.

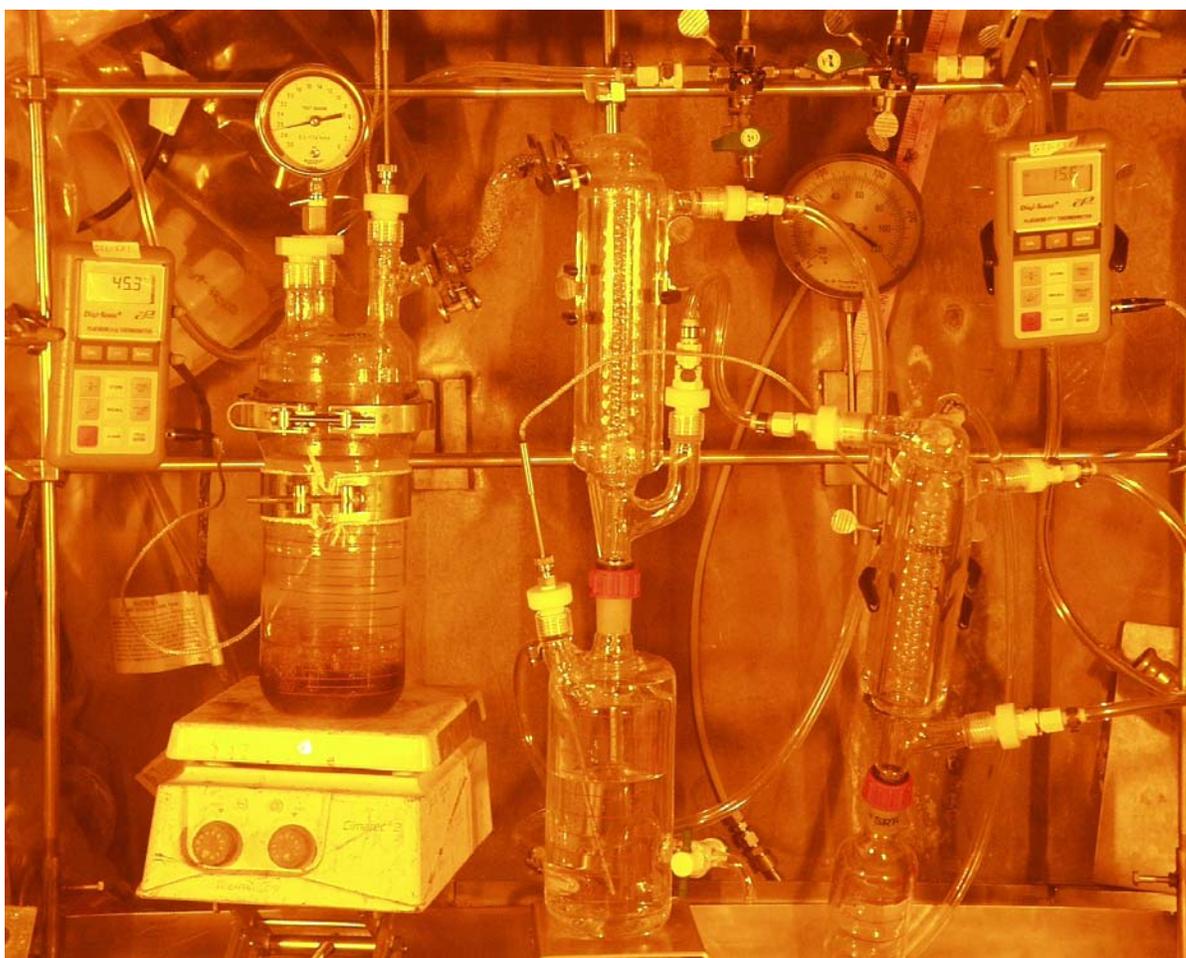


Figure 4. Evaporation Experimental Setup

This photo was taken in the 10:45 to 11:00 timeframe of testing on 3/27/03 with approximately 300 mL of concentrate in the evaporator pot (left with dark liquid) and approximately 260 mL of condensate in the condensate collection pot (bottom-middle). Note that approximately 100 mL of condensate from the original evaporator feed has been removed or boiled off relative to Figure 2 and Figure 3 photos. – See Table 8.

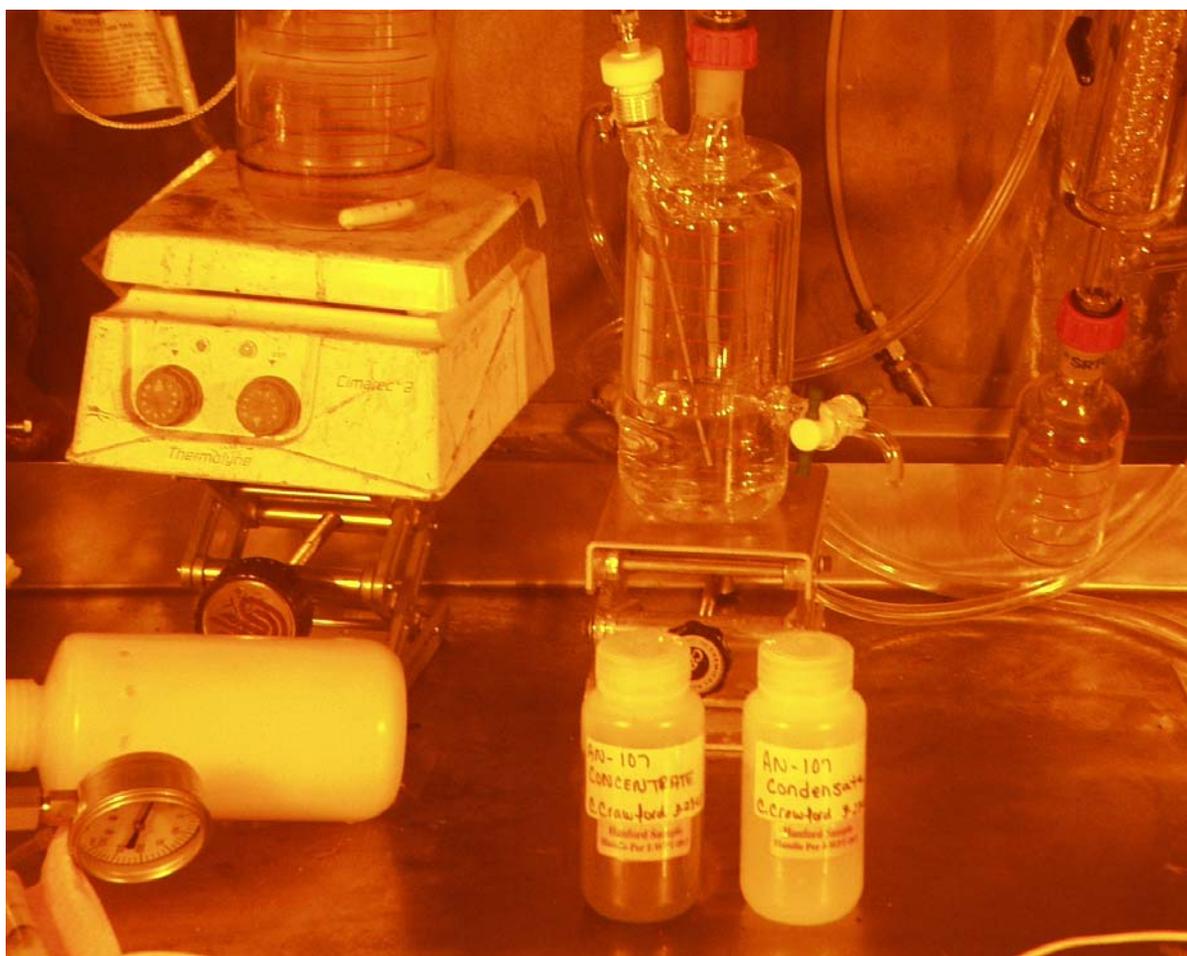


Figure 5. Evaporation Experimental Setup

This photo was taken after collection of the final concentrate (lower left-middle 250-mL poly bottle) and a portion of the final condensate (lower right-middle 250-mL poly bottle). Approximately 150 mL of the final condensate remains in the glass condensate collection pot in upper middle of photograph. Note residue ring at the approximately 200-mL mark on the emptied concentrate pot (upper left), indicative of the approximate final concentrate level in the pot.

Table 8. Details of Evaporation

Time	Pot Temp (°C)	Condensate Temperature (°C)	Vacuum (inches Hg)	Chiller Temp (°C)	Hot Plate Setting	Concentrate Pot Volume (mL)	Condensate Pot Volume (mL)
3/26/03 13:00	24.9	18.9	28	21.1	1	560	0
13:15	29.4	15.4	28	14.9	1		
13:30	33.6	15.7	27.5	14.9	1		
14:00	33.2	15.8	27.6	15	2		
14:15	39	16.3	27.6	15.5	2		
Noticed boiling start at approximately 41.8 °C and condensate drops initiated.							
14:30	42	17.8	27.6	18	2		
14:45	41.6	18.4	27.6	18	2		
15:00	41.8	15.8	27.6	15.2	2		
15:15	41.7	16.4	27.6	14.9	3		
15:30	41.8	17.1	27.6	16.2	3	500	60
15:45	42	16.7	26.8	14.9	4		
16:00	42.2	16	26.8	15.7	4	450	110
16:15	42.2	16.4	26.8	15.7	4	425	135
16:20	51.4	16.3	25.2	15.3	4	400	160
16:30	59	16.4	25.2	14.8	4		
16:35	59	16.8	25.2	15	4		
System heat turned off and vacuum discontinued. Evaporation restarted on 3/27/03							
09:30	24.2	16.9	26.2	15.1	3		
10:00	46.2	15.3	26.4	15	3		
10:30	46.4	16.1	26.2	15	3		
10:45	46.6	15.7	26.2	15.3	3		
11:00	44.3	15.6	26.8	14.7	5	350	210
11:30	44.7	15.7	26.8	15	5	300	260
11:45	44.7	15.7	26.8	15	5	275	285
12:00	45.1	15.6	26.8	15	5	230	330
12:15	46	16	26.8	15.9	5	200	360
System heat turned off and vacuum discontinued.							

3.2 BLENDING OF CONCENTRATED RECYCLE WITH AS-RECEIVED AN-107

A sample of the recycle was concentrated from 0.79M Na⁺ to a target sodium molarity of 3.07M, so that when mixed with the AN-107 waste, the resulting sodium concentration would be approximately 6M. This is the reference target for Envelope C material feed to the Sr/TRU precipitation and filtration process. The ratio of streams for this test is given as:

- 1) 100 mL of as-received AN-107 (8.6 +/- 0.6 M Na⁺)
- 2) 89.9 mL of concentrated recycle-only (3.01 M Na⁺)

The combination of the above streams resulted in 189.9 mL of mixed product targeted at 6M Na⁺. Using the uncertainty in the as-received AN-107 sodium concentration of about 7%, this AN-107 supernatant mixed with concentrated recycle was estimated to be in the range of 5.58 – 6.42M Na⁺.

Table 9 and Figure 6 show the data used in determining the concentration endpoint of the recycle solution in the recycle-only evaporation. These operating region plots have been explained in detail by Stone, et al., Waste Feed Evaporation Report.⁸ The intersection of the solid line and the lower dashed line indicates the operating point or target concentration of the recycle. These data are plotted with a starting recycle-to-waste volume ratio of 3.5 L recycle : 1 L waste. Figure 6 shows that the starting 0.788M recycle stream would have to be concentrated to 3.07M (the intersection of the solid line and the dashed line representing the 8.6M AN-107 waste feed) to be mixed with the 8.6M AN-107 waste. This would result in a final product recycle content of 47 vol%, or 0.9 L in the total volume of 1.9 L. Note that Figure 6 also shows traces for hypothetical 7M and 8M AN-107 feed for the same assumed starting recycle: waste ratio of 3.5L:1L. These data show that as the starting sodium molarity of the feed decreases, the evaporation of the recycle would have to be increased to produce a more concentrated recycle and, accordingly, less of the concentrated recycle would be required.

