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COMPLEXANT CONCENTRATE WASTE

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7. Abstract

A laboratory scouting test was conducted of precipitation methods for reducing the solubility of radionuclides in complexant concentrate (CC) waste solution. The results show that addition of strontium nitrate solution is effective in reducing the liquid phase activity of ^{90}Sr in CC waste from tank 107-AN by 94% when the total strontium concentration is adjusted to 0.1 M. Addition of ferric nitrate solution is effective in reducing the ^{241}Am activity in CC waste by 96% under the conditions described in the report. Ferric nitrate was also marginally effective in reducing the solubility of $^{239/240}\text{Pu}$ in CC waste.

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1.0 INTRODUCTION

A laboratory scouting test was conducted using precipitation methods for reducing the solubility of radionuclides in complexant concentrate (CC) waste solution. The results show that addition of strontium nitrate solution is effective in reducing the solubility of ^{90}Sr in CC waste from tank 107-AN by 94% when the total strontium concentration is adjusted to 0.1 M. Addition of ferric nitrate solution is effective in reducing the solubility of ^{241}Am in CC waste by 96% under the conditions described in the report. Ferric nitrate was also marginally effective in reducing the solubility of $^{239/240}\text{Pu}$ in CC waste.

1.1 Possible Mechanisms

In water, strontium carbonate is a sparingly soluble salt having a solubility product constant (K_{sp}) of 1.6×10^{-9} at 25 °C (Weast, 1980). If we neglect the effect of ionic strength on the K_{sp} , we can calculate an approximate value for the theoretical amount of strontium that should be in solution for a given level of carbonate. The concentration of carbonate ion in tank 107-AN waste is approximately 1.0 M (Herting, 1993a). If all of the strontium in the waste is assumed to be ^{90}Sr (a very conservative assumption), then the theoretical maximum activity of ^{90}Sr in the liquid phase should be:

$$K_{sp} = [\text{Sr}^{2+}][\text{CO}_3^{2-}] = 1.6 \times 10^{-9}$$

$$[\text{Sr}^{2+}] = 1.6 \times 10^{-9} / 1.0 = 1.6 \times 10^{-9} \text{ mol Sr per liter of CC waste}$$

$$(1.6 \times 10^{-9} \text{ mol Sr}) \times (87.6 \text{ g/mol}) = 1.4 \times 10^{-7} \text{ g Sr per liter}$$

$$(1.4 \times 10^{-7} \text{ g Sr}) \times (7.0 \times 10^7 \text{ } \mu\text{Ci } ^{90}\text{Sr/g Sr}) = 9.8 \text{ } \mu\text{Ci } ^{90}\text{Sr per liter}$$

The actual activity of ^{90}Sr in CC waste from tank 107-AN is 93,000 $\mu\text{Ci/L}$ (Herting, 1993a), nearly 10,000 times the theoretical solubility limit. The reason for the apparent high solubility of strontium in the CC waste is presumably that the strontium ions are complexed by the organic molecules prevalent in the waste. The activity of ^{90}Sr in the liquid phase in tank 107-AN is higher than in any other double shell tank liquid at Hanford (Brevick et al., 1995), and tank 107-AN also has the highest level of total organic carbon (TOC).

Addition of nonradioactive strontium nitrate to the waste causes formation of a precipitate, which is most likely SrCO_3 . Assuming that there is at least some degree of exchange between the complexed radioactive ^{90}Sr and the added nonradioactive strontium, some of the ^{90}Sr can be freed from the complexes to precipitate as $^{90}\text{SrCO}_3$. The process can be called "isotopic dilution".

A similar mechanism could be responsible for the effectiveness of ferric nitrate in lowering the solubility of ^{241}Am . In the absence of a K_{sp} value for $\text{Am}(\text{OH})_3$, it is worth noting that the activity of ^{241}Am is higher in tank 107-AN liquid phase than in any other double shell tank liquid at Hanford (Brevick et al., 1995), again presumably because of the organic complexing agents. Both Fe and Am exist as +3 ions under these conditions, and it is possible that the $\text{Fe}(\text{III})$ ions displace $\text{Am}(\text{III})$ ions from the organic complexes, allowing the ^{241}Am to precipitate as the hydroxide.

