Phase I and II Results from Sr and TRU Precipitation Tests

by

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Phase 1 and 2 Results from Sr/TRU Precipitation Tests

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1.0 Summary

The BNFL removal processes for strontium and transuranic components from the AN-102 and AN-107 supernate originally proposed are co-precipitation methods. In initial testing, the precipitates formed during the strontium and ferric nitrate additions were not filterable. Two series of statistically designed tests examined over a dozen variables that possibly could affect the filterability of the precipitate and the associated decontamination of the species of interest. The conclusions from these tests are:

- Filterability is influenced by the amount of ferric ion added to co-precipitate the actinide elements and the amount of at least one organic component in the gluconate grouping (gluconate, glycolate and citrate).
- Hydroxide ion concentration did not affect filterability.
- Filter fluxes in sheared environments with ferric ion present are too low for cross flow filtration to be successful. Investigations are currently focused on alternative precipitating agents.
- The sodium ion concentration and level of organic complexants influence decontamination of the supernate.
- Decontamination factors measured for americium using maximum iron nitrate addition were not sufficient to assure required decontamination.
- Testing with real waste (AN-102) showed higher filterability than previous work along with equivalent decontamination.
- Initial simulant composition included higher sodium gluconate levels than real waste based on iron chemistry. Test results indicate that iron and gluconate form a soluble complex with a 1:1 stoichiometry.

2.0 Introduction

Strontium and the actinide elements U, Pu and Am are present in the High Level Liquid Waste in the Hanford tanks AN-102 and AN-107 due to the presence of complexing agents used in the processes within B Plant.¹ The mission of B Plant was to recover Sr-90 from Purex Acidified Sludge (PAS). Hydroxyethylene diamine triacetic acid (HEDTA), ethylene diamine tetraacetic acid (EDTA), and citric acid were added to complex many of the di- and trivalent metals in order to prevent their extraction from the

aqueous phase into the organic solvent. The tributylphosphate-normal paraffin hydrocarbon solvent was then washed in several stages before recycle. Dilute glycolic acid was used to wash sodium from the solvent. After nitric acid washes to remove desired product the organic phase was washed with sodium gluconate and excess hydroxide to remove yttrium and uranium.

The proposed BNFL removal processes for strontium and transuranic components from the AN-102 and AN-107 supernate are co-precipitation methods. In Part A, tests were performed to decontaminate real waste samples using natural strontium nitrate in an isotopic dilution and ferric nitrate to co-precipitate the actinides.²⁻⁵ This work was based on earlier investigations by Herting⁶ and Orth, *et al.*⁷ In general, the results of the experiments indicated a successful decontamination for Sr-90 and the actinides. However, the liquid/solid separation was accomplished by centrifugation. The resulting precipitates could not be filtered. In scoping tests, Nash⁸ found that precipitate filterability improved if strontium was added before iron.

The objective of this work was to examine several factors that potentially influence filtration of the Sr/TRU precipitate. The research was performed using statistically designed experiments in a three-phased approach. Variables in the first non-radioactive phase included temperature, sodium ion concentration, hydroxide ion concentration, strontium concentration and total amount, ferric ion concentration and method of addition. Organic components in Envelope C are the complexants that increase the solubility of the strontium and that of the actinides and are thought to alter the filterability of the Sr/TRU precipitate. A second set of statistically defined experiments examined the influences of each of the organic components in Envelope C simulant spiked with Sr-85, Pu-239 and Am-241 tracers. The results of these phases of tests are reported. The third phase of statistically designed experiments examined permanganate as a precipitation reagent for the transuranic elements instead of ferric ion. A separate report will address the results from the third phase of experiments.

3.0 Experimental

3.1 Phase 1: Screening Tests to Determine Variables that Influence Filterability

Phase 1 examined several variables that possibly effect the filterability of the Sr/TRU precipitate produced from the AN-107 simulant (See composition in Appendix 1).⁹ The nine factors investigated were: sodium ion, hydroxide ion, and aluminum concentrations; temperature; the total amount of strontium added to the system; the strontium ion concentration of the reagent solution; the total amount of iron added to the system; the iron concentration of reagent solution; and method of addition. The sodium, hydroxide and aluminum concentrations spanned the AN-102 and AN-107 tank chemistries. Typical concentrations of the organic complexants were used. The organic complexants were ethylenediaminetetraacetic acid (EDTA), citric acid, glycolic acid, N-(2-hydroxyethyl)ethylene ethylenediaminetetraacetic acid (HEDTA), and iminodiacetic acid

(IDA). Lanthanum, cerium and neodymium were used as surrogates for plutonium and americium.

Approximately 50 mL of the simulant was placed into a 200-mL beaker. The simulant had been adjusted to the hydroxide and aluminum concentration specified in Table 1. The solution was stirred with a magnetic stirrer and then heated to the required temperature in a water bath on a heater/stirrer plate. An aliquot of the precipitating agent (strontium nitrate or ferric nitrate) was delivered to the beaker using a Kd syringe pump, model 100, at a delivery rate of 0.5 mL/min.¹⁰ The precipitate slurry was stirred for 15 minutes at temperature prior to the addition of the second precipitating agent. After the second addition, the slurry was stirred for 15 minutes, allowed to cool to room temperature and filtered through a 0.45-micron filter. The filtrate was collected for 1 minute and weighed or volumetrically measured. The time intervals used in these experiments are shorter than those used during the previous work. However, sufficient decontamination should occur during the first 15 minutes to allow for analysis and efficient completion of the tests. After the samples have cooled, the remaining slurry was filtered. Samples were submitted to the Analytical Development Section of SRTC for analyses.¹¹ These analyses included elemental analysis by Inductively Coupled Plasma – Emission spectroscopy,¹² Inductively Coupled Plasma – Mass Spectrometry,¹³ gamma spectroscopy,¹⁴ liquid scintillation analysis for strontium-90,¹⁵ and alpha pulse height analysis.¹⁶ Shown in Figure 1 is the experimental setup used in the real waste tests. This experimental protocol was used in each phase of Sr/TRU precipitation testing.