Table 9. Data Used to Determine Concentrated Recycle Target Sodium Molarity with a Recycle to Waste ratio of 3.5L:1L

Sodium Molarity	Recycle Volume (L)	Total Volume (L)	Recycle Vol %
1.4	1.97	2.97	66.33
1.8	1.58	2.58	61.18
2	1.38	2.38	57.97
2.5	1.10	2.10	52.45
3	0.92	1.92	47.90
3.5	0.79	1.79	44.07
4	0.69	1.69	40.81
4.5	0.61	1.61	38.00
5	0.55	1.55	35.55
3.1	0.90	1.90	47.32

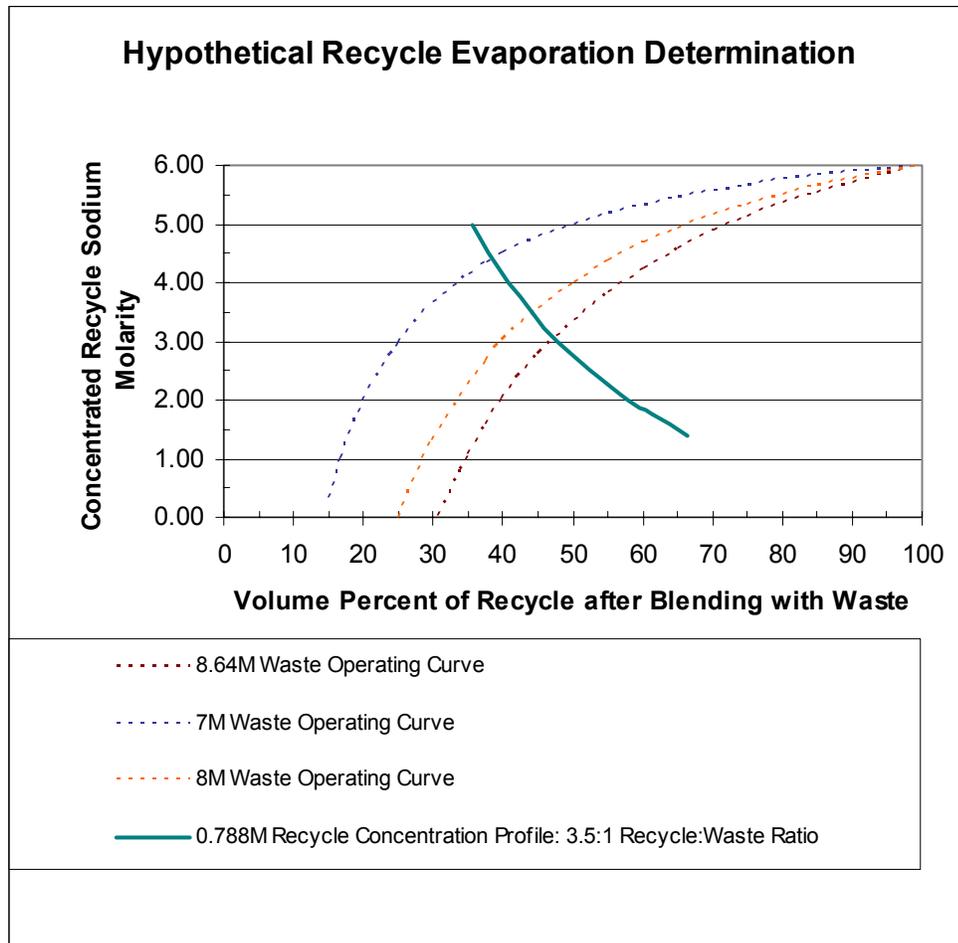


Figure 6. Hypothetical Recycle Evaporation Determination for 8.6M AN-107 and Recycle

Evaporation of recycle-only was performed in a low temperature vacuum evaporator that was mixed and heated by an infrared heater/stirrer hotplate located below the evaporator pot. The experimental setup has been discussed in detail in the Waste Feed Evaporation Report, Appendix B.

Evaporation testing used 800 mL of the 0.788M recycle stream as a single batch added to the evaporator. Details of the evaporation testing that lasted approximately 3 hours on 1/30/03 are given in Table 10. Condensate collection volumes for each 100-mL segment divided by evaporation times indicate a condensate flux rate for this system of approximately 5 mL/min. From the original 800 mL of recycle feed, 205 mL of concentrate was produced and 588.1 mL of condensate was produced. The 205 mL of concentrate was targeted to be 3.07M concentrated recycle to be used in the subsequent blending test with as-received AN-107. Note from the evaporation data that the concentrate sample was actually evaporated to 150 mL, approximately 25% past the target 205 mL, then condensate was used to dilute the 150 mL of concentrate back up to the exact target of 205 mL. This method was used to arrive at a more exact final volume of product concentrate, since trying to target exactly 205 mL of concentrate in the evaporator pot during actual testing is less accurate due to variation in the concentrate level during the boiling process. The slight over-concentration is primarily performed to allow the excess condensate to be used as rinse to ensure complete transfer of solids from the evaporator concentrate pot to a collection bottle.

Table 10. Details of Recycle-Only Evaporation

Time	Pot Temp (°C)	Vacuum (inches Hg)	Chiller Temperature (°C)	Hot Plate Setting	Concentrate Pot Volume (mL)	Condensate Pot Volume (mL)
07:36	-	-	-	8	800	0
07:45	40.95	26.90	15	8	800	0
07:47	43.31	27.05	15	8	<800	*
08:11	45.01	26.85	15	8	700	100
08:31	43.75	27.05	15	8	600	200
08:51	43.86	27.05	15	8	500	300
09:15	43.95	27.05	15	8	400	400
09:36	44.17	26.95	15	8	300	500
09:57	44.73	26.95	15	8	200	600
10:07	45.42	26.95	15	8	150	650
System shutdown – heat off, cool down						

* First notice of condensate collection

Figure 7 through Figure 9 show the before and after pictures of the surrogate recycle samples.



Figure 7. Recycle Solution from Phase 3B Testing⁶ Before Evaporation



Figure 8. Product Concentrate from Recycle Evaporation



Figure 9. Settled Product Concentrate and the Condensate from Recycle Evaporation

Figure 10 and Figure 11 show samples of the pre-mixed and final mixed blend from this Scenario #2 testing. This small-scale beaker test was actually a scoping or qualitative test to determine any significant effects, for instance visual off gassing or gel formation, of blending the concentrated recycle with the as-received AN-107. The product blend did not exhibit any offgassing or gel formation. The product mixture looked much like the original as-received feed, i.e., a dark opaque solution. Similar results were also reported for the analogous testing in which recycle (without pre-concentration) was mixed with as-received AN-107.⁶



Figure 10. Beaker samples of as-received AN-107 (left) and the concentrated recycle (right) before mixing.

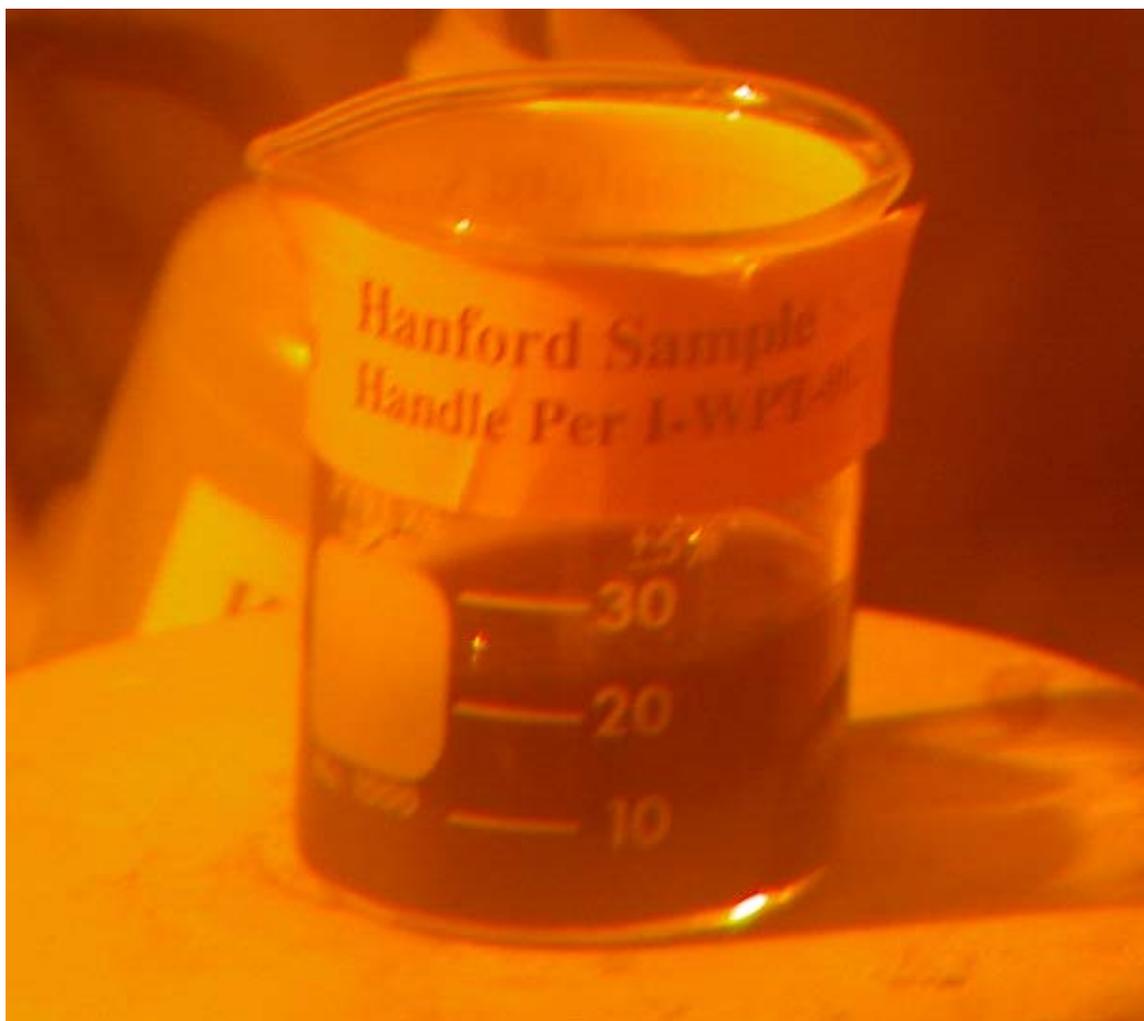


Figure 11. Beaker sample of as-received AN-107 mixed with concentrated recycle

3.3 ANALYSES OF PRODUCT SOLUTIONS

Both product solutions from the first scenario evaporation testing of AN-107 mixed with recycle described in Section 3.1 and for the second scenario mixing of AN-107 with concentrated recycle described in Section 3.2, were analyzed for metals, anions, and select radionuclides. The condensate from the first scenario evaporation testing was also analyzed. Data for these analyses are shown in Table 11 and Table 12. All data shown in Table 11 and Table 12 were generated from duplicate samples. Analytical samples for the concentrated products were prepared by adding 1 mL of well-mixed product solution to 10 mL of deionized water. The condensate was analyzed without dilution. The precision from these duplicate analyses was shown to be good, as the percent relative standard deviations (%RSDs) were typically less than 5 %. Silver and sodium duplicate analyses from the Scenario #1 concentrate were not as precise. Also, the Cu, Na, Sn, V and total alpha show lower precision for the Scenario #1 condensate. All of the duplicate analyses for the Scenario #2 product show good precision except for the total alpha values.

Table 11. Evaporation Data from Scenario #1 Task

Analyte	Concentrate			Condensate		
	Average mg/L	St. Dev. mg/L	% RSD	Average mg/L	St. Dev. mg/L	% RSD
Ag	0.62	0.05	8.8	< 0.04	0.00	0.0
Al	2387.79	21.63	0.9	< 0.29	0.00	0.0
B	118.55	0.68	0.6	< 0.14	0.00	2.0
Ba	15.25	0.66	4.3	< 0.10	0.00	0.0
Ca	287.74	0.38	0.1	< 0.12	0.00	0.0
Cd	60.72	0.20	0.3	< 0.02	0.00	0.0
Ce	18.43	0.46	2.5	0.34	0.01	2.1
Cr	57.83	0.16	0.3	< 0.03	0.00	0.0
Cu	15.14	0.03	0.2	0.06	0.00	7.2
Fe	306.68	1.43	0.5	< 0.02	0.00	0.0
Gd	< 1.30	0.02	1.2	< 0.12	0.00	0.0
K	986.72	10.63	1.1	< 4.69	0.00	0.0
La	10.63	0.20	1.9	< 0.04	0.00	0.0
Li	3.21	0.13	3.9	< 0.22	0.00	0.0
Mg	< 0.30	0.00	1.2	< 0.03	0.00	0.0
Mn	61.95	1.16	1.9	< 0.00	0.00	0.0
Mo	23.60	1.08	4.6	< 0.27	0.00	0.0
Na	106641.25	9582.31	9.0	4.32	2.95	68.3
Na (M)	4.64	-	-	1.9E-04	-	-
Ni	247.69	3.85	1.6	< 0.07	0.00	0.0
P	377.34	1.63	0.4	< 0.35	0.00	0.0
Pb	151.95	1.09	0.7	< 0.16	0.00	0.0
S	1636.33	4.48	0.3	< 0.69	0.00	0.0
Sb	< 21.04	0.26	1.2	< 1.89	0.00	0.0
Si	19.59	0.08	0.4	0.21	0.00	1.6
Sn	10.37	0.40	3.9	0.27	0.05	18.9
Sr	75.14	0.77	1.0	< 0.04	0.00	0.0
Ti	< 0.43	0.01	1.2	< 0.04	0.00	0.0
U	< 12.69	0.16	1.2	< 1.14	0.00	0.0
V	0.56	0.02	3.0	0.01	0.00	14.6
Zn	21.54	0.19	0.9	< 0.02	0.00	0.0
Zr	15.75	0.12	0.7	< 0.11	0.00	0.0
Anions	mg/L	mg/L	-	mg/L	mg/L	-
F	1469	2	0.2	< 2.00	0.00	0.0
Cl	1252	8	0.6	< 2.00	0.00	0.0
CHO2	6239	53	0.9	< 10.00	0.00	0.0
NO2	37681	229	0.6	< 10.00	0.00	0.0
NO3	112986	609	0.5	< 10.00	0.00	0.0
PO4	2182	43	2.0	< 10.00	0.00	0.0
C2O4	1108	22	1.9	< 10.00	0.00	0.0
SO4	4792	51	1.1	< 5.00	0.00	0.0
Radionuclides	mCi/L	mCi/L	-	mCi/L	mCi/L	-
Total alpha	5.92E-01	2.20E-04	0.0	7.17E-04	4.51E-04	62.9
Total beta	263	3.96	1.5	1.63E-04	7.64E-06	4.7
Cs-137	159	3.03	1.9	< 3.56E-04	1.50E-05	4.2