Another possible mechanism that could be operating is traditional carrier precipitation. When the molar concentration of a metal ion (Am^{3+} or Pu^{3+} or Pu^{4+}) is so low that the solubility product for the insoluble salt [e.g., $\text{Am}(\text{OH})_3$] is not exceeded, then adding a metal that forms an analogous insoluble salt [e.g., $\text{Fe}(\text{OH})_3$] will cause the first metal to co-precipitate. The results with the $^{239/240}\text{Pu}$ were less dramatic than with the ^{241}Am (i.e., the percent removal was not as good for $^{239/240}\text{Pu}$), possibly because the initial activity of $^{239/240}\text{Pu}$ was much lower.

Sodium phosphate was added to some of the samples to test the possibility that the phosphate ion might combine with either Fe^{3+} or Sr^{2+} to form a more effective carrier precipitate than $\text{Fe}(\text{OH})_3$ or SrCO_3 .

1.2 Earlier Laboratory Studies

The first laboratory study to examine the possibility of precipitating radionuclides from waste solutions was also performed on CC waste from tank 107-AN (Herting, 1993b). The original attempt was to precipitate hydroxides of the radionuclides by a simple caustic adjustment. This approach was almost completely ineffective. Very low levels (0.01 M in the treated solution) of ferric nitrate and strontium nitrate were added, and these also were largely ineffective at lowering the solubilities of the radionuclides. (The amount of precipitating agent was 10 to 30 times higher in the current tests than in the first study.)

A more extensive scouting study (Herting, 1994) was performed using a waste composite from three tanks. Most of the composite (70% by volume) was from tank 101-AW. The other tanks (20% from tank 106-AP and 10% from tank 102-AP) contained relatively dilute waste. The composite sample was further diluted with water to achieve 5 M sodium before the precipitation tests were done.

Precipitating agents that were tested on the 101-AW composite sample were $\text{Mn}(\text{NO}_3)_2$, $\text{Ca}(\text{NO}_3)_2$, $\text{Ni}(\text{NO}_3)_2$, $\text{Sr}(\text{NO}_3)_2$, Na_2CO_3 , Na_2SO_4 , Na_3PO_4 , and Na_2S . Only the +2 metal ions were effective in removing ^{90}Sr from this waste sample. The activity of ^{90}Sr in the liquid phase was reduced by 83%, 86%, 98.5%, and 99.4% for the Mn, Ca, Ni, and Sr salts, respectively. The $\text{Sr}(\text{NO}_3)_2$ was not only the most effective precipitating agent tested, it was also the most efficient (i.e., produced the smallest volume of centrifuged solids). None of the precipitating agents was effective in removing transuranic (TRU) isotopes from solution.

2.0 LABORATORY STUDIES

Precipitation tests were conducted on a liquid waste sample from tank 107-AN. The precipitating agents used were solutions of dissolved salts containing ferric nitrate and/or strontium nitrate. Trisodium phosphate, as the solid salt $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, was also added to some of the tests.

2.1 Experimental

The detailed procedure and data for these tests are recorded in controlled laboratory notebook WHC-N-384-2, pages 25-40.

2.1.1 Preparation of Reagents

Regent grade salts were dissolved in water to prepare the following solutions:

10 mL of 1.1 M $\text{Fe}(\text{NO}_3)_3$ (ferric nitrate)

10 mL of 1.1 M $\text{Fe}(\text{NO}_3)_3$ / 1.1 M $\text{Sr}(\text{NO}_3)_2$ (strontium nitrate)

10 mL of 3.3 M $\text{Fe}(\text{NO}_3)_3$

10 mL of 1.0 M $\text{Sr}(\text{NO}_3)_2$

The following solutions were provided by Standards Laboratories:

20 mL of 19.492 M NaOH (sodium hydroxide), 1.52877 g/mL

50 mL of 30% NaNO_3 (sodium nitrate), 1.1830 g/mL

2.1.2 Preparation of Tank Waste Sample

Tank 241-AN-107 (107-AN) bottle-on-a-string sample R3250 (laboratory identification number)/R3153 (tank farm identification number) was retrieved from storage. This sample was taken from the tank on February 7, 1993, from Riser 1A, at a depth recorded as "6 inches below solid level". It was intended to be a sludge sample, but upon receipt at the 222-S laboratory, the sample was found to contain only liquid.