Figure 1. Experimental Apparatus for Sr/TRU Precipitation

The objective of Phase 1 was to determine the primary variables that influence filterability. JMP¹⁷ was used to generate the fractional factorial design that appears in Table 1. Software validation¹⁸ was performed on the JMP software. The statistical design allowed for all primary variables to be found. Filterability is the primary response variable. However, other responses are important to the overall Hanford flowsheet. Included in these are the fates of sulfate, carbon and aluminate. Analysis of the filtrate was performed to measure these species. The amount in the precipitate was calculated by difference.

The aliasing structure for these trials indicated that most of these 9 main effects are confounded with two-way interaction terms in this screening experiment. The distinction between a significant main effect and a possible two-way interaction will be facilitated by the Phase 2 tests.

As indicated in Table 1, a center point was run as part of each day's processing with the method of iron addition being alternated between "drop" and "needle." A minimum of 2 center points was performed, one by each method of iron addition. "Drop" refers to dropwise addition of reagent and "needle" refers to the use of a fine hypodermic needle with its tip submerged in the agitated solution. Needle would introduce a fine thread of reagent into the mixture in contrast to the dropwise method.

Table 1: 30-Beaker Test

Pattern	Na+ (M)	OH- (M)	Al (M)	Temp (Degrees C)	Sr (total) (M)	Sr (conc) (M)	Fe (total) (M)	Fe (conc) (M)	Method of Iron Addition	Experimental Order
-----+	5.5	0.2	0.05	35	2	0.025	1	0.075	Drop	14
-----+	5.5	0.2	0.05	75	1	0.075	2	0.025	Needle	9
-----+	5.5	0.2	0.3	35	1	0.075	2	0.025	Drop	10
-----+	5.5	0.2	0.3	75	2	0.025	1	0.075	Needle	8
-----+	5.5	1	0.05	35	1	0.075	1	0.075	Needle	6
-----+	5.5	1	0.05	75	2	0.025	2	0.025	Drop	1
-----+	5.5	1	0.3	35	2	0.025	2	0.025	Needle	7
-----+	5.5	1	0.3	75	1	0.075	1	0.075	Drop	4
-----+	7	0.2	0.05	35	1	0.025	2	0.075	Needle	11
-----+	7	0.2	0.05	75	2	0.075	1	0.025	Drop	12
-----+	7	0.2	0.3	35	2	0.075	1	0.025	Needle	3
-----+	7	0.2	0.3	75	1	0.025	2	0.075	Drop	2
-----+	7	1	0.05	35	2	0.075	2	0.075	Drop	15
-----+	7	1	0.05	75	1	0.025	1	0.025	Needle	5
-----+	7	1	0.3	35	1	0.025	1	0.025	Drop	16
-----+	7	1	0.3	75	2	0.075	2	0.075	Needle	13

Pattern	Na+ (M)	OH- (M)	Al (M)	Temp (Degree C)	Sr (total) (M)	Sr (conc) (M)	Fe (total) (M)	Fe (conc) (M)	Method of Iron Addition	Experimental Order
0000000-	6.25	0.6	0.175	55	1.5	0.050	1.5	0.05	Drop	Every other day
0000000+	6.25	0.6	0.175	55	1.5	0.050	1.5	0.05	Needle	Every other day

3.2 Phase 1B: Revision to AN-107 Simulant Composition

SRTC personnel reviewed Phase 1 results with the BNFL technical representative during December 1998. Data review indicated that the gluconate level in the simulant was too high. As a result of these discussion three small sets of tests will be conducted prior to the actual start of the 25-Beaker tests. These three sets of tests are:

- Iron Titration – The objective of this test was to determine the stoichiometry of the Fe-gluconate complex. In this test, ferric nitrate was incrementally added to a fixed gluconate solution. The amount of ferric ion in solution was measured by ICP-ES.
- An initial grouping of organic complexing agents for the 25-Beaker tests was recommended. This grouping is shown in Table 2. After the Fe titration experiment provided the stoichiometry of the Fe-gluconate complex, the simulant was revised. Single effects tests were performed on the nominal simulant. Both filterability and DF were measured.
- A series of confirmatory tests was performed using the revised simulant recipe. The objective of these tests was to span the variables which were shown to be statistically significant. Filterability and a decontamination factor based on La, Ce, and Nd were measured.
- Test the sample of AN-102 for filterability and decontamination. A set of three test was performed to include: a test with the precipitating reagent in the method used in Part A; a test reversing the order of addition; and a test in which the precipitate formed with the order reversed followed by shearing in a blender. These tests were conducted to determine if the model used to predict filterability from the AN-107 simulant would be valid for the AN-102 radioactive waste.

3.3 Phase 2: Influence of Organic Complexation and Filterability

The BNFL technical representative approved the Phase 2 matrix before SRTC personnel proceeded with testing. The objective of Phase 2 was to determine the influence of each of the organic complexants has on the filterability of the Sr/TRU precipitate. The levels for each primary variable were the same as in Phase 1. The coded levels (1, 0, -1) of organic complexants were established to span the range of their concentrations measured in the actual waste (AN-102 and AN-107).