Table 12. Analytical Characterization of Product Mixture from Scenario #2 Task

Analyte	Average mg/L	St. Dev. Gm/L	%RSD
Ag	0.96	0.03	3.2
Al	2736.25	12.52	0.5
B	136.53	1.03	0.8
Ba	16.26	0.90	5.5
Ca	308.04	1.50	0.5
Cd	66.17	0.79	1.2
Ce	28.10	0.45	1.6
Cr	80.11	0.17	0.2
Cu	16.47	0.06	0.4
Fe	587.38	6.91	1.2
Gd	1.41	0.02	1.6
K	1079.68	24.39	2.3
La	15.52	0.01	0.0
Li	< 2.43	0.04	1.6
Mg	< 0.30	0.00	1.6
Mn	99.76	1.90	1.9
Mo	26.35	0.18	0.7
Na	123555.54	436.98	0.4
Na(M)	5.38	-	-
Ni	274.18	2.04	0.7
P	428.20	3.59	0.8
Pb	176.01	2.00	1.1
S	1889.85	25.92	1.4
Sb	< 21.33	0.34	1.6
Si	23.98	0.18	0.7
Sn	13.09	0.27	2.1
Sr	81.25	0.17	0.2
Ti	< 0.44	0.01	1.6
U	< 12.87	0.20	1.6
V	0.73	0.01	1.6
Zn	24.66	0.15	0.6
Zr	27.14	0.03	0.1
Anions	mg/L	mg/L	-
F	1619	14	0.9
Cl	1394	2	0.1
CHO2	5508	231	4.2
NO2	42320	670	1.6
NO3	130339	1266	1.0
PO4	2376	46	1.9
C2O4	1162	18	1.6
SO4	5507	7	0.1
Radionuclides	mCi/L	mCi/L	-
Total Alpha	6.28E-01	9.26E-02	14.7
Total Beta	296	3.25	1.1
Cs-137	169	2.35	1.4

Comparison of concentrate and condensate stream characterizations from evaporation processes to the evaporator feed characterization provides concentration factors (concentrate/feed) and decontamination factors (feed/condensate). The evaporator feed material used in this Scenario #1 testing was not characterized after blending the AN-107 waste feed, recycle, and the Hanford process water. However, analytical data for the three input streams can be used to calculate the final feed composition for key analytes. These data can be compared to the measured concentrate and condensate data shown in Table 11. Table 13 shows the calculated concentration of certain key analytes in the evaporator feed along with the corresponding analyte concentrations from Table 11. The concentration factor determined from this data is in the range of 2.2 – 2.7; the target is 3. The decontamination factors determined from this data are in the range of 10^5 for the radionuclides, 10^4 for sodium and $> 10^3$ for the other analytes.

Table 13. Concentration Factors and Decontamination Factors for Scenario #1 Testing

Analyte	Calculated Feed (mg/L)	Analyzed Concentrate (mg/L)	Concentration Factor	Analyzed Condensate (mg/L)	Decontamination Factor
Na	46017	106641	2.32	4.32	1.07E+04
Al	948	2388	2.52	< 0.285	> 3.33E+03
S	738	1636	2.22	< 0.69	> 1.07E+03
Ca	106	287.74	2.72	< 0.12	> 8.83E+02
NO3	52144	112986	2.17	< 10	> 5.21E+03
NO2	13856	37681	2.72	< 10	> 1.39E+03
	(mCi/L)	(mCi/L)		(mCi/L)	
Total Beta	103	263	2.55	1.63E-04	6.33E+05
Cs-137	58	159	2.73	3.56E-04	1.64E+05

3.4 PHYSICAL PROPERTIES ANALYSES

Both product solutions from the first scenario evaporation testing of AN-107 mixed with recycle described in Section 3.1 and for the second scenario mixing of AN-107 with concentrated recycle described in Section 3.2, were analyzed for physical properties including slurry and filtered supernatant density, volume and weight percent centrifuged solids, weight percent total dry solids (in centrifuged solids), and weight percent dissolved solids in the supernatant. These measurements were performed in duplicate. Data for these analyses are shown in Table 14 for the concentrate from Scenario #1 and the mix from Scenario #2. Table 14 also shows the original data for the as-received AN-107 for comparison. These data were determined from centrifuging 5-mL aliquots of the product solutions from both scenarios in 10-mL centrifuge tubes. The total amount of dried solids was determined from separate tests that dried 5-mL samples in small glass beakers.

The slurry density values indicate (as expected) that the Concentrate solution ($\text{Na}^+ \sim 4.6\text{M}$) is less dense than the Mix solution ($\text{Na}^+ \sim 5.4\text{M}$) due to the higher salt concentration of the mix solution. The data shown for the vol% and wt% centrifuged solids is suspect due to the difficulty in measuring the small amount of insoluble solids in these very dark, opaque AN-107 product solutions. The wt% total solids average was also higher for the mix sample vs. the concentrate sample. Little difference was observed in the wt% total solids and the wt% soluble solids for each product sample. Thus the calculated wt% insoluble solids gives negative numbers in three of the four tests. Thus application of centrifuge, decant and drying methods to determine the wt% undissolved solids in the product solutions from these tests was unsuccessful. This is likely due to the fact that these product solutions have relatively low amounts of undissolved solids and the AN-107 waste feed mixed with recycle solution matrix is a very dark, opaque liquid.

A direct measure of the amount of insoluble solids can be obtained from separate tests in which filtered solids were obtained for crystalline and elemental characterization (See Section 3.5). In those tests, 30-mL aliquots of the concentrate and mix solutions were centrifuged and the supernatant was decanted, followed by rinsing, filtration, and drying of the remaining centrifuged solids. Those samples produced at least 50 mg of solids for each of the Concentrate and Mix products. Therefore, using the measured slurry density of each of these product solutions from Table 14, there was at least $(0.05 \text{ g} / (30 \text{ mL} * 1.266 \text{ g/mL})) * 100 = 0.132 \text{ wt\%}$ insoluble solids for the Concentrate (Scenario #1 product) and at least $(0.05 \text{ g} / (30\text{mL} * 1.297 \text{ g/mL})) * 100 = 0.128 \text{ wt\%}$ insoluble solids for the Mix (Scenario #2 product).

Table 14. Physical Properties of Product Solutions

Property	Units	AN-107* Avg.	%RSD	Concentrate (Scenario #1)				Mix (Scenario #2)			
						Avg.	%RSD			Avg.	%RSD
Slurry Density	g/mL	1.42	0.41	1.267	1.266	1.266	0.02	1.295	1.300	1.297	0.25
Filtered Supernate Density ¹	g/mL	1.415	0.04	1.218	1.219	1.218	0.05	1.300	1.299	1.300	0.01
Vol % Centrifuged Solids ²	Vol%	8	25	~4	~4	~4	0	~4	~4	~4	0
Wt. % Centrifuged Solids ³	Wt%	6.879	4.95	3.23	3.98	3.61	14.71	8.11	7.46	7.79	5.90
Wt% Total Dried Solids ⁴	Wt%	49.5	0.27	33.70	33.90	33.80	0.42	36.50	37.80	37.15	2.47
Wt% Dissolved Solids ⁵	Wt%	49.25	0.51	36.76	33.95	35.36	5.62	38.40	36.26	37.33	4.05
Wt% Insoluble Solids ⁶	Wt%	0.483	77.6	-4.84	-0.08	NA	NA	-3.08	2.42	NA	NA

Notes: * AN-107 data from Ref.9.

- 1) Determined for supernate that was decanted off of the centrifuged samples for the Concentrate and Mix
- 2) All centrifuged samples from the Concentrate and Mix had ~ 0.2 mL of solids after centrifuging.
- 3) Centrifuge solids dried overnight at 115 °C
- 4) 5-mL aliquots of Concentrate and Mix solutions dried overnight at 115 °C
- 5) Decanted supernatants dried overnight at 115 °C
- 6) Calculated from total dried solids and dissolved solids

Both product solutions from the first scenario evaporation testing of AN-107 mixed with recycle and for the second scenario mixing of AN-107 with concentrated recycle, were also analyzed for solution pH, oxidation-reduction potential (ORP) and turbidity via the methods discussed in the Stream Blending Report.⁶ Table 15 shows the data for the concentrate and mix samples. Each sample had a pH of approximately 13 and a negative redox potential. The turbidity for both product solutions was indeterminate with readings in ‘nephelometric turbidity units’ (ntu) > 1,000. The turbidity meter was checked with standards in the range of 5 to 500 ntu.

Viscosities were not measured for the product solutions due to a technical problem with the current configuration for remote rheology testing for low viscosity samples at SRTC. However, modeling calculations suggest a supernatant viscosity of about 2 cP that is similar to the experimental value reported for the recycle mixed with as-received AN-107 shown in Table 15. These viscosity values and those reported for the as-received AN-107 sample in the range of 5.9 to 9.4, are all in the range of 0.4 to 15 cP, which is the range associated with the Low Activity Waste (LAW) Concentrate Receipt Vessel (CRV) in the WTP.¹⁰

Table 15. Properties of Product Solutions Compared to AN-107 Mixed with Recycle

Properties	AN-107¹ Mixed with Recycle	Scenario #1, Evaporation of AN-107 Mixed with Recycle	Scenario #2, AN-107 Mixed with Concentrated Recycle-Only
pH	13	13	13
Turbidity (ntu)	NA	>1000	>1000
Redox Potential (mv)	-217	-309	-385
Initial Temperature (°C)	25	27	27
Viscosity (cP)	3	NM	NM

Notes:

1) Reference 6

NM = not measured due to rheometer instrument malfunction. Modeled viscosity values for the supernatant phase (approximately 2 cP) are discussed in Appendix A.

3.5 RESULTS OF SOLIDS ANALYSIS

Both of the product solutions from these tests were analyzed for solids characterization to investigate the qualitative content of the trace amount of undissolved solids using both x-ray diffraction and scanning electron microscopy with energy dispersive x-ray analysis. The product solutions from the Scenario #1 Test as well as the product solution from the Scenario #2 Test were centrifuged. After centrifuging these 30-mL solutions, the supernatant was decanted off of the trace amount of solids remaining in the bottom of the centrifuge tube. These damp solids were transferred to a 0.45 micron filter where they were rinsed with approximately 70 mL of deionized water to remove any interstitial liquid and soluble salts. After drying the rinsed solids overnight at ambient conditions, the solids were sampled into shielded bottles for removal from the SCF up to the Analytical Development Section (ADS) x-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) instruments for analysis. Approximately 50 mg of each solid was transferred for XRD analyses. Milligram quantities of the solids were also mounted on special SEM mounts for microscopy and energy dispersive x-ray analysis.

3.5.1 Solids Analysis from Scenario #1 Testing – Evaporation of AN-107 Mixed with Recycle and Process Water

Figure 12 shows the XRD pattern resulting from the rinsed and dried solid sample. Rinsing of the original filtered solids was performed to remove any salts associated with residual supernatant phase, i.e., interstitial supernatant, that could have resulted from subsequent drying of the filtered solids. No analyses were performed on the solids before rinsing, so there is no way to conclude if the rinsing removed any soluble solids that were originally present in the total insoluble solids resulting from evaporation. This XRD trace shows Fe as hematite (Fe_2O_3) and Si as quartz (SiO_2). The main peak for the quartz crystal is located at approximately 2-theta of 26.5. All of the other main peaks identified in the trace are attributed to the hematite. Small peaks are observed in the trace at 2-theta degrees of approximately 12 that could also be attributed to aluminum-containing crystals similar to those reported by Barnes (see Appendix D of Ref. 6), but were not present in large enough quantity to be identified by the XRD.

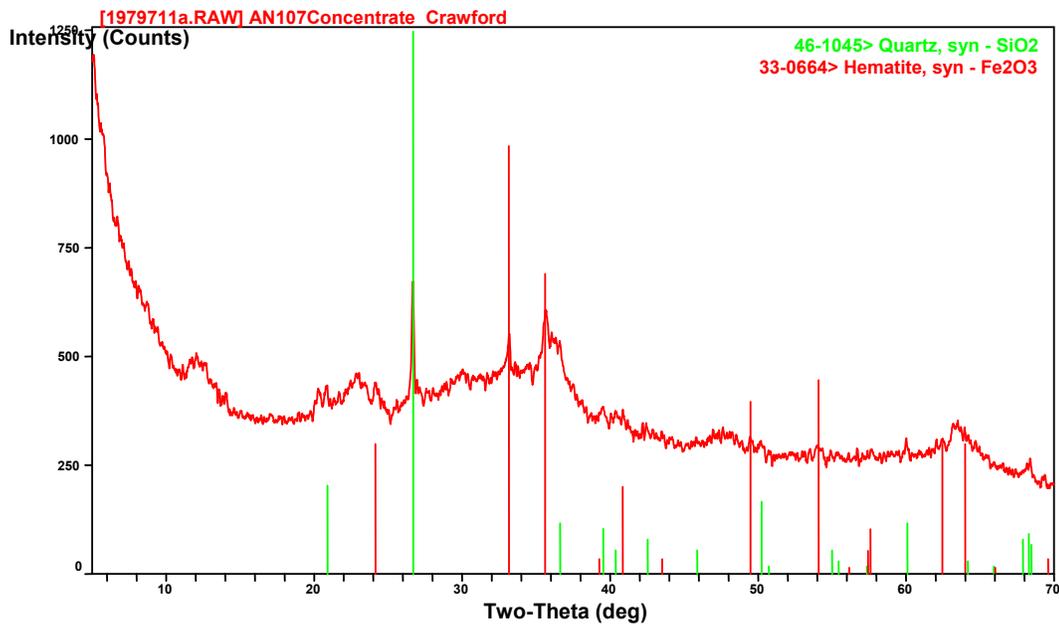


Figure 12. XRD Pattern Resulting from the Rinsed and Dried Solid Sample from Scenario #1 Testing.