In an open-faced fume hood in room 1G-A of the 222-S Laboratory, the sample bottle was agitated to achieve homogeneity. The sample was then poured into two 50 mL centrifuge cones. Both cones were centrifuged for one hour. Cone A contained 71.49 g/51.0 mL of supernatant liquid and 0.70 g/0.5 mL of centrifuged solids. Cone B contained 71.06 g/50.7 mL of supernatant liquid and 0.70 g/0.5 mL of centrifuged solids.

The supernatant liquid from cone A was decanted into a tared glass bottle labeled "107-AN Supernate/R-3250/1.5 M NaOH", hereafter referred to as "Bottle A". The liquid from cone B was poured into a similar bottle labeled "107-AN Supernate/R-3250/3.0 M NaOH", hereafter referred to as "Bottle B". The light brown, slightly orange centrifuged solids were discarded.

Baseline analytical samples were prepared by diluting 2.00 mL of supernate from each of the glass bottles in 10.0 mL of water in sample vials labeled HE38429A (for the liquid from cone A) and HE38429B (cone B). The water volume was measured by weight/density, and the supernatant liquid volume was measured by automatic pipet, which had been calibrated using the 30% NaNO₃ solution supplied by Standards Laboratory (see Section 2.1.1). Both of the sample vials were stirred for 5 minutes, then submitted for the following analyses: free hydroxide (OH⁻), ⁹⁰Sr, ^{239/240}Pu, ²⁴¹Am, total inorganic carbon (TIC), and metals by inductively coupled plasma spectroscopy (ICP).

2.1.3 Caustic Adjustment

Tank 107-AN supernatant liquid has a free hydroxide concentration of approximately 0.02 M, and has a buffering capacity that had been measured earlier (Herting, 1993a). Thus, the observed concentration of NaOH (i.e., measured by free hydroxide analysis) in a 107-AN solution to which NaOH has been added is consistently found to be less than the calculated concentration based on the known amount of NaOH added. The following relationship between observed NaOH molarity ([NaOH]_{obs}) and calculated NaOH molarity ([NaOH]_{calc}) was developed:

$$[\text{NaOH}]_{\text{obs}} = 0.88 \times [\text{NaOH}]_{\text{calc}} - 0.13$$

Based on this relationship, the amounts of 19.492 M NaOH required to adjust the two 107-AN supernatant solutions in the glass bottles to 1.5 M and 3.0 M free hydroxide were calculated as 5.14 mL and 10.88 mL, respectively. After the volumes were converted to weights using the density supplied by Standards Laboratory (Section 2.1.1), 7.87 g and 16.64 g of 19.492 M NaOH were added to the respective glass bottles. Each bottle was stirred for 24 hours to equilibrate the samples. Precipitation of Na₂CO₃·H₂O in Bottle B was so extensive that the slurry effectively solidified, so that the bottle could be inverted without draining any liquid. The bottle was heated to 40 °C to fluidize the sample enough that it could be pipetted.

2.1.4 Precipitation

The precipitation reactions were carried out in twenty-four 15 mL centrifuge cones labeled 1A, 1B, 2A, 2B ... 12A, 12B. Cones 4A, 4B, 10A and 10B each contained 0.45 to 0.47 g of reagent grade Na₃PO₄·12H₂O, which was enough to make the 107-AN waste solution 0.3 M in phosphate. An automatic pipet was used to deliver 4.0 mL of 107-AN caustic-adjusted solution from Bottle A into centrifuge cones 1A through 6B. Similarly, Bottle B samples (slurries) were pipetted into cones 7A through 12B. Volumes of samples were determined by weight/density.

Precipitation reagents were added to the cones according to the pattern shown in Table 1. Reagents were added using a pipet calibrated with water to deliver 0.40 mL of solution. Immediately after addition of the reagent, the cone was vortexed for 10-15 seconds, then placed on a tumbler which gently tumbled the cones end-over-end. All of the cones were tumbled for 5 days, at ambient temperature, changing the direction of tumbling every 12 hours. The ambient air temperature in the laboratory ranged from 27 to 31 °C during the tumbling period.