Table 2: Experimental Design for 25-Beaker Test

Pattern	Na	Al	OH	Fe Conc.	Gluconate Citrate Glycolate	EDTA/HEDTA/IDA/NTA
-----	-1	-1	-1	-1	-1	-1
---++++-	-1	-1	-1	1	1	1
--+++++	-1	-1	1	-1	-1	1
-++---++	-1	-1	1	1	1	-1
-++++++	-1	1	-1	-1	1	-1
-++++++	-1	1	-1	1	-1	1
-++---++	-1	1	1	-1	1	1
-++++--	-1	1	1	1	-1	-1
+----+++	1	-1	-1	-1	1	1
+---+++	1	-1	-1	1	-1	-1
++---+-	1	-1	1	-1	1	-1
+++++-	1	-1	1	1	-1	1
+++++-	1	1	-1	-1	-1	1
++---+-	1	1	-1	1	1	-1
+++----+	1	1	1	-1	-1	-1
+++++++	1	1	1	1	1	1

Organic levels based on 8.7 M sodium ion in starting salt solution concentrations were:

Level	Organic Group	Concentration (M)
-1	EDTA	0.0075
	HEDTA	0.0078
	NTA	0.0
	IDA	0.0375
	Glycolate	0.07
	Citrate	0.0128
	Gluconate	0.0
0	EDTA	0.022
	HEDTA	0.023
	NTA	0.004
	IDA	0.113
	Glycolate	0.21
	Citrate	0.038
	Gluconate	0.009
1	EDTA	0.037
	HEDTA	0.039
	NTA	0.0073
	IDA	0.188
	Glycolate	0.35
	Citrate	0.064
	Gluconate	0.018

Iron concentration levels were 1 (0.1M), 0 (0.06 M), -1 (0.02 M). Additionally, a test was conducted with an iron concentration of 0.075 M. Tests were conducted at 50 °C. Plutonium-239 and americium-241 were spiked into the simulated salt solutions. The gamma-emitting Sr-85 was used instead of Sr-90. As in Phase 1, filterability is the primary response. Laboratory notebook WSRC-NB-97-63 contains data obtained during these tests and the procedures used. Personnel used routine analytical protocol for the samples in this report.¹¹

4.0 Discussion

4.1 Phase 1 (30 Beaker Tests)

In addition to the experimental matrix, Table 3 contains the filtration results measured in the first phase of testing. The filtrate flux is provided in terms of weight of filtrate (g) per second of filtration time. As mentioned in the experimental section, the filtrate was collected for the first minute of filtration and then weighed. In all experiments, the Sr/TRU precipitate formed by stepwise addition of strontium nitrate and ferric nitrate was filterable. The filtrate flux ranged from 0.18 to 2.6 g/s. The filtrates were very highly colored being very dark red.

Table 3. Filterability Data from Phase 1 Tests

Pattern	Na+ (M)	OH- (M)	Al (M)	Temp (°C)	Sr (total) (M)	Sr (conc) (M)	Fe (total) (M)	Fe (conc) (M)	Method of Iron Addition	Exp. Order	Filtrate Flux g/s
-----+	5.5	0.2	0.05	35	2	0.025	1	0.075	Drop	14	1.6409
-----+	5.5	0.2	0.05	75	1	0.075	2	0.025	Needle	9	1.3552
-----+	5.5	0.2	0.3	35	1	0.075	2	0.025	Drop	10	0.1799
-----+	5.5	0.2	0.3	75	2	0.025	1	0.075	Needle	8	0.1872
-----+	5.5	1	0.05	35	1	0.075	1	0.075	Needle	6	0.6269
-----+	5.5	1	0.05	75	2	0.025	2	0.025	Drop	1	0.5607
-----+	5.5	1	0.3	35	2	0.025	2	0.025	Needle	7	2.6071
-----+	5.5	1	0.3	75	1	0.075	1	0.075	Drop	4	0.2727
-----+	7	0.2	0.05	35	1	0.025	2	0.075	Needle	11	0.9539
-----+	7	0.2	0.05	75	2	0.075	1	0.025	Drop	12	0.8984
-----+	7	0.2	0.3	35	2	0.075	1	0.025	Needle	3	0.3007
-----+	7	0.2	0.3	75	1	0.025	2	0.075	Drop	2	0.3254
-----+	7	1	0.05	35	2	0.075	2	0.075	Drop	15	0.317
-----+	7	1	0.05	75	1	0.025	1	0.025	Needle	5	0.8098
-----+	7	1	0.3	35	1	0.025	1	0.025	Drop	16	1.6904
-----+	7	1	0.3	75	2	0.075	2	0.075	Needle	13	0.8235
0000000-	6.25	0.6	0.175	55	1.5	0.05	1.5	0.05	Drop	Every other day	1.5128
0000000+	6.25	0.6	0.175	55	1.5	0.05	1.5	0.05	Needle	Every other day	1.877

The results from the statistical analysis of the filtration data are provided in Table 4. The JMP Statistical program, version 3.2.2 was used to fit the filtrate flux data to a linear function of the nine variables. The values shown in Table 4 are the model estimates (coefficients) of the linear response model, the standard errors of the estimates, the statistical t ratio's, and the significance levels (i.e., $\text{Prob} > |t|$). For a parameter to be statistically significant, the significance level should be less than or equal to 0.05. The results from the filtration data from the Phase 1 tests show that no parameter studied was statistically significant for filterability. It had been thought that the bulk iron or strontium concentration would affect the filterability of the Sr/TRU precipitate. However, none of the nine parameters tested were significant at 95 % confidence level. Actually, none of the parameters were close to being significant with the feed strontium concentration ($[\text{Sr}]_{\text{conc}}$) significant only at slightly greater than 75 %. In addition, the precipitates from these tests were predominantly white to cream colored. This color indicates that Fe is not participating in the precipitation step. The lack of iron in the precipitate along with lower than expected lanthanide decontamination were factors that lead to the post Phase 1 tests discussed earlier.

Table 4. Statistical Analysis Results from Filtration Data in Phase 1 Tests

Term	Estimate	Std Error	t Ratio	Prob > t
Intercept	2.61	2.04	1.28	0.231
Na+	-0.11	0.26	-0.42	0.685
OH-	0.29	0.49	0.60	0.566
Al	-0.39	1.57	-0.25	0.810
Temp	-0.01	0.01	-0.98	0.351
Sr(total)	0.14	0.39	0.36	0.729
Sr(conc)	-10.0	7.83	-1.28	0.234
Fe(total)	0.09	0.39	0.22	0.829
Fe(conc)	-8.14	7.83	-1.0	0.326

The soluble lanthanide (La, Ce, and Nd) concentrations were measured by ICP-ES in the initial simulant at nominally 8.8 M sodium ion concentration and in the filtrate following the chemical adjustments (Na, Al, and OH) and the addition of the precipitating agents Sr and Fe nitrates. The decontamination factor for each lanthanide was computed and the factors are shown in Table 5. Decontamination factors ranged from essentially no decontamination (DF = 1.1) to 2.7.