SEM photographs of these solids are shown in Figure 13 - Figure 16. These photos are taken of the images resulting from either Secondary Electron (SE) shown in Figure 13 and Figure 14, or Backscattered Electron (BSE) shown in Figure 15 and Figure 16, modes of the SEM. Generally, the SEM technique uses backscattered electrons, or incident electrons, to indicate potential density differences in the image particles. Images using secondary electrons that involve actual electrons from the matrix material provide topography images of the matrix. From these SEM images certain elemental characterization information can be obtained by performing the energy dispersive x-ray analysis on localized areas or spots of the SEM images. Figure 17 shows the EDAX trace for the spots 'A' indicated on Figure 16. This EDAX trace shows the elements Al, K, V, Cr, Mn and Fe are present. The gold and palladium result from the coating used in mounting the samples for this technique. Aluminum and iron are the prominent peaks.

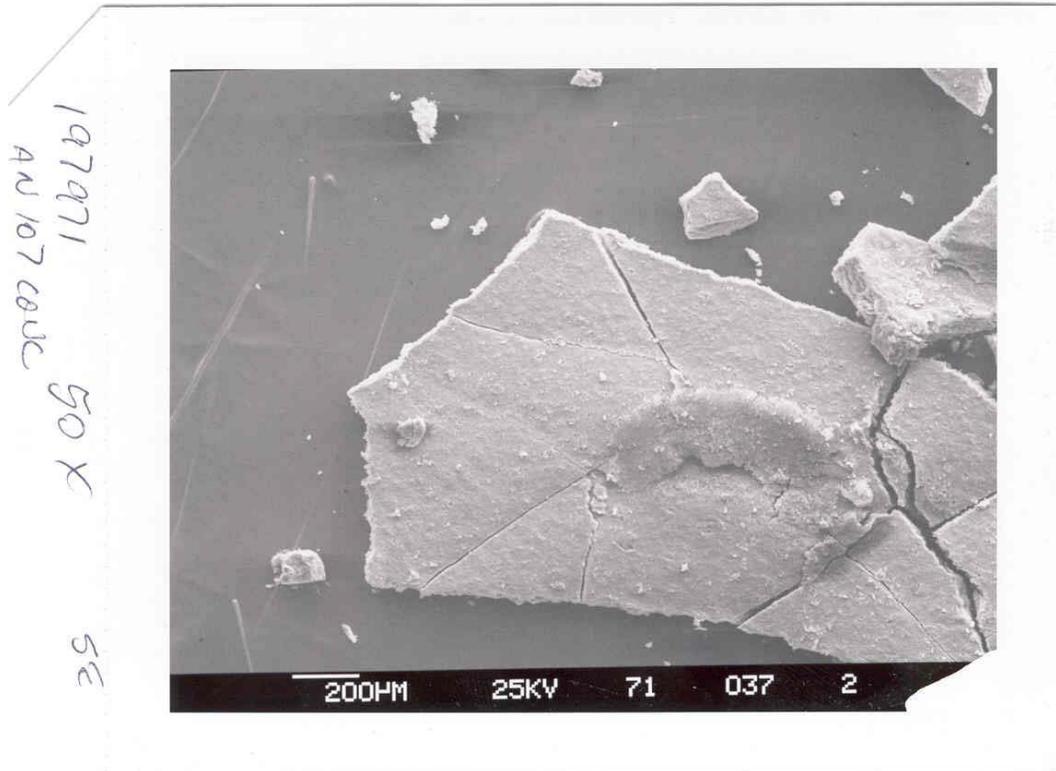


Figure 13. 50X Magnification, Secondary Electron Image

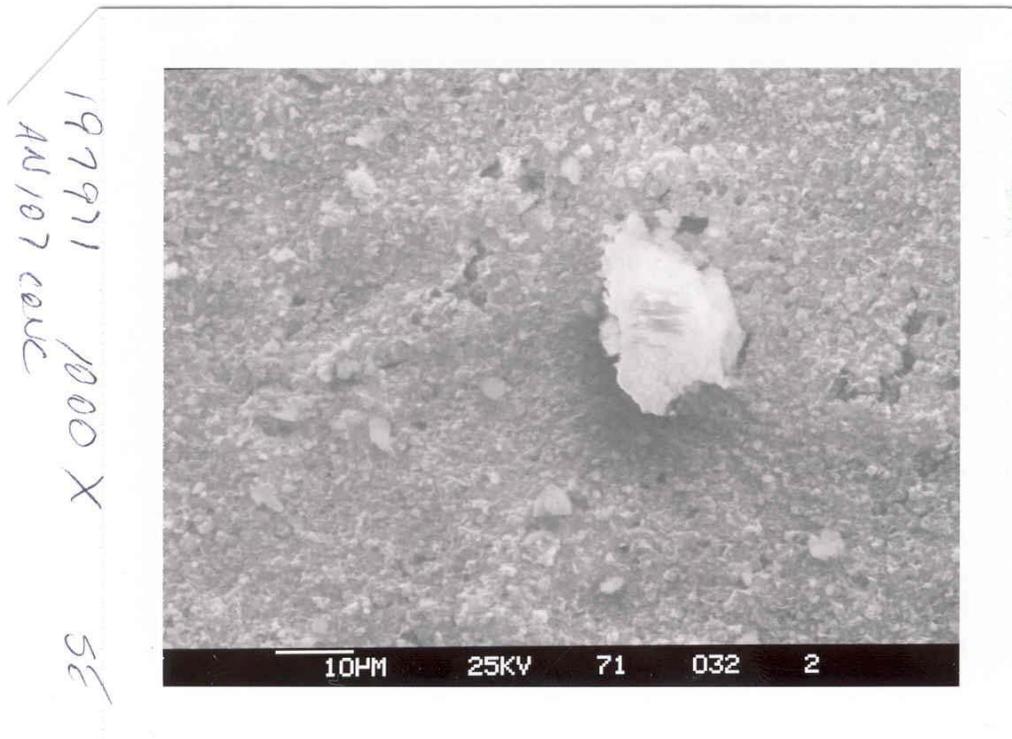


Figure 14. 1,000X Magnification, Secondary Electron Image

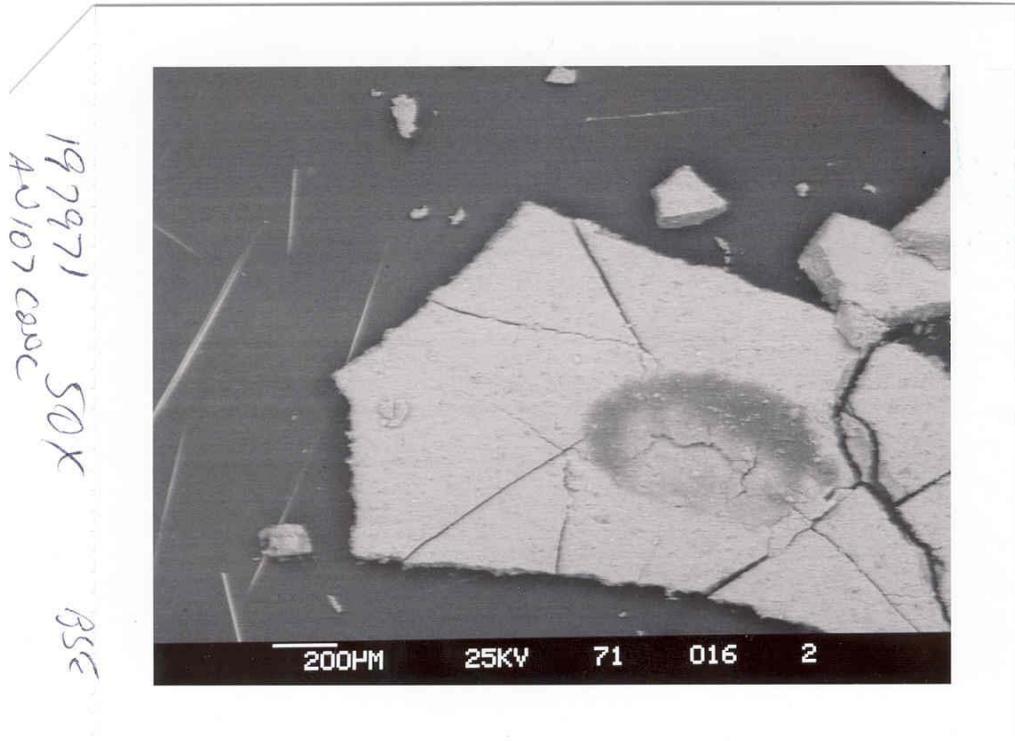


Figure 15. 50X Magnification, BackScattered Electron Image

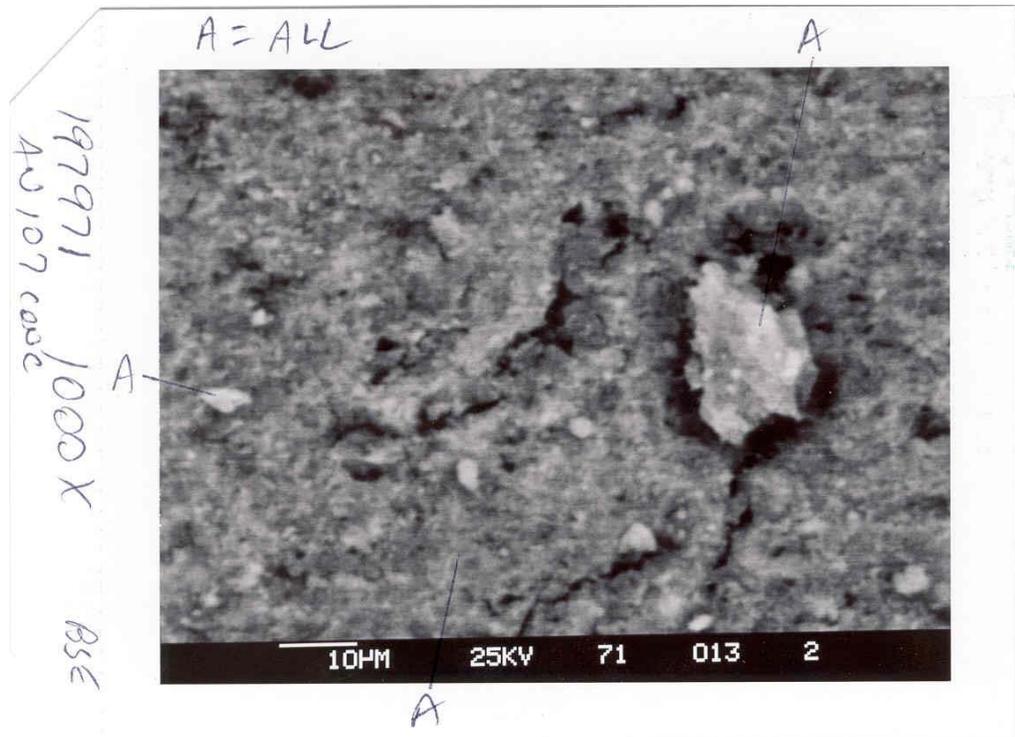


Figure 16. 1,000X Magnification, BackScattered Electron Image

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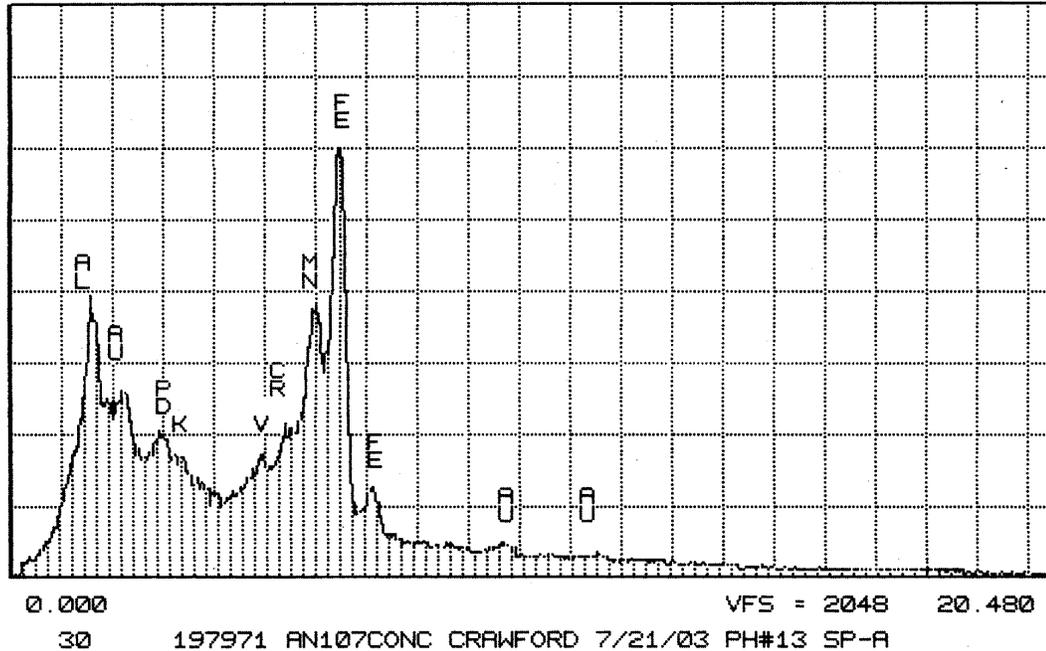