After the 5 days of tumbling, all of the cones were centrifuged for 20 minutes. Weights and volumes of supernatant solutions and centrifuged solids were recorded. The supernatant solutions from cones 6A, 6B, 12A and 12B were transferred into four new cones labeled 6A', 6B', 12A' and 12B'. Cones 6A' and 6B' were treated with a second addition of 0.40 mL of 1.1 M $\text{Fe}(\text{NO}_3)_3$ solution, and cones 12A' and 12B' were treated with a second addition of 0.30 mL the same reagent. (The volume was smaller for 12A' and 12B' because the volume of supernatant solution was correspondingly smaller.) These four cones were each vortexed as before, then tumbled for another 5 days on the rocker. Then they were centrifuged for 20 minutes.

Supernatant solutions from all cones were diluted by decanting the solutions from the centrifuge cones into sample vials which contained 10 mL of water. The sample vials were labeled as shown in Table 1. Each vial was stirred for 5 minutes, then submitted for the same set of analyses that were performed on the baseline samples. The centrifuged solids were discarded.

2.2 Data

2.2.1 Sample Volumes

All 24 centrifuge cones contained 4.00 mL (± 0.04 mL) of caustic-adjusted 107-AN sample from either Bottle A or Bottle B. The volume of reagent added to each cone was 0.40 mL (± 0.01 mL), except for the following:

- Cones 1A/B and 7A/B were controls, with no reagents added
- Cones 4A/B and 10A/B contained an additional 0.28 mL (± 0.01 mL) of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$
- The addition of reagent to cones 6A'/B' and 12A'/B' was based on the volume of supernatant liquid decanted from cones 6A/B (4.1 and 4.2 mL, respectively) and 12A/B (3.3 and 3.2 mL). Therefore, 0.40 mL of reagent was added to cones 6A'/B', and 0.30 mL of reagent was added to cones 12A'/B'.

The final total sample volume in each cone, as measured in the centrifuge cone, is shown in Table 2 along with the volume of centrifuged solids and volume of decanted supernatant liquid.

Table 1. Precipitation Reagents Added to 15 mL Centrifuge Cones

107-AN Source	Cone #	Reagent Added	Sample Vial
Bottle A (1.5 M NaOH)	1A 1B	none (control)	HE38437A HE38437M
	2A 2B	1.1 M $\text{Fe}(\text{NO}_3)_3$	HE38437B HE38437N
	3A 3B	3.3 M $\text{Fe}(\text{NO}_3)_3$	HE38437C HE38437O
	4A 4B	Na_3PO_4 /1.1M $\text{Fe}(\text{NO}_3)_3$ / 1.0 M $\text{Sr}(\text{NO}_3)_2$	HE38437D HE38437P
	5A 5B	1.0 M $\text{Sr}(\text{NO}_3)_2$	HE38437E HE38437Q
	6A (1st add) 6B (1st add)	1.1 M $\text{Fe}(\text{NO}_3)_3$	--- ---
	6A' (2nd add) 6B' (2nd add)	1.1 M $\text{Fe}(\text{NO}_3)_3$	HE38437F HE38437R
Bottle B (3.0 M NaOH)	7A 7B	none (control)	HE38437G HE38437S
	8A 8B	1.1 M $\text{Fe}(\text{NO}_3)_3$	HE38437H HE38437T
	9A 9B	3.3 M $\text{Fe}(\text{NO}_3)_3$	HE38437I HE38437U
	10A 10B	Na_3PO_4 /1.1 M $\text{Fe}(\text{NO}_3)_3$ / 1.0 M $\text{Sr}(\text{NO}_3)_2$	HE38437J HE38437V
	11A 11B	1.0 M $\text{Sr}(\text{NO}_3)_2$	HE38437K HE38437W
	12A (1st add) 12B (1st add)	1.1 M $\text{Fe}(\text{NO}_3)_3$	--- ---
	12A' (2nd add) 12B' (2nd add)	1.1 M $\text{Fe}(\text{NO}_3)_3$	HE38437L HE38437X