Table 5. Lanthanide Concentrations (mg/L) measured in Phase 1 Tests

La	La DF	Ce	Ce DF	Nd	Nd DF
21.1	1.7	41.19	1.6	20.39	1.6
19.73	1.8	46.97	1.4	19.17	1.7
16.17	2.2	32.63	2.1	15.61	2.1
18.63	1.9	39.49	1.7	18.49	1.8
16.35	2.2	33.79	2.0	16.73	2.0
19.36	1.9	38.77	1.7	20.05	1.7
13.59	2.7	26.2	2.6	13.19	2.5
13.86	2.6	27.94	2.4	13.53	2.5
24.95	1.4	44.28	1.5	21.49	1.5
26.86	1.3	55.9	1.2	26.16	1.3
22.07	1.6	43.63	1.5	21.63	1.5
26.02	1.4	51.42	1.3	23.86	1.4
21.98	1.6	42.58	1.6	21.61	1.5
29.6	1.2	59.09	1.1	28.54	1.2
15.05	2.4	30.47	2.2	13.61	2.4
18.71	1.9	39.29	1.7	18.2	1.8
20.88	1.7	40.3	1.7	20.46	1.6
21.84	1.7	40	1.7	19.97	1.7
36.08	original	67.11	Original	33.17	original

Statistical analysis of the lanthanide decontamination factors versus the input variables (sodium level, hydroxide ion concentration, etc.) revealed three variables that were statistically significant for the La, Nd, and Ce DF with an additional variable near the 5 % significance level. Sodium level, hydroxide ion concentration, and aluminum concentration proved significant for each of the lanthanide elements and temperature was nearly significant.

Figure 2 contains the graphical representations of the computer fit of a linear response model for the decontamination factors for neodymium. Sodium level and temperature exhibit negative relationships with the DF. Increased sodium level equates to increased ion strength of the solution. In these high salt solutions, it can become very difficult to hydrolyze the lanthanide ion from the organic chelant species. The temperature effect in the range of 35 to 75 °C is very minimal and can be neglected. Estimates of the coefficients are shown in Table 6.

Table 6. Statistical Analysis Results from Nd DFs in Phase 1 Tests

Term	Estimate	Std Error	t Ratio	Prob > t
Intercept	3.44	0.60	5.77	0.0004
Na+	-0.275	0.076	-3.60	0.007
OH-	0.422	0.14	2.94	0.019
Al	1.75	0.46	3.81	0.005
Temp	-0.005	0.003	-1.85	0.010
Sr(total)	-0.135	0.115	-1.20	0.265
Sr(conc)	0.75	2.29	0.33	0.75
Fe(total)	-0.013	0.0115	-0.11	0.92
Fe(conc)	-0.75	2.29	-0.33	0.75

Figure 2. Neodymium DF as a Function of Sodium Level

Table 7 contains the aluminum concentrations measured in the post-precipitation liquid phase. Aluminum losses to the precipitate were very low. One sample, however, did exhibit a 10 % reduction in aluminum concentration after precipitation. The sulfate level in 5 samples of the filtered solids was determined by ion chromatography. All solid samples exhibited measured concentrations of sulfate below the detection limit (20 mg/L). Total organic carbon measurements were obtained for the feed filtrate along with

the post-precipitation filtrate from each beaker test. The data had very large fluctuations due to the large organic concentrations in the AN-107 simulant and were not meaningful.

Table 7. Al Concentrations (mg/L) in Phase 1 Tests

Al Measure after ppt	Al As prepared	Al % in Liq after ppt
1534.0	1575.7	97.4
2052.0	1575.7	130.2
8257.0	8279.8	99.7
10640.0	8279.8	128.5
1507.0	1535.5	98.1
2085.0	1535.5	135.8
9621.0	8229.7	116.9
8253.0	8229.7	100.3
1665.0	1645.6	101.2
1966.0	1645.6	119.5
7518.0	8330.8	90.2
8854.0	8330.8	106.3
1596.0	1614.5	98.9
1956.0	1614.5	121.2
7738.0	8301.0	93.2
10250.0	8301.0	123.5
5149.0	4965.4	103.7
4938.0	4965.4	99.4

4.2 Fe Titration

With the results from the Phase 1 tests showing no variables affecting filterability, the simulant composition was determined questionable. In particular, the concentration of sodium gluconate, a known complexing agent for Fe (III), was suspected because it was based on process knowledge from the Pacific Northwest National Laboratory rather than analytical measurement. Therefore, experiments were performed to determine the maximum loading of the simulant for Fe (III) ions. The results of this iron titration are shown in Figure 3.

Figure 3. Fe Titration of AN-107 Simulant

For each addition of an aliquot of a 2 M Fe (III) nitrate solution, the measured iron concentration increased incrementally until the total added iron concentration was 0.1 M. The slope of the fitted line is 1 suggesting a 1:1 Fe:gluconate stoichiometry to the organometallic complex. The iron concentration in the real AN-107 waste is known to be 0.036 M. This titration data indicate the gluconate level should also be 0.036 M rather than the 0.2 M concentration currently used in the simulant. Because there exists a possibility that iron could exist as a 2:1 complex with gluconate, a simulant was prepared that contained sodium gluconate at 0.018 M concentration and a second iron titration was performed. The results are shown in Figure 4. As observed by McCabe² in previous work, the iron concentration in the simulant decreased as ferric nitrate was added to the simulant. A red precipitate (FeOOH) was observed in each of the samples.