Figure 17. Energy Dispersive X-ray Analysis Trace of Spots A

3.5.2 Solids Analysis from Scenario #2 Testing – Evaporation of Recycle-Only, then Mixed with AN-107

Figure 18 shows the XRD pattern resulting from the rinsed and dried solid sample. Rinsing of the original filtered solids was performed to remove any salts associated with residual supernatant phase, i.e., interstitial supernatant, that could have resulted from subsequent drying of the filtered solids. No analyses were performed of the solids before rinsing so there is no way to conclude if the rinsing removed any soluble solids that were originally present in the total insoluble solids resulting from evaporation. These XRD traces show very similar results to the previous solids analysis with the Scenario #1 product, with Fe as hematite (Fe_2O_3) and Si as quartz (SiO_2) being the only crystalline solids identified. Small peaks are observed in each trace at 2-theta degrees of approximately 12 that could also be attributed to aluminum-containing crystals similar to those reported by Barnes (see Appendix D of Ref. 6), but were not present in large enough quantity to be identified by the XRD.

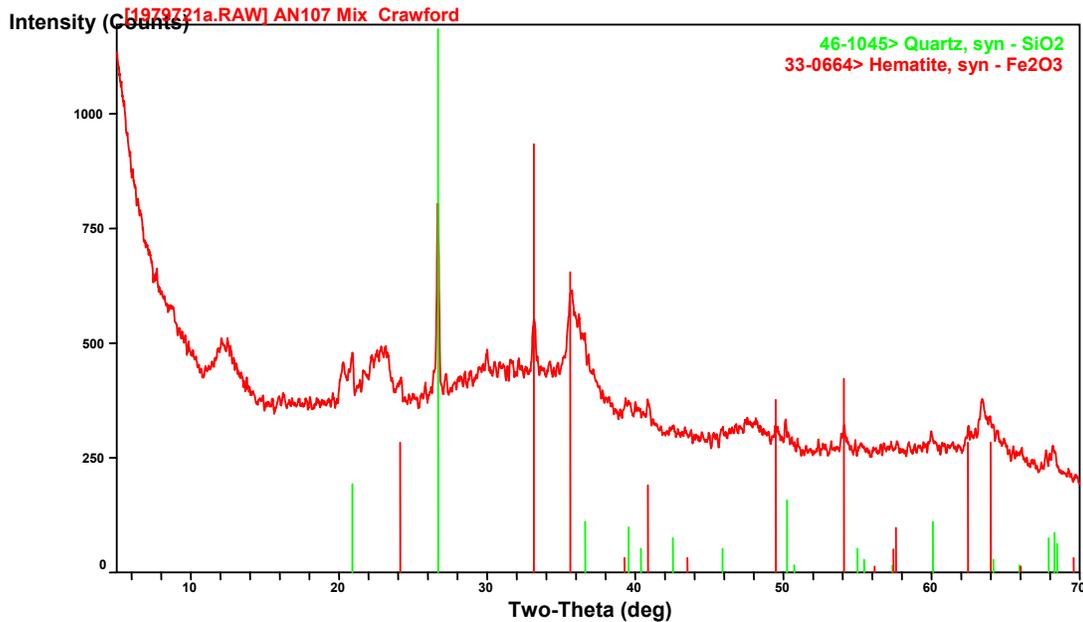


Figure 18. XRD Pattern Resulting from the Rinsed and Dried Solid Sample from Scenario #2 Testing

SEM photographs of these solids are shown in Figure 19 - Figure 24. These photos are taken of the images resulting from SE shown in Figure 19 - Figure 21, or BSE shown in Figure 22 - Figure 24, modes of the SEM. From these SEM images certain elemental characterization information can be obtained by performing the energy dispersive x-ray analysis on localized areas or spots of the SEM images. Figure 25 shows the EDAX trace for the spot 'A' of Figure 23. This EDAX trace shows the elements Zr, K, V, Mn and Fe are present. The gold and palladium result from the coating used in mounting the samples for this technique. Figure 26 and Figure 27 show similar traces from spots labeled as 'B' and 'C' in Figure 24. These two spots show similar elemental composition of Si, Mn and Fe. Some vanadium also shows in spot B and some S is indicated in spot C.

Elementals measured in these solids derived from scenario #1 and scenario #2 testing have also been measured in the feed streams used in this testing. Table 3 and Table 4 of this report indicate that all of the elementals (Al, Cr, Fe, Mn, Si, Fe, V, Zr, S and K) were present at detectable levels in the insoluble solids associated with the actual as-received AN-107.⁹ Similar data for the insoluble solids associated with the recycle stream (Table 7) indicate that this stream also had detectable levels of Al, Cr, Fe, Mn, Si and V in the insoluble solids.⁶