Table 2. Sample Volume Data

Cone #	Total Volume, mL	Centrifuged Solids Vol, mL	Decanted Supernate Vol, mL
1A 1B	4.0 4.0	< 0.1 < 0.1	4.0 4.0
2A 2B	4.6 4.6	0.3 0.3	4.3 4.3
3A 3B	4.7 4.6	0.5 0.6	4.2 4.0
4A 4B	4.7 4.7	1.2 1.2	3.5 3.5
5A 5B	4.5 4.5	0.2 0.2	4.3 4.3
6A 6B	4.5 4.6	0.4 0.4	4.1 4.2
6A' 6B'	4.4 4.5	0.2 0.2	4.2 4.3
7A 7B	4.0 4.0	1.9 1.9	2.1 2.1
8A 8B	4.5 4.3	1.1 1.1	3.4 3.2
9A 9B	4.4 4.5	1.1 1.1	3.3 3.4
10A 10B	4.7 4.8	1.8 1.8	2.9 3.0
11A 11B	4.5 4.4	1.0 1.0	3.5 3.4
12A 12B	4.5 4.4	1.2 1.2	3.3 3.2
12A' 12B'	3.5 3.5	0.15 0.15	3.35 3.35

2.2.2 Analytical Results

In order to provide sufficient sample volume for analysis, all samples were diluted with water. In all cases, the volume of water was 10 mL. The volume of sample added to the water was the same as the "Decanted Supernate Volume" in Table 2. For the two baseline samples (taken from Bottles A and B before the hydroxide adjustments were made), the sample volume was 2.005 mL.

Dilution factors were calculated to convert the raw analytical data (shown in Appendix A) to a uniform comparison basis. The original 107-AN supernatant liquid was used as the basis. Conversion factors are needed to account for (a) dilution of the decanted supernatant liquid with water, (b) addition of NaOH solution during caustic adjustment, and (c) addition of precipitation reagents. All conversion factors are calculated with the assumption that volumes are additive whenever two liquids are mixed. This assumption may not be exactly true in all cases, but the error arising from this assumption is probably small in relation to the analytical error.

To convert from raw data to concentrations in the decanted liquid, the raw data result is multiplied by the factor $[(\text{decanted supernate volume} + 10)/(\text{decanted supernate volume})]$.

To account for caustic adjustment (for all samples except the baseline samples), the decanted liquid concentrations are multiplied by the factor (54/49) for samples 1A-6B, and (60/49) for samples 7A-12B. These factors are derived from the volumes of 107-AN supernate in Bottles A and B (49 mL in each bottle after the baseline samples were removed), and the volumes of 19.492 M NaOH added (5 mL and 11 mL, respectively).

To account for the dilution caused by adding the precipitation reagents, the factors are (4.4/4.0) for cones 2A-5B and 8A-11B, and (4.8/4.0) for cones 6A'/B' and 12A'/B'.

The following example calculation will illustrate how the raw data is converted to the common basis. The raw analytical result for ^{90}Sr in the diluted sample from cone 2A (see sample HE-38437B in Appendix A) was 12.8 $\mu\text{Ci/mL}$. Converted to the common basis, the result would be:

$$^{90}\text{Sr} = 12.8 \mu\text{Ci/mL} \times \frac{14.3}{4.3} \times \frac{54}{49} \times \frac{4.4}{4.0} = 51.6 \mu\text{Ci/mL}$$

The "common-basis" analytical results are shown in Appendix B. Tables 3a through 3c give the averages of the common-basis results of duplicate samples (1A and 1B, etc.), arranged according to analyte and treatment.

Table 3a. Common-Basis Analytical Results for Radionuclides
(all values in $\mu\text{Ci/mL}$)

Analyte	^{90}Sr		^{241}Am		$^{239/240}\text{Pu}$	
	1.5 M	3.0 M	1.5 M	3.0 M	1.5 M	3.0 M
Baseline	93.0 ^a		0.67		0.037	
NaOH only	86.6	74.3	0.53	0.19	0.027	0.029
0.1 M Fe	47.0	51.2	0.11	0.028	0.016	0.10 ^b
0.3 M Fe	26.4	18.7	0.066	0.030	0.010	0.008
0.1 M Fe (2x)	22.3	18.1	0.030	0.021	0.012	0.015
Fe/Sr/ PO_4	5.7	5.0	0.092	0.057	0.015	0.014
0.1 M Sr	4.8	6.1	0.40	0.42	0.033	0.031

^a An average of several historical values (Herting, 1993a) was used for the ^{90}Sr baseline, because the baseline sample results were very different from one another and inconsistent with the historical samples (see Appendix B). For all other analytes, the baseline sample results were reasonably consistent with the historical results.

^b Suspect result may be factor of 10 high; should be 0.010 $\mu\text{Ci/mL}$.