Figure 4. Fe Titration of Revised AN-107 Simulant

4.3 Confirmatory Tests

With the change to the gluconate concentration (0.018 M) in the simulant formulation for the AN-107 tank, a set of confirmatory tests was conducted. The experimental matrix for

this set of tests along with the measured filtrate flux is shown in Table 8. Several variables that had not shown to be influencing the decontamination process were deleted. Although temperature was a significant variable, the magnitude of the effect was negligible. Elemental analysis of the simulated supernate was performed pre- and post-precipitation. The resulting data were regressed using the JMP statistical analysis software. Figure 5 shows photographs of the starting filtrate, precipitation filtrate and the precipitate solids. The filtrate remains very dark in color after the precipitation. The precipitates are reddish to brown in color, indicating the presence of iron in the precipitate.

Table 8. Experimental Matrix for Confirmatory Tests

Test No.	Pattern	Na+ (M)	OH- (M)	Al (M)	Sr (M)	Fe (M)	Filtrate Flux (g/s)
1	----+	5.5	0.4	0.05	0.025	0.075	0.083
2	--++-	5.5	0.4	0.3	0.075	0.025	0.128
3	-+-+-	5.5	1	0.05	0.075	0.025	0.186
4	---++	5.5	1	0.3	0.025	0.075	0.072
5	+---+	7	0.4	0.05	0.075	0.075	0.072
6	++--	7	0.4	0.3	0.025	0.025	0.128
7	+++--	7	1	0.05	0.025	0.025	0.114
8	+++++	7	1	0.3	0.075	0.075	0.061
9	ooooo	6.25	0.7	0.175	0.05	0.05	0.096

Figure 5. AN-102 Simulate Supernate, Sr/TRU Filtrate and Sr/TRU Precipitate

JMP® statistical analysis program was used to analyze the filtration data. Table 9 contains the results of the statistical analysis. As expected, the total iron concentration

added to precipitate the lanthanide ions was statistically significant and a plot of this effect is shown in Figure 6. However, iron concentration was the only significant variable. Ionic strength (sodium level) and hydroxide ion concentration did not affect filterability. It had been previously believed that increased ionic strength (sodium level) would decrease the filterability and increased hydroxide ion concentration would increase filterability.

Table 9. Statistical Analysis of the Filtration Data from Confirmatory Tests

Term	Estimate	Std Error	t Ratio	Prob > t
Intercept	15.8	4.1	3.83	0.03
Na + (M)	-0.94	0.6	-1.55	0.22
OH- (M)	0.42	1.13	0.37	0.73
Al (M)	-3.9	3.6	-1.07	0.36
Sr conc (M)	15.4	18.1	0.85	0.46
Fe conc (M)	-80.2	18.1	-4.42	0.02

Figure 6. Filtrate Mass versus Iron Concentration in Confirmatory Tests

Table 10 contains the lanthanide concentrations measured in the precipitation tests with the revised AN-107 simulant. The process decontamination factors (DFs) include the ionic strength and caustic adjustments. The DFs for the lanthanide elements ranged from 2 – 7.2, 2 – 6.3 and 1.8 – 6 for lanthanum, cerium and neodymium, respectively. This

trend of lower DFs for Nd compared to La has been observed in several scoping studies performed at SRTC and may be indicative of the increased charge density on the Nd ion.

Table 10. Lanthanide Concentrations (mg/L) in Confirmatory Tests

Test No.	Initial			Post			DF*		
	La	Ce	Nd	La	Ce	Nd	La	Ce	Nd
1	23.02	22.5	42.76	7.167	7.135	18.03	5.3	5.4	3.8
2	18.83	18.72	34.21	6.246	6.589	19.73	6.1	5.8	3.5
3	21.44	19.73	37.88	10.18	11.46	30.82	3.7	3.3	2.2
4	27.29	14	15.07	5.288	6.091	15.32	7.2	6.3	4.5
5	29.27	27.76	51.95	9.25	9.077	23.59	4.1	4.2	2.9
6	17.08	1.475	26.52	17.6	18.1	38.89	2.2	2.1	1.8
7	24.92	23.88	45.54	17.32	19.01	39.01	2.2	2.0	1.8
8	32.93	19.18	17.15	8.184	8.933	23.4	4.6	4.3	3.0
9**	22.33	38.3	21.15	10.91	28.1	11.63	3.5	1.4	6.0
Stock	38.01	38.2	69.26						

*DF = [stock]/[post]

**Test 9 was a mid point test

Statistical analysis of the DF data is provided in Table 11. There are some differences between results for each of the lanthanide elements; however, the effect of each of the variables is the same. In the lanthanum results, sodium level was statistically significant at a 5 % significance level and iron concentration was nearly significant. The magnitude of the effect for each of these variables is shown in Figure 7 and was as expected. Sodium ion increases resulted in a slight decrease in the DF (estimate of slope of DF vs [Na] = -1.5). An increase in iron concentration showed a large increase in the DF (estimate of DF vs [Fe] = 35). Additionally, hydroxide ion did not have an effect on the DF obtained for lanthanum, neodymium, or cerium.

The results for cerium and neodymium are not statistically significant but the estimates from the fitted models support the same conclusion. In the results for cerium DF, there were no statistically significant variables. However, the magnitudes of the estimates are nearly identical to those for lanthanum. In the neodymium results, there are no significant effects and the estimates are slightly different but of the same direction. This delta for neodymium may be indicative of its higher charge density due to its smaller size.

The results of these tests indicate that successful decontamination of the actinide surrogates (La, Ce, and Nd) can be obtained. The optimum conditions would be to utilize

high concentrations of ferric nitrate (0.075 M) and with low sodium ion concentration (5.5 M). These results agree with those obtained with radioactive samples of AN-107 performed by McCabe.⁵

Table 11. Statistical Analysis Result from the Confirmatory Tests

Term	Estimate	Std Error	t Ratio	Prob > t
Lanthanum				
Intercept	10.9	3.0	3.59	0.037
Na + (M)	-1.5	0.45	-3.43	0.041
OH- (M)	3.4e-17	0.84	0.00	1.0
Al (M)	4.8	2.7	1.79	0.17
Sr conc (M)	8	13.4	0.60	0.59
Fe conc (M)	35	13.4	2.61	0.08
Cerium				
Intercept	9.9	5.4	1.83	0.16
Na + (M)	-1.4	0.8	-1.73	0.18
OH- (M)	-0.5	1.5	-0.34	0.76
Al (M)	3.6	4.7	0.76	0.5
Sr conc (M)	9	23.7	0.38	0.73
Fe conc (M)	35	23.7	1.48	0.24
Neodymium				
Intercept	6.5	5.5	1.19	0.32
Na + (M)	-0.75	0.8	-0.93	0.42
OH- (M)	-0.16	1.5	-0.10	0.92
Al (M)	2.1	4.8	0.43	0.69
Sr conc (M)	-1.5	24.3	-0.06	0.95
Fe conc (M)	24.5	24.3	1.01	0.39