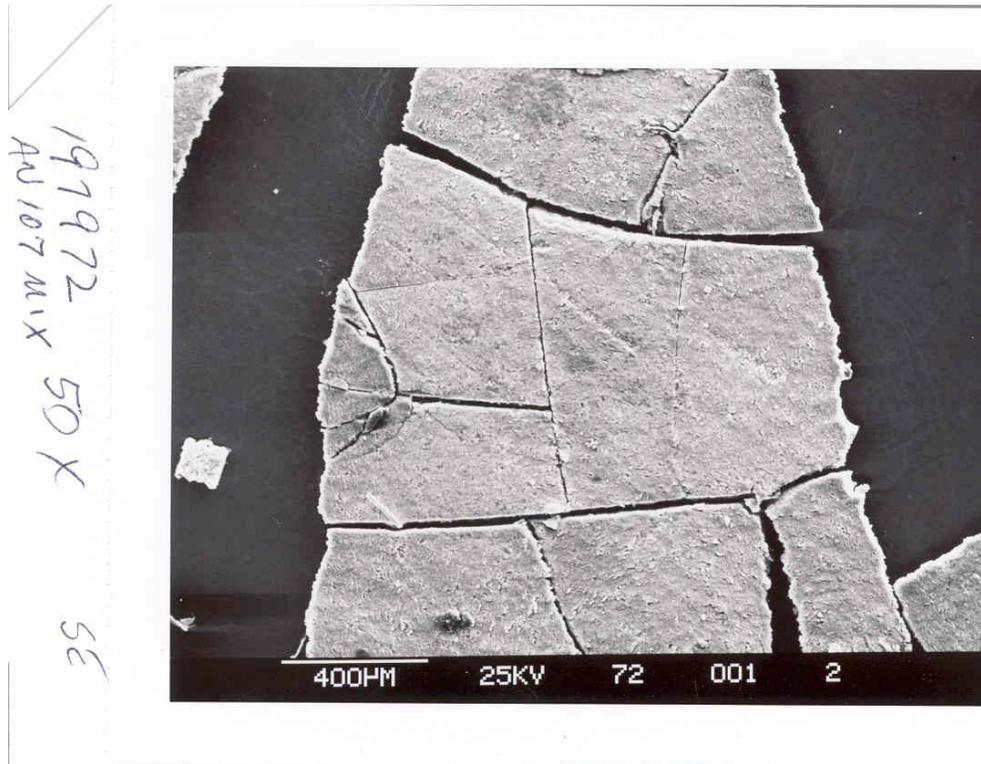


Figure 19. 50X Magnification, Secondary Electron Image

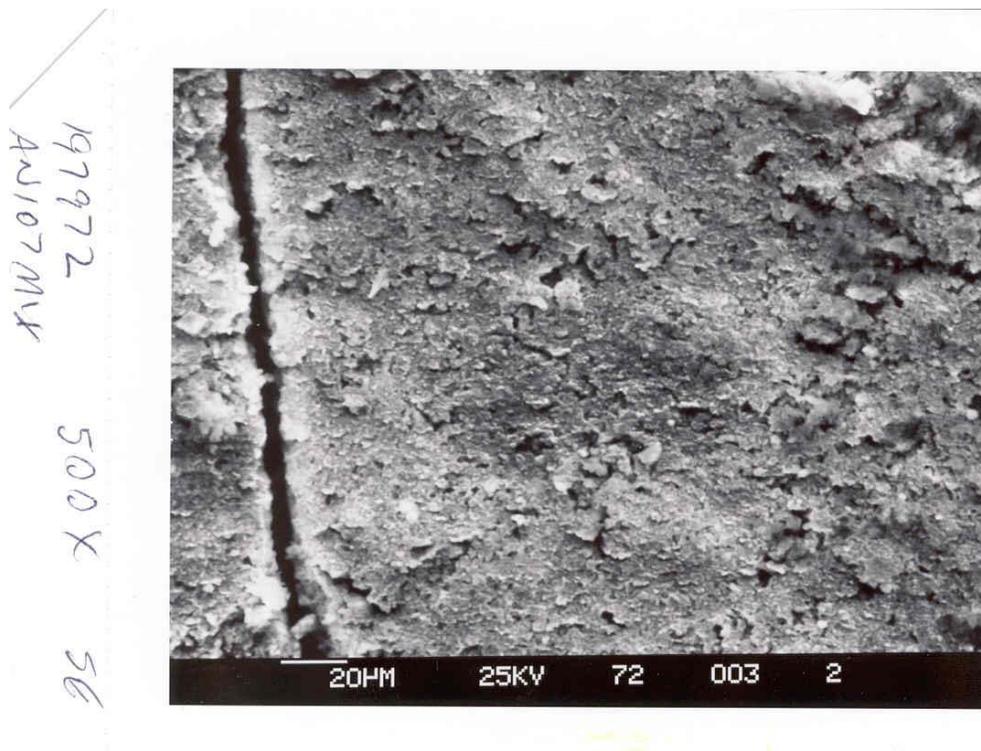


Figure 20. 500X Magnification, Secondary Electron Image

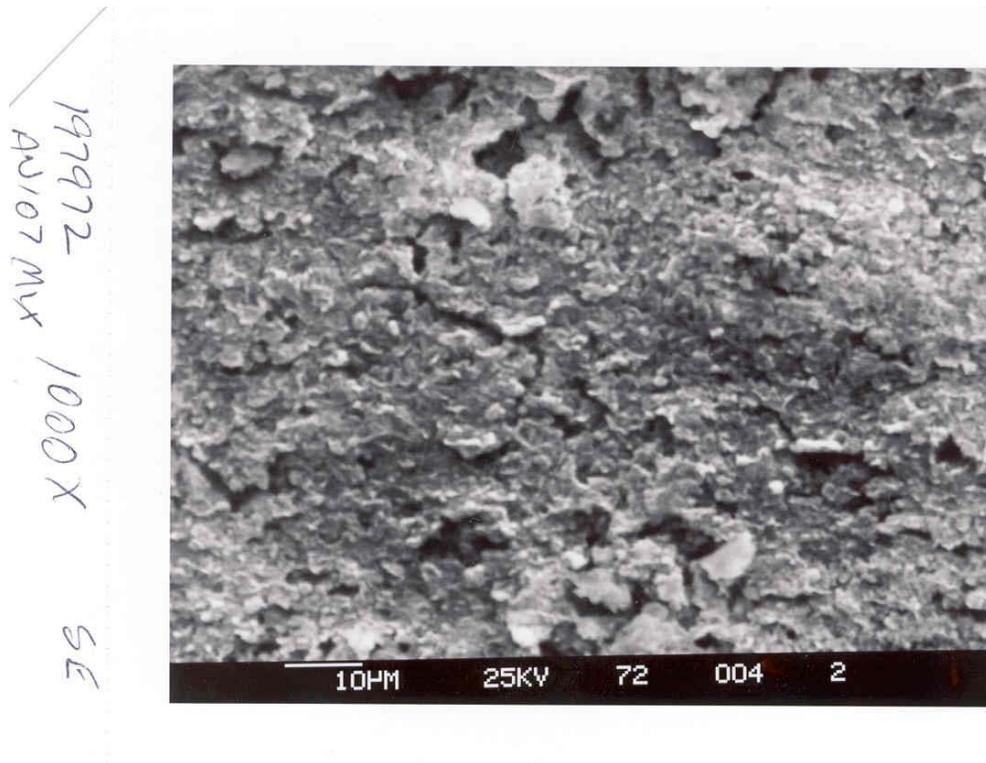


Figure 21. 1,000X Magnification, Secondary Electron Image

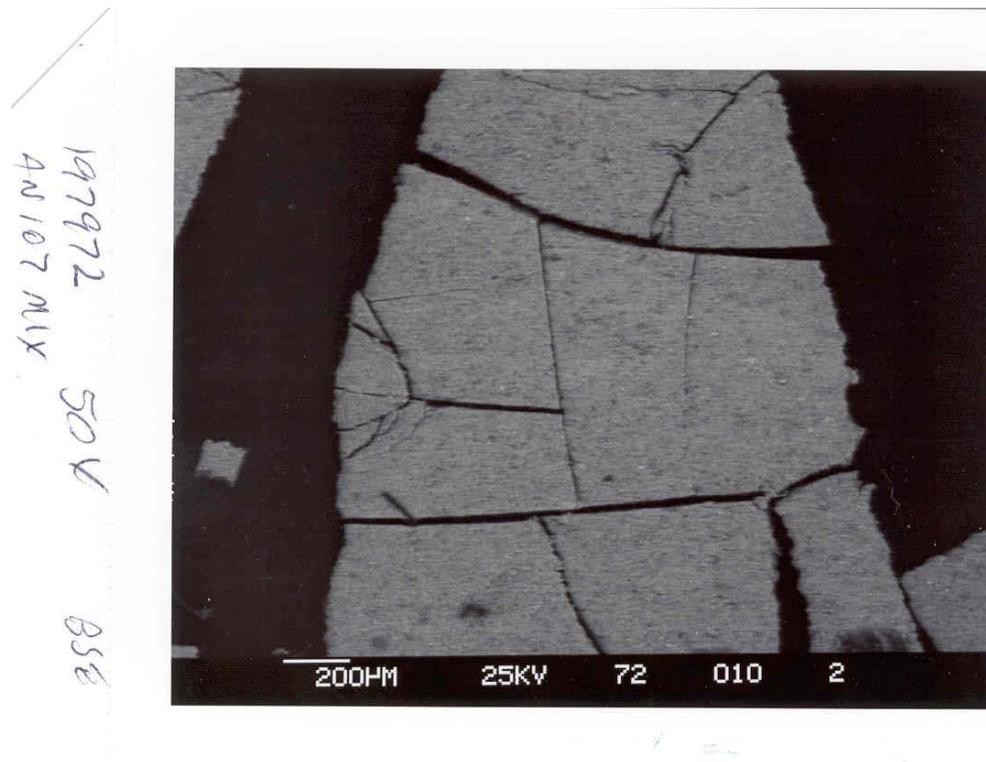


Figure 22. 50X Magnification, BackScattered Electron Image

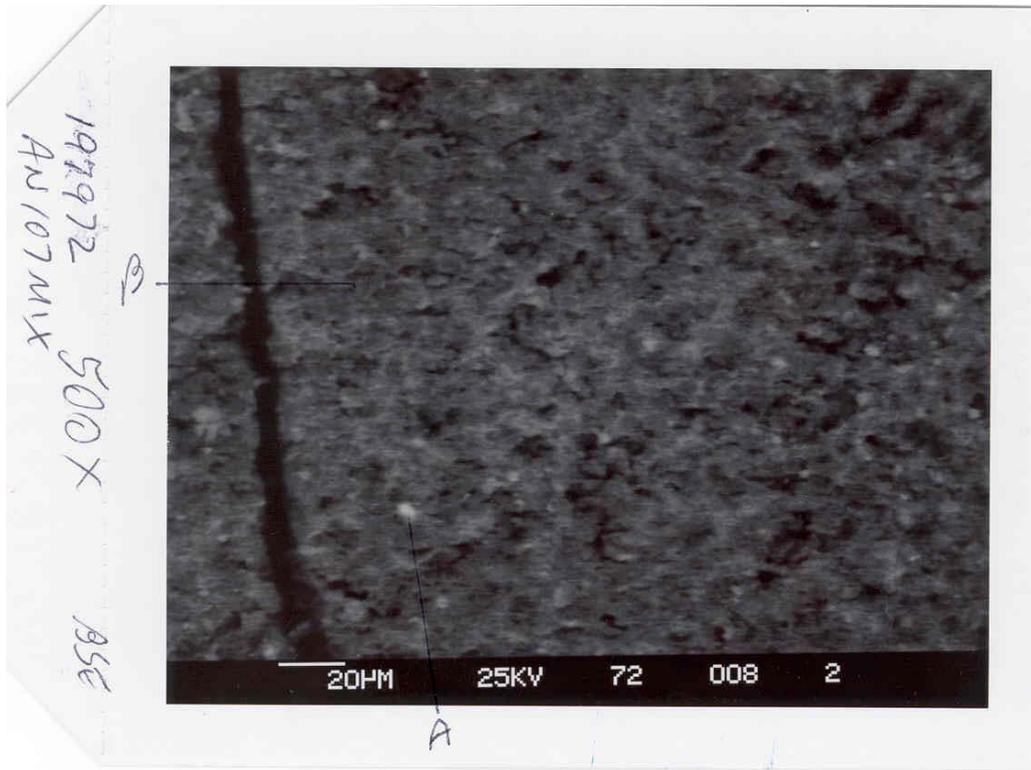


Figure 23. 500X Magnification, BackScattered Electron Image

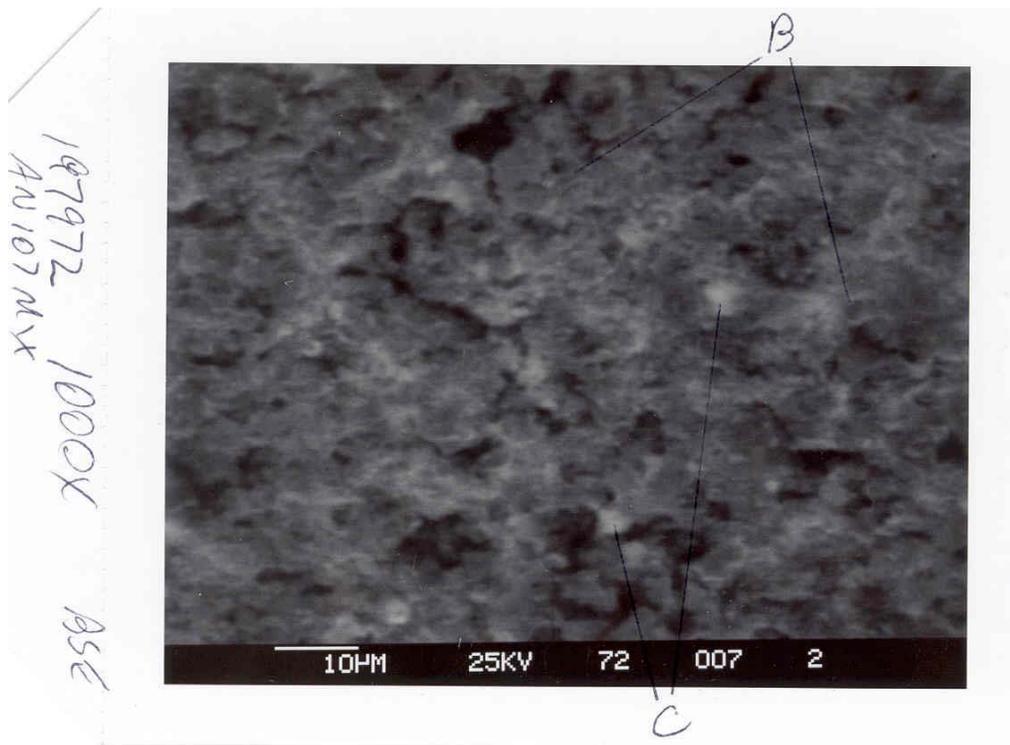


Figure 24. 1,000X Magnification, BackScattered Electron Image

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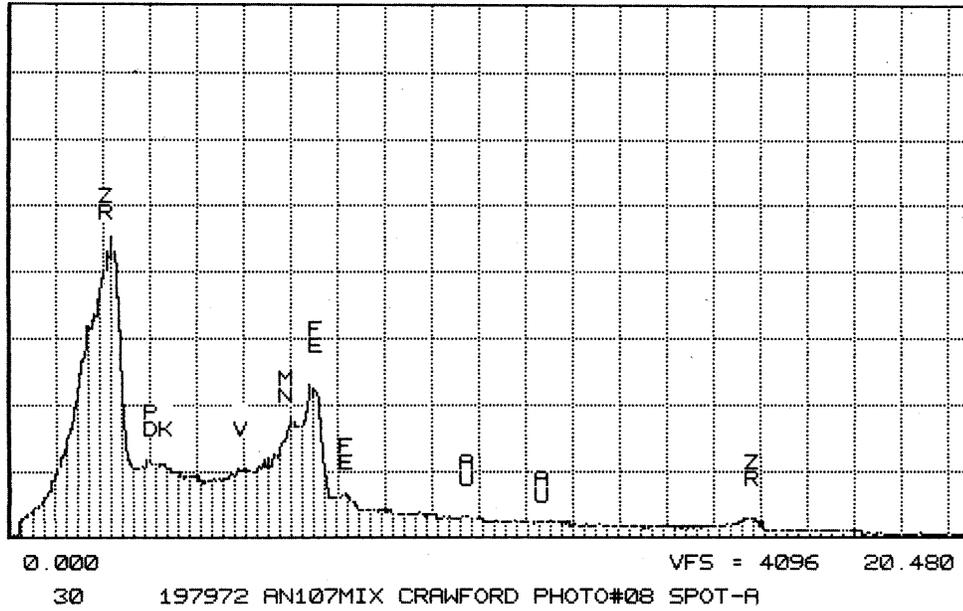


Figure 25. Energy Dispersive X-Ray Analysis Trace of Spot A

TN-5502 WSRC CSEM.
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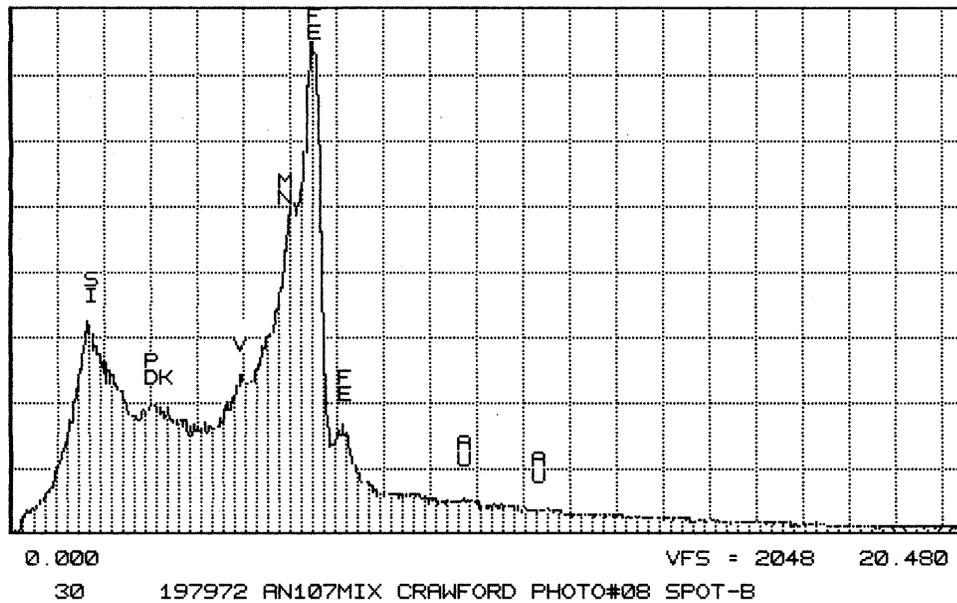


Figure 26. Energy Dispersive X-Ray Analysis Trace of Spot B

TN-5502 WSRC CSEM.
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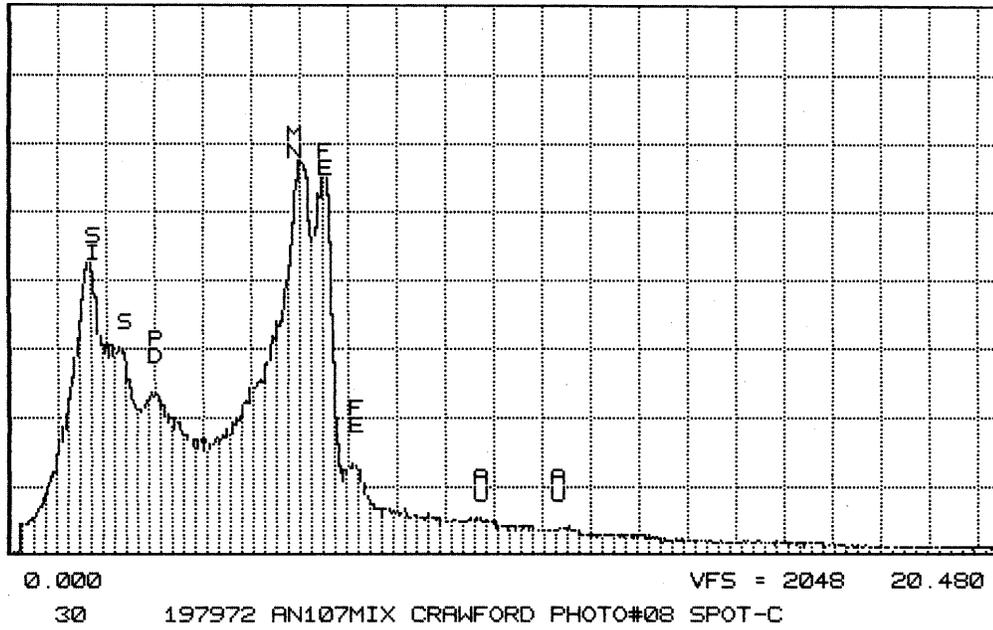


Figure 27. Energy Dispersive X-Ray Analysis Trace of Spot C

4.0 CONCLUSIONS

The proof-of-technology testing described in this report suggests that secondary-waste recycle streams will not have significant influences or deleterious impacts on the planned waste feed evaporation process for the WTP. It was shown in this testing that in either scenario of recycle introduction with the actual AN-107 waste feed, that the final products will be sufficient for processing through the Sr/TRU precipitation and filtration pretreatment step for Envelope C supernatants. The modeled viscosities of these product solutions are similar to measured values reported for related stream blending tests,⁶ and for the original as-received AN-107.⁹ The product solutions derived from this testing contained no potentially troublesome solids or gels and the mixing of recycle with actual AN-107 produced no de gassing or extreme temperature deviations.