Table 3b. Common-Basis Analytical Results for Chemicals - Part I.
(all values in $\mu\text{g/mL}$)

Analyte	TIC ^a		OH ⁻		Na	
	1.5 M	3.0 M	1.5 M	3.0 M	1.5 M	3.0 M
Baseline	14,800		n.d. ^b		186,000	
NaOH only	15,200	7,100	30,200	74,100	225,000	250,000
0.1 M Fe	15,100	10,300	26,700	59,400	224,000	229,000
0.3 M Fe	15,100	11,800	19,300	57,700	225,000	262,000
0.1 M Fe (2x)	15,400	11,400	23,400	62,300	219,000	268,000
Fe/Sr/PO ₄	14,300	10,500	27,000	67,700	237,000	273,000
0.1 M Sr	14,500	10,300	31,200	65,700	229,000	270,000

^a TIC = total inorganic carbon^b n.d. = not detected (detection limit approximately 400 $\mu\text{g/mL}$)Table 3c. Common-Basis Analytical Results for Chemicals - Part II.
(all values in $\mu\text{g/mL}$)

Analyte	Fe		K		Al		S	
	1.5 M	3.0 M	1.5 M	3.0 M	1.5 M	3.0 M	1.5 M	3.0 M
Baseline	784		1,630		847		2,630	
NaOH only	1,050	398	1,600	1,580	924	1,000	2,610	2,770
0.1 M Fe	300	110	1,630	1,400	883	859	2,580	2,490
0.3 M Fe	148	146	1,600	1,580	871	996	2,580	2,730
0.1 M Fe (2x)	127	118	1,530	1,680	900	990	2,580	2,710
Fe/Sr/PO ₄	201	194	1,690	1,750	935	1,020	2,660	2,800
0.1 M Sr	760	493	1,660	1,630	955	998	2,620	2,680

3.0 DISCUSSION

3.1 ⁹⁰Sr Removal

Precipitation of ⁹⁰Sr from 107-AN liquid waste can be effectively achieved using strontium nitrate solution as the precipitating agent. The results in Table 3a show that the solubility of ⁹⁰Sr is reduced by approximately 94% when strontium nitrate is added, either alone or in combination with iron and phosphate. (To calculate the percent reduction in solubility, divide the final concentration by the baseline concentration, multiply by 100 to convert to percent remaining in solution, and subtract from 100% to find percent removed.)

Addition of NaOH alone had a small effect on ⁹⁰Sr solubility, reducing it by only 7% at 1.5 M OH⁻ and 20% at 3.0 M OH⁻.

Addition of ferric nitrate also had a small effect, which increased with the amount of iron added. Two successive additions at 0.1 M Fe worked slightly better than a single addition at 0.3 M Fe. Overall, the percent ⁹⁰Sr removal achieved with ferric nitrate ranged from 50% (single addition at 0.1 M Fe) to 80% (double addition at 0.1 M Fe).

3.2 TRU Removal

Strontium nitrate solution did not appear to be very effective for precipitation of the transuranic (TRU) isotopes ²⁴¹Am and ^{239/240}Pu. When Sr(NO₃)₂ was added alone, only 39% of the ²⁴¹Am and 14% of the ^{239/240}Pu were removed.

Addition of NaOH alone had a small effect on TRU solubility, although 72% of the ²⁴¹Am was removed at 3.0 M hydroxide.

Addition of ferric nitrate was quite effective in precipitating ²⁴¹Am. The effect increased with increasing iron concentration and increasing hydroxide concentration. The optimal precipitation occurred with two successive ferric hydroxide additions, wherein the percent ²⁴¹Am removed was 96% and 97% at 1.5 M and 3.0 M OH⁻, respectively. Ferric nitrate was less effective for ^{239/240}Pu. The percent removal ranged from 57% to 78%, and did not appear to depend on the amount of hydroxide or iron added.

3.3 Behavior of Chemical Components

3.3.1 Hydroxide

The hydroxide analyses in Table 3b indicate that the target concentrations of 1.5 M and 3.0 M free hydroxide were over-shot. The value of 30,200 µg/mL for the control samples corresponds to 1.78 M free hydroxide. The value of 74,100 µg/mL corresponds to 4.36 M free hydroxide. In the case of the 3.0 M target, the over-shoot may have been due to the change in solution volume when Na₂CO₃·H₂O precipitated (see Section 3.4).