Figure 7. Sodium and Iron Statistical Plots for DF in Confirmatory Tests

4.4 Organic Grouping Single Effects Tests

The purpose of the Phase 2 tests was to further investigate the variables shown in Phase 1 tests to affect filterability and DF and to estimate the effects of the various organic complexing agents. In order to minimize the number of variables, the organic complexing agents were grouped into three groupings based on their chemistries. The EDTA group was comprised of EDTA, HEDTA, NTA, and IDA. These complexing agents are derivatives of the same function group and typically target divalent cations. Gluconate was placed into a separate group because of its affinity for the trivalent iron species. Thus, the last group of organic complexing agents contained glycolate and citrate. Three scoping tests were performed to determine the magnitude of the effects these groups had on the filterability. The test conditions were: 5.5 M Na, 1.0 M OH, 0.4 M Al, 0.075 M Sr and Fe and 50 °C. Shown in Table 12 are the results of Sr/TRU precipitation tests with the three organic complexant groups. Also included is the result from the AN-107 simulant. The data are displayed in Figure 8.

Table 12. Organic Group Single Effects Tests

	Filtrate Mass (g)	Filtrate Flux (g/s)	Flux (gpm/ft ²)
EDTA/HEDTA/NTA/IDA	3.795	0.063	0.038
Glycolate/Citrate	14.649	0.244	0.148
Gluconate	17.541	0.292	0.177
AN-107 Simulant	3.685	0.061	0.037

The filtrate flux measured in the three experiments ranged from 0.063 to 0.292 g/s compared to the flux of 0.061 g/s measured for the AN-107 simulant. The precipitate from the glycolate and citrate group simulant filtered nearly as well as the precipitate from the gluconate-containing simulant. These results agree with the low complexing ability of citrate and glycolate. The results from the gluconate test agree with the results from the Phase 1 tests previously reported. The EDTA/HEDTA/NTA/IDA-containing simulant filtered very poorly almost identically to the AN-107 simulant containing all of the organic complexing agents. These results suggest incorporation of some organic material into the Sr/Fe precipitate. This organic material could help blind the filter lowering the filtrate flux. With these results, the seven organic complexants have been separated into two groups: group 1 consisting of EDTA/HEDTA/NTA/IDA and group 2 consisting of Glycolate/Citrate/Gluconate.

Figure 8. Filtrate Flux Results from Organic Grouping Tests

4.5 Real Waste Scoping Tests

In order to test the filterability of the sample of AN-102 supernate and compare this to the AN-107 simulant filterability results, three Sr/TRU precipitation tests were performed. In the first test, ferric nitrate was added to a 0.75 M concentration followed by the addition of strontium nitrate to the same concentration. This is the order of addition used in previous testing⁸ that proved un-filterable. The second test reversed the order of addition (Sr followed by Fe) that was used in the other tests reported herein. Lastly, a test was performed which examined the effect of shear on the filterability of the Sr/TRU precipitate formed by strontium addition followed by iron addition.

Figure 9. Filtration Data from Real Waste Tests with AN-102 Samples

The filtration data from the real waste scoping tests are shown in Figure 9. The measured filtrate mass from the first and second tests exceed the model developed for the AN-107 simulant. The reverse order of addition of the precipitating agents caused an increase in the amount of filtrate. The third test examined the effect of shear on the filterability of the Sr/TRU precipitate. It was expected that the filtrate flux would be reduced due to the shearing forces in the blender. The filtrate flux was reduced from 0.3 g/s to 0.07 g/s. The filtration performance of this sample of AN-102 supernate is greatly increased over that of the previous sample during Part A.²

While the filterability of the AN-102 sample was very encouraging, the results from the Sr-90 and actinide analyses were not. Shown in Table 13 are the measured decontamination factors (DFs) for the radionuclides of interest. McCabe² had previously measured DFs in a sample from AN-102.

Table 13. DFs from Real Waste Tests of AN-102

Test	Sr-90	Pu-239	Am-241
1	31	1.03	1.3
2	37	1.5	2.1
3	40	1.4	2.1
Required DF	6	1.0	1.25
McCabe ²	200	1.6	1.9 – 2.1

The data from both sets of experiments agree fairly well with the exception of the DF for Sr-90. McCabe added strontium to a concentration of 0.1 M while the current tests utilized 0.075 M strontium. Other differences between the experimental conditions are the current tests used a 6.8 M sodium ion concentration prior to the hydroxide adjustment, and McCabe started with a 5.5 M sodium ion concentration. McCabe reported that the resulting americium concentration (0.032 $\mu\text{Ci/mL}$) in the supernate was below the acceptance limit; whereas, the Am concentration (0.075 $\mu\text{Ci/mL}$) is above the limit (100 nCi/g) for the tests reported herein.

4.6 Phase 2 (25 Beaker) Tests

In the Phase 2 tests, simulated AN-107 waste was spiked with Sr-85, Am-241 and Pu-239 radio-tracers on the order of 10^5 dpm/mL. Various levels of organic complexing agents were added according to the experimental matrix. The effects of these organic agents on the filterability of the Sr/TRU precipitation process were examined along with the

decontamination of the radio-tracers. Three other variables were studied and were the sodium, aluminum, and hydroxide concentrations.