Evaporation of either recycles blended with AN-107 or evaporation of recycle-only, followed by blending with AN-107 were shown to not be problematic since no significant foaming occurred. In the case of Scenario #1 testing (recycle blended with AN-107, followed by evaporation) comparison of the evaporation product solutions (concentrate and condensate) with the evaporator feed indicates that concentration factors were similar for several different analytes and that acceptable decontamination factors as high as 10^5 were attained.

The rinsed insoluble solids derived from both of the Scenario #1 and #2 product solutions in these tests contained primarily Fe and Si as crystalline hematite (Fe_2O_3) and quartz (SiO_2), respectively. Several other elements were determined in the solids Al, K, V, Cr, Mn, Zr and S. All of these elements were also detected in the insoluble solids associated with each feed stream for these tests, i.e., either the as-received AN-107 or the surrogate recycle stream. Attempts to measure the amount of insoluble solids in the product solutions were unsuccessful using centrifuge, decant and drying techniques. Estimates from tests involving separation of the solids for XRD and SEM analyses indicate at least 0.13 wt% insoluble solids were present. The model calculations indicated insoluble solids predicted at levels of 1.3 to 1.6 wt%. Comparison of insoluble solids and density values for modeled outputs versus measured values indicate adequate agreement. The modeled pH values were in poor agreement with measured values. However, OLI software vendors have confirmed in previous related testing that the pH predictions by OLI have never been previously validated.

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5.0 FUTURE WORK

No future work is planned related to these tasks involving evaporation of recycle streams mixed with actual radioactive samples.

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6.0 REFERENCES

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3. R. Longwell, Test Exception 24590-WTP-TEF-RT-02-045, 7/31/02.
4. M. J. Barnes, 'Task Technical and Quality Assurance Plan for Recycle Stream Blending for High and Low Level Waste,' WSRC-RP-2002-00158, SRT-RPP-2001-00243, February 27, 2002.
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6. M. J. Barnes, et al., 'Recycle Stream Blending for High and Low Level Waste', WSRC-TR-2003-00156, SRT-RPP-2003-00059, Rev. 0, August 20, 2003.
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10. WTP-RPT-075, Rev. 0, "Interim Report: Development of LAW and HLW Vitrification Physical Property Bounding Conditions and Simulant Verification Criteria," A. Poloski, H. Smith, G. Smith, and B. Calloway, March 2003

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**APPENDIX A.
OLI SIMULATION OF AS-RECEIVED AN-107 EVAPORATION
WITH RECYCLE**

Simulation Scenarios

The recycle stream used in these studies was generated under the Stream Blending task.⁶ In the first scenario testing, this recycle stream was added to as-received AN-107 along with Hanford process water that had been adjusted with NaNO₂ and NaOH. The volumetric ratios of waste : recycle: process water used were 100 : 116.7 : 358. This dilute composite stream was then evaporated to target 6M Na⁺. In the second scenario testing the recycle stream was evaporated to an endpoint such that when blended with actual as-received AN-107, the blend would be at 6M Na⁺. The volumetric ratio of recycle to waste feed prior to concentration for evaporation of recycle only was 3.5:1.

The total recycle stream (composite) used was represented by several input recycles. The 11 recycle inputs are listed in Table A- 1 with the overall volumetric fraction in the composite product recycle.⁶ These data show that recycle #5 HLW SBS, the recycle #10 Spent resin liquor, the recycle #11 first wash, and the recycles #2 and #1 UF caustic and acid cleaning were the largest volume fraction contributors to the overall recycle composite.

Table A- 1. Recycle Stream Inputs

Recycle Stream	Volume Percent
1) UF acid cleaning	0.0850272
2) UF caustic rinse	0.1275408
3a) Cs IX pre-elution rinse AN-105 Simulant	0.00642309
3b) Cs IX pre-elution rinse 0.08M NaOH	0.0160577
4) Pretreatment condenser condensate	0.0136272
5) HLW SBS	0.3383596
6) HLW canister decon	0.0329582
7) Pretreatment scrubber condensate	0.0143902
8) Pretreatment HEME	0.0295822
9) RDF HEME	0.0067098
10) Spent resin liquor	0.170
11) First wash (leach and second wash steps excluded for envelope C)	0.159528

Except for streams listed below, simulant recipes used to make up the recycle streams were followed for the simulation. The relative volumetric ratios used for each of the streams are those given in Tables 3-34 – 3-35 of recycle stream blending report (WSRC-TR-2003-00156)⁶.

- Cs IX pre-elution rinse: A blend of 0.08M NaOH with AN-105 simulant recipe at a volumetric ratio of 5:2 was used, as was done for the actual simulant
- HLW SBS: The composition used for all other pretreatment models was used here, and is based on analytical results of the HLW SBS from Duratek

Some of the analytical data of the actual as-received AN-107 is not precise.⁹ For example, results of the microwave digested centrifuged solids measured Si (on average) at 442 mg/kg, while the blank gave a value of 326 mg/kg. Blanks for many of the centrifuged solids species measured significant values. Therefore, the simulation results should be viewed in light of the possible error in the initial input composition. Because Si was an extreme case, two simulations were run for each of the scenarios described above at the upper and lower Si concentration bounds given by the analytical data.

The target of the concentrated blend was 6M Na in the aqueous phase for both scenarios testing in this work. However, the bench-scale runs did not meet this target as determined from sodium analysis of the products. Therefore, two runs of each evaporation method were done, one to the Na endpoint target concentration, and one to the bench-scale Na endpoint analyzed concentration. The analyzed sodium concentration from the first scenario testing was 4.5M Na⁺ and the analyzed sodium concentration from the second scenario testing for evaporation of the recycle only was 5.4M Na⁺.

Simulations that considered the high and low silica and the analyzed and target sodium values were performed. The simulation matrix is summarized in Table A- 2. In all, eight different simulations were performed.

The outputs from the OLI simulations for simulation runs 1-4 at analyzed sodium product concentrations are presented in Table A- 3 and Table A- 4. Table A- 3 presents the OLI mass fraction of undissolved solids output for the scenario #1 blended waste/recycle for high/low silica values (top 2 data sets of Table A- 3) and the OLI mass fraction of solids output for the scenario #2 recycle only for high/low silica values (bottom 2 data sets of Table A- 3). Similar OLI outputs for general properties are shown in Table A- 4 for these same simulations.

The outputs from the OLI simulations for simulation runs 5-8 at target sodium product concentrations of 6M are presented in Table A- 5 and Table A- 6. Table A- 5 presents the OLI mass fraction of undissolved solids output for the scenario #1 blended waste/recycle for high/low silica values (top 2 data sets of Table A- 5) and the OLI mass fraction of solids output for the scenario #2 recycle only for high/low silica values (bottom 2 data sets of Table A- 5). Similar OLI outputs for general properties are shown in Table A- 6 for these same simulations.

Table A- 2. OLI Model Simulations

Simulation	Scenario	Silica Content	Product Sodium Concentration
1	Blended Waste/Recycle, #1	High Si	4.5M (analyzed)
2	Blended Waste/Recycle, #1	Low Si	4.5M (analyzed)
3	Recycle Only, #2	High Si	5.4 M (analyzed)
4	Recycle Only, #2	Low Si	5.4 M (analyzed)
5	Blended Waste/Recycle, #1	High Si	6 M (target)
6	Blended Waste/Recycle, #1	Low Si	6 M (target)
7	Recycle Only, #2	High Si	6 M (target)
8	Recycle Only, #2	Low Si	6 M (target)

Model Predictions for Analyzed Sodium Concentrations

Table A- 3 data indicates that the primary solids (> 10% of total insoluble solids) predicted in the recycle stream (see top row of Table A- 3) are $\text{NaAlCO}_3(\text{H}_2\text{O})$ and $\text{Al}(\text{OH})_3$. The Waste Feed stream shows Na_3FSO_4 and $\text{Fe}(\text{OH})_3$ as predicted primary solids. The product samples from Scenarios #1 and #2, at both the lower and higher silicon levels, in the Cooled Bottoms rows, indicate that for both Scenario #1 and Scenario #2 that $\text{NaAlCO}_3(\text{H}_2\text{O})$ and $\text{Fe}(\text{OH})_3$ are the predicted primary solids. All of these streams also showed smaller amounts of various predicted solids, i.e., less than 10% of total solids. Data shown in Table A- 4 indicate that the predicted properties of the product solutions from Scenario #1 testing (Cooled Bottoms) are insoluble solids at 1.3 wt%, density at 1.21 g/cc, final pH at 10.48 and a final solution viscosity at 1.84 cP. The density, pH and viscosity predicted values are calculated for the solution phase only. Similar data for the Scenario #2 testing indicates insoluble solids at 1.5 wt%, density at 1.25 g/cc, final pH at 10.29 and a final solution viscosity at 2.18 cP. Both of these data sets for Scenario #1 and #2 testing show little variability between the data sets calculated based on either high or low silica input.

Model Predictions for Target 6M Sodium Concentrations

Table A- 5 data indicates that the primary solids (> 10% of total insoluble solids) predicted in the recycle stream (see top row of Table A- 5) are $\text{NaAlCO}_3(\text{H}_2\text{O})$ and $\text{Al}(\text{OH})_3$. The Waste Feed stream shows Na_3FSO_4 and $\text{Fe}(\text{OH})_3$ as predicted primary solids. The product samples from Scenarios #1 and #2, at both the lower and higher silicon levels, in the Cooled Bottoms rows indicate that for both Scenario #1 and Scenario #2 that $\text{NaAlCO}_3(\text{H}_2\text{O})$ and $\text{Fe}(\text{OH})_3$ are the predicted primary solids. All of these streams also showed smaller amounts of various predicted solids, i.e., less than 10% of total solids. Data shown in Table A- 6 indicate that the predicted properties of the product solutions from Scenario #1 testing (Cooled Bottoms) are insoluble solids at 1.6 wt%, density at 1.27 g/cc, final pH at 10.58 and a final solution viscosity at 2.45 cP. The density, pH and viscosity predicted values are calculated for the solution phase only. Similar data for the Scenario #2 testing indicates insoluble solids at 1.6 wt%, density at 1.27 g/cc, final pH at 10.26 and a final solution viscosity at 2.44 cP. Both of these data sets for Scenario #1 and #2 testing show little variability between the data sets calculated based on either high or low silica input. These model predictions at the higher sodium endpoint of 6M also suggest that no significant changes should take place in going from the actual measured sodium values in the range of 4.6M to 5.4M up to the 6M sodium endpoint.

Comparison of Model Output to Measured Values

Certain data from the model outputs shown in Table A- 3 and Table A- 4 can be compared to the measured values from earlier Sections in this report. Table A- 7 lists the model output data versus the measured values for solids (total insoluble and identified individual solids), pH and density. The individual solids listed for the model output are listed in decreasing order of the predicted mass fraction. These data are from the modeled outputs at the 'as-measured' sodium endpoints of approximately 4.6M for Scenario #1 and 5.4M for Scenario #2.

The amount of insoluble solids predicted by the model is in the range of 1.3 to 1.5 wt%. Estimates from experimental tests showed that the amount of solids after rinsing were at least 0.13 wt%. Iron-solids were indicated by the model and were measured in the rinsed solids by XRD. Sodium-aluminum-carbonate solids were also predicted. No crystalline sodium-aluminum-carbonate crystals were detected. However, as indicated earlier in the text, a small peak in both XRD patterns (Figure 12 and Figure 18) could possibly be due to aluminum-containing crystalline solids. Aluminum was also identified in the elemental composition of certain washed solids as shown in Figure 17. The solids MnCO_3 and ZrO_2 were also predicted. Manganese and zirconia were identified in the energy-dispersive elemental analysis of the washed solids.

The pH values measured for the final product solutions were about 13 versus lower values in the range of 10.3 to 10.4 predicted for the product solutions from OLI modeling. Discrepancies between predicted pH values and measured pH values have been reported before in similar waste feed evaporation testing with surrogate systems.⁸ Predicted density values from OLI modeling were in good agreement with the final product measured slurry densities.

The overall results of the model predictions seem to agree adequately with the measured parameters for both of the two scenarios investigated in this work.