The free hydroxide is seen to decrease when the ferric nitrate is added (see Table 3b), due to neutralization of the ferric ion $[\text{Fe}^{3+} + 3 \text{OH}^- \rightarrow \text{Fe}(\text{OH})_3]$. Addition of strontium nitrate does not appear to cause a decrease in hydroxide concentration, implying that the solid phase that forms is SrCO_3 , not $\text{Sr}(\text{OH})_2$. (The hydroxide does appear to decrease somewhat for the 3 M OH^- /0.1 M Sr case in Table 3c, but only because of an apparently anomalous result for one of the duplicate sample -- see Appendix B, Table B6.)

Whether ferric nitrate or strontium nitrate was added the volume of Na_2CO_3 solids formed in the 3.0 M OH^- sample was reduced. In both cases, there is a 10% dilution of carbonate concentration due to addition of the precipitating agent solution. With the ferric nitrate, there is a further increase in Na_2CO_3 solubility due to the reduction in hydroxide caused by the reaction of OH^- with the ferric ion. With the strontium nitrate, there is a further reduction in the carbonate concentration in solution due to the precipitation of SrCO_3 .

3.3.2 TIC

Tank 107-AN waste is saturated in Na_2CO_3 , so it was not surprising to find $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ crystals forming in abundance when the hydroxide was raised to 3.0 M. Analytically, this is observed (Table 3b) as a reduction in the TIC concentration (Table 3b). Note that the TIC concentration increases compared to the 3.0 M control solution "NaOH only" when ferric ion or strontium ion is added, for the reasons just discussed in the previous section.

3.3.3 Sodium

Table 3b shows that the Na concentration increases with increasing hydroxide. This is because the hydroxide was added as NaOH. Precipitation of $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ should cause a small change in the amount of sodium left in solution, but the change is too small to observe given the amount of scatter in the analytical results.

3.3.4 Iron

The solubility of Fe in the 107-AN solution appears to be a function of hydroxide concentration and of the amount of iron added. Surprisingly, the more iron added to the solution, the lower the solubility appears to be. The solubility of iron is approximately a factor of five lower in the solution treated twice with ferric nitrate than in the original (baseline) solution.

3.3.5 Potassium, Aluminum, Sulfur

Potassium, aluminum and sulfur (presumably present as sulfate) concentrations appear to be unaffected by the addition of any of the reagents. In the case of Al, it might appear that addition of NaOH caused a slight increase in Al solubility, but remember that there are no Al solids present that could dissolve. Therefore, the apparent increase is probably attributable to a slightly low value for the baseline.

3.4 Percent Solids

In all cases, precipitation of ^{90}Sr and other radionuclides is achieved at the expense of forming high level waste solids. The goal of any waste treatment scheme based on precipitation would be to maximize the amount of the radionuclides removed while minimizing the amount of solids formed.

Table 4 shows a summary of the volume percent solids formed in each test, relative to the original volume of waste before any caustic adjustment. To calculate the volume percent solids, divide the volume of centrifuged solids from Table 2 by the pre-caustic-adjustment sample volume ($4.0 \times 49/54 = 3.63$ mL for 1.5 M NaOH and $4.0 \times 49/60 = 3.27$ mL for 3.0 M NaOH), and multiply by 100 to convert the fraction to percent.

Table 4. Volume Percent Centrifuged Solids^a

Reagent Added	1.5 M NaOH	3.0 M NaOH
NaOH only	< 3	58
0.1 M Fe	8	34
0.3 M Fe	15	34
0.1 M Fe (2x)	16	41
Fe/Sr/ PO_4	33	55
0.1 M Sr	6	31

^a Relative to volume of 107-AN supernate before caustic adjustment.

The data in Table 4 show that adjustment of the 107-AN solution to 3.0 M hydroxide significantly increases the volume of solids, due to the precipitation of Na_2CO_3 . Tables 3a and 4 provide the necessary input data for an engineering evaluation to calculate the financial costs and implications of the waste treatment trade-offs (reduced hazard of the 107-AN supernate vs. increase in high level waste solids).

4.0 FUTURE DATA NEEDS

There is an obvious need to determine how varying the concentration of the precipitating reagent would affect the radionuclide removal efficiency and the volume percent solids. If 94% of the ^{90}Sr is removed at 0.1 M $\text{Sr}(\text{NO}_3)_2$ added, then how much ^{90}Sr could be removed at 0.05 M added strontium? at 0.02 M? And what volume percent solids is formed in each case?