The results of the filtration tests are shown in Table 14. Filtrate volume was measured after one minute in the Phase 2 studies instead of the filtrate mass due to contamination control measures. Filtrate volumes ranged from 0 mL to 32.3 mL. In general, the filterability of the precipitates formed in this phase was poor. The filtration data shows six conditions in which little or filtrate was obtained. These conditions span the sodium and hydroxide conditions used in the tests. Additionally, the mid point filtration data showed a very low filtrate flux of 0.05 g/s.

The filtration data was regressed using the JMP software. Table 15 contains the results of this regression. The concentration of the gluconate group agents was shown to be a statistically significant variable. The group of complexing agents included gluconate, glycolate, and citrate. This group had a negative impact on filterability, i.e., increased level decrease filterability. This result is somewhat surprising since increased gluconate levels increases the solubility of iron and should have provided for better filtration

Table 14. Filtrate Volumes* from Phase 2 Beaker Tests

Pattern	Test No.	Na	OH	Al	Fe Conc	EDTA Gp.	Gluconate	Filtrate vol (mL)
-----	1	5.5	0.4	0.05	0.02	-1	-1	19.5
---++++	2	5.5	0.4	0.05	0.1	1	1	1
-+++++	3	5.5	0.4	0.3	0.02	-1	1	0
---++++	4	5.5	0.4	0.3	0.1	1	-1	19
-+++++	5	5.5	1	0.05	0.02	1	-1	32.3
---++++	6	5.5	1	0.05	0.1	-1	1	1
-+++++	7	5.5	1	0.3	0.02	1	1	1
---++++	8	5.5	1	0.3	0.1	-1	-1	16.4
-+++++	9	7	0.4	0.05	0.02	1	1	0
---++++	10	7	0.4	0.05	0.1	-1	-1	9.4
-+++++	11	7	0.4	0.3	0.02	1	-1	7.2
---++++	12	7	0.4	0.3	0.1	-1	1	10
-+++++	13	7	1	0.05	0.02	-1	1	0
---++++	14	7	1	0.05	0.1	1	-1	7.2
-+++++	15	7	1	0.3	0.02	-1	-1	5.3
++++++	16	7	1	0.3	0.1	1	1	0
Oooooo	17	6.25	0.6	0.175	0.06	0	0	2.9
ooooo*	18	6.25	0.6	0.175	0.075	0	0	3.3

*Volumes measured after one minute filtration.

performance with corresponding reduction in the TRU DF. The citrate component should not have an effect; however, glycolate could potentially act as a surfactant. The

concentration of the gluconate group spanned the concentrations of these organic species in the real AN-102 and AN-107 wastes. This action could peptize the precipitate. Additionally, the sodium level was significant, but only at a 10 % level. The Phase 2 data model predicts poorer filterability at high sodium levels. This is indicative of the high ionic strength of the solution. Shown in Figure 10 are the statistical plots for gluconate group and sodium levels. This data clearly indicates the glycolate/gluconate concentrations present in the real AN-102 and AN-107 wastes will affect the filterability of the ferric hydroxide precipitate. Without removal of the glycolate/gluconate, the ferric hydroxide precipitate can not be filtered in the AN-102 and AN-107 solutions.

Table 15. Regression Results from Phase 2 Filtration Tests

Term	Estimate	Std Error	t Ratio	Prob > t
Intercept	35.4	15.6	2.27	0.04
Sodium	-4.3	2.3	-1.83	0.09
Hydroxide	0.007	5.77	0.00	0.99
Aluminum	-5.75	13.9	-0.41	0.69
Fe Conc.	-4.5	43.4	-0.10	0.92
EDTA Gp	0.38	1.7	0.22	0.83
Gluconate Gp	-6.45	1.7	-3.7	0.0035

Figure 10. Statistical Plot for the Gluconate Group and the Sodium Levels

Table 16. Decontamination Factors from Phase 2 Tests

Pattern	Test No.	Sr DF	Am	Pu
-----	1	32.2		6.6
--++++	2	29.3	1.5	2.2
+++++	3	37.8	3.0	1.3
+++++	4	10.0	1.4	5.6
+++++	5	7.5	3.3	2.4
+++++	6	13.2	1.4	1.2
+++++	7	4.8	1.0	1.0
+++++	8	39.2		
+++++	9	10.8	1.3	1.7
+++++	10	52.6		3.6
+++++	11	4.5	1.5	4.6
+++++	12	24.0	2.9	1.3
+++++	13	15.7	0.9	11.9
+++++	14	32.9	1.8	1.0
+++++	15	26.6		1.0
+++++	16	14.3	1.4	1.3
oooooo	17	21.2	2.3	3.3
ooooo*	18	17.0	1.3	1.4

Table 16 compiles the decontamination factors (DFs) neglecting the effect of dilution obtained in the Phase 2 tests for Sr-85, PAM-241, and Pu-239. The starting, spiked concentrations were nominally 10^4 dpm/mL. The DFs for Sr ranged from 4.5 to 52.6. The DFs for the actinides ranged from 1 to 3 for Am and 1 to 12 for Pu. The DFs measured for americium are not sufficient to operate the proposed flowsheet which requires the Am-241 DF to be greater than 1.25 not including dilution. The DFs for Sr and Pu provide sufficient decontamination of the supernate.

Regression of DF data for strontium revealed only one statistically significant variable, the concentration of the EDTA group. An increase in the concentration of the EDTA group (EDTA, HEDTA, IDA, and NTA) caused a decrease in the DF for strontium. Figure 12 displays the statistical plot of the strontium DF data. Strontium is known to complex with EDTA and the other members of this organic grouping. Because only a single level of non-radioactive strontium was added in these tests, the data imply that some of this strontium was complexed by the chelants. This would effectively lower the isotope dilution effect of adding non-radioactive strontium.

Conversely, the statistical analysis of the actinide DF data did not reveal any variable as significant. Even iron concentration was not significant indicating a very large experimental error. Any experimental error could mask the effects of the process variables. If the response (DF) was larger than measured, then perhaps the effect each variable has could be measured. The low DFs for Am and the inability to measure an iron concentration effect are very detrimental to this flowsheet.