Table A-3. Species Mass Fraction of Undissolved Solids for Analyzed Sodium Product Concentrations

	RECYCLE	NAALCO3OH2	ALOH3	ZRSO42.4H2O	FEIIIPO4.2H2O	NASGEL.15.5H2O	ZNC2O4.2H2O	CAF2	CEPO4	CDC2O4	NIC2O4.2H2O	MGCRO4	FE8SEO14		
		5.86E-01	2.20E-01	7.51E-02	4.61E-02	1.98E-02	1.49E-02	1.42E-02	1.11E-02	6.23E-03	5.53E-03	1.68E-03	4.09E-06		
Scenario	Blended Waste/Recycle, High Si, 4.5M Na Endpoint														
#1		NA3FSO4	FEIIIHO3	NAALCO3OH2	NAHCO3	CAF2	MNCO3	NA2C2O4	SRCO3	NDPO4.2H2O	CEPO4				
	WASTE FEED	4.22E-01	2.17E-01	9.98E-02	8.40E-02	7.00E-02	6.00E-02	2.84E-02	1.30E-02	4.31E-03	1.74E-03				
	COOLED BOTTOMS	NAALCO3OH2	FEIIIHO3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3 2.46E- 04	FE8SEO14 2.73E-06
		7.24E-01	1.17E-01	4.21E-02	3.68E-02	3.16E-02	1.74E-02	8.22E-03	6.71E-03	5.92E-03	5.05E-03	2.74E-03	1.97E-03		
Scenario	Blended Waste/Recycle, Low Si, 4.5M Na Endpoint														
#1		NA3FSO4	FEIIIHO3	NAALCO3OH2	NAHCO3	CAF2	MNCO3	NA2C2O4	SRCO3	NDPO4.2H2O	CEPO4				
	WASTE FEED	4.20E-01	2.16E-01	9.93E-02	8.88E-02	6.97E-02	5.97E-02	2.82E-02	1.29E-02	4.29E-03	1.73E-03				
	COOLED BOTTOMS	NAALCO3OH2	FEIIIHO3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3 2.46E- 04	FE8SEO14 2.73E-06
		7.28E-01	1.18E-01	4.21E-02	3.68E-02	2.67E-02	1.74E-02	8.23E-03	6.72E-03	5.93E-03	5.06E-03	2.75E-03	1.97E-03		
Scenario	Recycle Only, High Si, 5.4M Na Endpoint														
#2		NAALCO3OH2	FEIIIHO3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3 2.48E- 04	FE8SEO14 2.72E-06
	COOLED BOTTOMS	7.27E-01	1.19E-01	4.26E-02	3.74E-02	2.55E-02	1.73E-02	8.22E-03	6.36E-03	6.03E-03	4.97E-03	2.78E-03	2.00E-03		
Scenario	Recycle Only, Low Si, 5.4M Na Endpoint														
#2		NAALCO3OH2	FEIIIHO3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3 2.48E- 04	FE8SEO14 2.73E-06
	COOLED BOTTOMS	7.32E-01	1.19E-01	4.27E-02	3.75E-02	2.04E-02	1.74E-02	8.23E-03	6.37E-03	6.03E-03	4.97E-03	2.78E-03	2.01E-03		

Table A- 4. General Properties from OLI Output for Analyzed Sodium Product Concentrations

	Stream name	total liters	total mass(g)	liquid mass(g)	water mass(g)	insol. solids mass(g)	insol. solids mass fract.	water mass fract.	density(g/L)	pH	viscosity cP	Temp °C
Scenario	Blended Waste/Recycle, High Si, 4.5M Na Endpoint											
#1	ADJUST WASTE											
	FEED	0.100	1.38E+02	1.36E+02	73.134	1.70E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01
	ADJUST											
	RECYCLE	0.358	3.69E+02	3.66E+02	350.112	2.48E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01
	COOLED											
	BOTTOMS	0.235	2.87E+02	2.83E+02	203.566	3.72E+00	0.013	0.710	1.21E+03	10.48	1.84E+00	2.50E+01
Scenario	Blended Waste/Recycle, Low Si, 4.5M Na Endpoint											
#1	ADJUST WASTE											
	FEED	0.100	1.38E+02	1.36E+02	73.135	1.71E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01
	ADJUST											
	RECYCLE	0.358	3.69E+02	3.66E+02	350.112	2.48E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01
	COOLED											
	BOTTOMS	0.235	2.87E+02	2.83E+02	203.517	3.71E+00	0.013	0.710	1.21E+03	10.48	1.84E+00	2.50E+01
Scenario	Recycle Only, High Si, 5.4M Na Endpoint											
#2	ADJUST WASTE											
	FEED	0.222	3.06E+02	3.03E+02	162.521	3.78E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01
	ADJUST											
	RECYCLE	0.780	8.04E+02	7.98E+02	762.705	5.41E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01
	COOLED											
	BLEND	0.433	5.45E+02	5.37E+02	361.355	8.12E+00	0.015	0.663	1.25E+03	10.29	2.18E+00	2.50E+01
Scenario	Recycle Only, Low Si, 5.4M Na Endpoint											
#2	ADJUST WASTE											
	FEED	0.222	3.06E+02	3.03E+02	162.522	3.79E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01
	ADJUST											
	RECYCLE	0.780	8.04E+02	7.98E+02	762.705	5.41E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01
	COOLED											
	BLEND	0.433	5.45E+02	5.37E+02	361.262	8.11E+00	0.015	0.663	1.25E+03	10.29	2.18E+00	2.50E+01

Table A- 5. Species Mass Fraction of Undissolved Solids for Target 6M Sodium Product Concentrations

Stream		NAALCO3OH2	ALOH3	ZRSO42.4H2O	FEIIIPO4.2H2O	NASGEL.15.5H2O	ZNC2O4.2H2O	CAF2	CEPO4	CDC2O4	NIC2O4.2H2O	MGCRO4	FE8SEO14		
RECYCLE		5.86E-01	2.20E-01	7.51E-02	4.61E-02	1.98E-02	1.49E-02	1.42E-02	1.11E-02	6.23E-03	5.53E-03	1.68E-03	4.09E-06		
Scenario	Blended Waste/Recycle, High Si, 6M Na Endpoint														
#1	WASTE FEED	NA3FSO4	FEIIIHOH3	NAALCO3OH2	NAHCO3	CAF2	MNCO3	NA2C2O4	SRCO3	NDPO4.2H2O	CEPO4				
		4.22E-01	2.17E-01	9.98E-02	8.40E-02	7.00E-02	6.00E-02	2.84E-02	1.30E-02	4.31E-03	1.74E-03				
	COOLED BOTTOMS	NAALCO3OH2	FEIIIHOH3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3	FE8SEO14
		7.20E-01	1.17E-01	4.20E-02	3.68E-02	3.54E-02	1.74E-02	8.22E-03	6.67E-03	5.92E-03	4.97E-03	2.78E-03	1.97E-03	2.47E-04	2.73E-06
Scenario	Blended Waste/Recycle, Low Si, 6M Na Endpoint														
#1	WASTE FEED	NA3FSO4	FEIIIHOH3	NAALCO3OH2	NAHCO3	CAF2	MNCO3	NA2C2O4	SRCO3	NDPO4.2H2O	CEPO4				
		4.20E-01	2.16E-01	9.93E-02	8.88E-02	6.97E-02	5.97E-02	2.82E-02	1.29E-02	4.29E-03	1.73E-03				
	COOLED BOTTOMS	NAALCO3OH2	FEIIIHOH3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3	FE8SEO14
		7.25E-01	1.18E-01	4.21E-02	3.68E-02	3.06E-02	1.74E-02	8.23E-03	6.68E-03	5.92E-03	4.98E-03	2.79E-03	1.97E-03	2.48E-04	2.73E-06
Scenario	Recycle Only, High Si, 6M Na Endpoint														
#2	COOLED BOTTOMS	NAALCO3OH2	FEIIIHOH3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3	FE8SEO14
		7.28E-01	1.19E-01	4.26E-02	3.74E-02	2.51E-02	1.73E-02	8.22E-03	6.35E-03	6.03E-03	4.93E-03	2.79E-03	2.00E-03	2.49E-04	2.72E-06
Scenario	Recycle Only, Low Si, 6M Na Endpoint														
#2	COOLED BOTTOMS	NAALCO3OH2	FEIIIHOH3	CAF2	MNCO3	NASGEL.15.5H2O	ZRO2	CEPO4	PBCO3	SRCO3	CDCO3	MGF2	NDPO4.2H2O	CROH3	FE8SEO14
		7.33E-01	1.19E-01	4.27E-02	3.75E-02	2.00E-02	1.74E-02	8.23E-03	6.36E-03	6.03E-03	4.94E-03	2.79E-03	2.01E-03	2.49E-04	2.73E-06

Table A- 6. General Properties from OLI Output for Target 6M Sodium Product Concentrations

	stream name	total liters	total mass(g)	liquid mass(g)	water mass(g)	insol. solids mass(g)	insol. solids mass fract.	water mass fract.	density(g/L)	pH	viscosity	Temp	ionic strength
Scenario	Blended Waste/Recycle, High Si, 6M Na Endpoint												
	ADJST WASTE												
#1	FEED	0.1	1.38E+02	1.36E+02	7.31E+01	1.70E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01	1.08E+01
	ADJST RECYCLE	0.3579986	3.69E+02	3.66E+02	3.50E+02	2.48E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01	5.86E-01
	COOLED												
	BOTTOMS	0.1764873	2.27E+02	2.23E+02	1.44E+02	3.72E+00	0.016	0.634	1.27E+03	10.58	2.45E+00	2.50E+01	6.92E+00
Scenario	Blended Waste/Recycle, Low Si, 6M Na Endpoint												
	ADJST WASTE												
#1	FEED	0.1	1.38E+02	1.36E+02	7.31E+01	1.71E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01	1.08E+01
	ADJST RECYCLE	0.3579986	3.69E+02	3.66E+02	3.50E+02	2.48E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01	5.86E-01
	COOLED												
	BOTTOMS	0.176441	2.26E+02	2.23E+02	1.44E+02	3.72E+00	0.016	0.634	1.27E+03	10.58	2.45E+00	2.50E+01	6.92E+00
Scenario	Recycle Only, High Si, 6M Na Endpoint												
	ADJST WASTE												
#2	FEED	0.2222221	3.06E+02	3.03E+02	1.63E+02	3.78E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01	1.08E+01
	ADJST RECYCLE	0.779886	8.04E+02	7.98E+02	7.63E+02	5.41E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01	5.86E-01
	COOLED												
	BLEND	0.3898037	5.00E+02	4.92E+02	3.17E+02	8.12E+00	0.016	0.633	1.27E+03	10.26	2.44E+00	2.50E+01	6.90E+00
Scenario	Recycle Only, Low Si, 6M Na Endpoint												
	ADJST WASTE												
#2	FEED	0.2222222	3.06E+02	3.03E+02	1.63E+02	3.79E+00	0.012	0.531	1.37E+03	9.72	4.00E+00	2.50E+01	1.08E+01
	ADJST RECYCLE	0.779886	8.04E+02	7.98E+02	7.63E+02	5.41E+00	0.007	0.949	1.03E+03	5.95	9.23E-01	2.50E+01	5.86E-01
	COOLED												
	BLEND	0.3896999	5.00E+02	4.92E+02	3.17E+02	8.11E+00	0.016	0.633	1.27E+03	10.26	2.44E+00	2.50E+01	6.90E+00

Table A- 7. Comparison of Modeled Properties versus Experimentally Measured Values

	Parameter	Model Predictions	Measured Values
Scenario #1, High Si, 4.6M Sodium Endpoint	Insoluble Solids (wt%)	1.3	> 0.132
	Insoluble Solids	NaAlCO ₃ (H ₂ O) Fe(OH) ₃ CaF ₂ MnCO ₃ NaSGel(H ₂ O) ZrO ₂	Fe ₂ O ₃ SiO ₂
	pH	10.48	13
	Density (g/mL)	1.21	1.27
Scenario #1, Low Si, 4.6M Sodium Endpoint	Insoluble Solids (wt%)	1.3	> 0.132
	Insoluble Solids	NaAlCO ₃ (H ₂ O) Fe(OH) ₃ CaF ₂ MnCO ₃ NaSGel(H ₂ O) ZrO ₂	Fe ₂ O ₃ SiO ₂
	pH	10.48	13
	Density (g/mL)	1.21	1.27
Scenario #2, High Si, 5.4M Sodium Endpoint	Insoluble Solids (wt%)	1.5	> 0.128
	Insoluble Solids	NaAlCO ₃ (H ₂ O) Fe(OH) ₃ CaF ₂ MnCO ₃ NaSGel(H ₂ O) ZrO ₂	Fe ₂ O ₃ SiO ₂
	pH	10.29	13
	Density (g/mL)	1.25	1.3
Scenario #2, Low Si, 5.4M Sodium Endpoint	Insoluble Solids (wt%)	1.5	> 0.128
	Insoluble Solids	NaAlCO ₃ (H ₂ O) Fe(OH) ₃ CaF ₂ MnCO ₃ NaSGel(H ₂ O) ZrO ₂	Fe ₂ O ₃ SiO ₂
	pH	10.29	13
	Density (g/mL)	1.25	1.3