Figure 11. Statistical Plot of Sr DF Data

5.0 Conclusions

Approximately a dozen variables have been examined for their influence on the proposed Sr/TRU decontamination process for the tanks AN-102 and AN-107 in the BNFL flowsheet. Three sets of statistically designed experiments have evaluated these variables under non-radioactive and radioactive conditions. Additionally, a set of tests using a sample from AN-107 was performed to assess the flowsheet on real waste.

The combined results of the statistically designed testing and the single effect testing for improved filterability of the resultant precipitate demonstrate the importance of three variables. Although no variable was statistically significant in the Phase 1 tests with simulant enriched in gluconate, the sodium level was important in the confirmatory tests and the Phase 2 tests. The effect was a reduction in filterability at higher sodium levels and the magnitude of the effect was comparable in both sets of tests. The variable with the largest detrimental effect on filterability is the concentration of ferric nitrate added to co-precipitate the actinide ions. In order to obtain acceptable filter performance, the amount of ferric nitrate added would be insufficient to remove the required amount of actinide ion for further processing. Additionally, the results from the Phase 2 tests indicate a member of the gluconate grouping of complexing agents is also detrimental to the filterability of the Sr/TRU precipitate. It is believed that the glycolate ion is in large enough concentration to cause the precipitate to peptize; thus, reducing the filterability. Visual observations of the precipitation indicate a change in the nature of the precipitate. Initially, the precipitate is discrete particles. Prior to filtration the precipitate appears more like a gel with less particulate features.

The results for the factors affecting decontamination are not as conclusive but show some agreement to the factors found effecting filterability. Increased sodium levels were detrimental to lanthanide decontamination in the Phase 1 and the confirmatory tests. The largest single variable affecting lanthanide decontamination was, as expected, the amount of ferric nitrate added. This effect was exhibited in the confirmatory tests. However, sufficient decontamination factors could not be obtained in any of the series of tests including the real waste tests. These real waste tests exhibited better filterability than previously observed and acceptable decontamination of radio-strontium. However, plutonium and americium DFs were marginally acceptable at maximum ferric nitrate addition.

Overall, the process and chemical variables are better understood following these series of tests. The improved baseline flowsheet for Sr/TRU removal is still insufficient in the two responses of interest, filterability and decontamination. Alternative precipitating agents such as other transition metals or oxidants should be examined to replace the ferric nitrate co-precipitation of the actinide ions.

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Appendix 1. AN-107 Simulant Composition

Component	Molecular weight	Concentration	Units	Concentration	Units
Acetate	59.04462	1100 mg/Liter		1.86E-02 M	
Aluminum	26.98154	386 mg/Liter		1.43E-02 M	
Ammonium	18.03846	22 mg/Liter		1.22E-03 M	
Barium	137.33	7 mg/Liter		5.42E-05 M	
Boron	10.81	35 mg/Liter		3.24E-03 M	
Bromide	79.904	1150 mg/Liter		1.44E-02 M	
Cadmium	112.41	64 mg/Liter		5.70E-04 M	
Calcium	40.08	591 mg/Liter		1.47E-02 M	
Carbonate	60.0092	83936 mg/Liter		1.40E+00 M	
Cerium	140.12	53 mg/Liter		3.77E-04 M	
Chloride	35.453	1830 mg/Liter		5.16E-02 M	
Chromium	51.996	176 mg/Liter		3.38E-03 M	
Copper	63.546	30 mg/Liter		4.74E-04 M	
Ethylenediamine triacetic acid	288.20824	5620 mg/Liter		1.95E-02 M	
Fluoride	18.9984	133 mg/Liter		7.00E-03 M	
Formate	45.01774	10400 mg/Liter		2.31E-01 M	
Glycolate	75.04206	18600 mg/Liter		2.48E-01 M	
Hydroxide	17.00734	340 mg/Liter		2.00E-02 M	
Iron	55.847	1690 mg/Liter		3.03E-02 M	
Lanthanum	138.9055	46 mg/Liter		3.28E-04 M	
Lead	207.2	388 mg/Liter		1.87E-03 M	
Magnesium	24.305	25 mg/Liter		1.03E-03 M	
Manganese	54.938	563 mg/Liter		1.02E-02 M	
Molybdenum	95.94	36 mg/Liter		3.73E-04 M	
Neodymium	144.24	96 mg/Liter		6.65E-04 M	
n- Hydroxyethylenedi aminetriacetic acid	275.23618	2140 mg/Liter		7.78E-03 M	
Nickel	58.69	530 mg/Liter		9.03E-03 M	
Nitrate	62.0049	230000 mg/Liter		3.71E+00 M	
Nitrite	46.0055	61000 mg/Liter		1.33E+00 M	
Oxalate	88.0196	826 mg/Liter		9.38E-03 M	
Phosphate	94.97136	1110 mg/Liter		1.17E-02 M	
Potassium	39.0983	1810 mg/Liter		4.63E-02 M	
Selenium	78.96	1 mg/Liter		6.33E-06 M	
Silicon	28.0855	#VALUE!	mg/Liter	#VALUE!	M
Silver	107.8682	14 mg/Liter		1.33E-04 M	
Sodium	22.9898	195000 mg/Liter		8.48E+00 M	
Sulfate	96.0576	8250 mg/Liter		8.59E-02 M	
TIC	12.011	16800 mg/Liter		1.40E+00 M	
TOC		40400 mg/Liter		40.40 g/L	
Uranium		127 mg/Liter		1.27E+02 ug/mL	
Zinc	65.38	45 mg/Liter		6.93E-04 M	
Zirconium	91.22	70 mg/Liter		7.67E-04 M	

Additional Organics(based upon PNNL Report on FY1997 Results)

Nitrilotriacetic Acid	188.11618	561 mg/Liter	2.98E-03 M
Citric Acid	189.09618	8495 mg/Liter	4.49E-02 M
Iminodiacetic Acid	131.08412	5947 mg/Liter	4.54E-02 M

Source Previous PNNL Recipe

Sodium Gluconate	218.14	43628 mg/Liter	2.00E-01 M
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