The data in this report provide a fairly good estimate of radionuclide removal efficiencies for a Sr/Fe mixed precipitating agent, but the volume percent solids generated is not available because of the presence of the Na_3PO_4 used in these tests. The Na_3PO_4 did not appear to have any observable effect on the removal efficiency, but it undoubtedly contributed substantially to the volume percent solids. Removal efficiencies and volume percent solids should be measured for the Sr/Fe mixed precipitating agent at various concentration levels.

5.0 REFERENCES

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- Herting, D. L., 1994, *Strontium Removal by Precipitation*, Internal Memo 12110-PCL94-026.
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APPENDIX A

ANALYTICAL RESULTS FOR DILUTED SAMPLES

Table A1. Baseline Sample Results

		Bottle A	Bottle B
Customer ID Number		HE-38429A	HE-38429B
Laboratory Serial Number		J1636	J1637
TIC	$\mu\text{g/mL}$	2470	2470
OH^-	$\mu\text{g/mL}$	< 125	< 125
Al	$\mu\text{g/mL}$	140	143
Fe	$\mu\text{g/mL}$	130	132
K	$\mu\text{g/mL}$	268	276
Na	$\mu\text{g/mL}$	30800	31500
S	$\mu\text{g/mL}$	435	443
$^{239/240}\text{Pu}$	$\mu\text{Ci/mL}$	0.00631	0.00599
^{241}Am	$\mu\text{Ci/mL}$	0.106	0.119
^{90}Sr	$\mu\text{Ci/mL}$	60.0	8.99

Table A2. Control Sample Results

		1.5 M NaOH		3.0 M NaOH	
		Cone 1A	Cone 1B	Cone 7A	Cone 7B
Customer ID Number		HE-38437A	HE-38437M	HE-38437G	HE-38437S
Lab Serial Number		J1639	J1653	J1646	J1660
TIC	$\mu\text{g/mL}$	3980	3920	1020	993
OH^-	$\mu\text{g/mL}$	7910	7750	10700	10300
Al	$\mu\text{g/mL}$	238	241	137	147
Fe	$\mu\text{g/mL}$	271	275	63.8	48.9
K	$\mu\text{g/mL}$	414	416	219	229
Na	$\mu\text{g/mL}$	58100	58400	34600	36400
S	$\mu\text{g/mL}$	671	680	393	392
$^{239/240}\text{Pu}$	$\mu\text{Ci/mL}$	0.0103	0.00394	0.00408	0.00422
^{241}Am	$\mu\text{Ci/mL}$	0.141	0.135	0.0258	0.0284
^{90}Sr	$\mu\text{Ci/mL}$	25.0	19.9	8.36	12.7

Table A3. Sample Results at 0.1 M Iron

		1.5 M NaOH		3.0 M NaOH	
		Cone 2A	Cone 2B	Cone 8A	Cone 8B
Customer ID Number		HE-38437B	HE-38437N	HE-38437H	HE-38437T
Lab Serial Number		J1640	J1654	J1647	J1661
TIC	µg/mL	3810	3670	2140	I.S. ^a
OH ⁻	µg/mL	6610	6630	12300	I.S.
Al	µg/mL	221	217	178	I.S.
Fe	µg/mL	75.4	73.2	22.8	I.S.
K	µg/mL	418	391	291	I.S.
Na	µg/mL	56400	54800	47400	I.S.
S	µg/mL	645	636	516	I.S.
^{239/240} Pu	µCi/mL	0.00299	0.00514	0.0214	I.S.
²⁴¹ Am	µCi/mL	0.0316	0.0242	0.00572	I.S.
⁹⁰ Sr	µCi/mL	12.8	10.5	10.6	I.S.

^a All analytes reported as "insufficient sample"

Table A4. Sample Results at 0.3 M Iron

		1.5 M NaOH		3.0 M NaOH	
		Cone 3A	Cone 3B	Cone 9A	Cone 9B
Customer ID Number		HE-38437C	HE-38437O	HE-38437I	HE-38437U
Lab Serial Number		J1641	J1655	J1648	J1663
TIC	µg/mL	3700	3560	2290	2110
OH ⁻	µg/mL	4690	4580	10500	11000
Al	µg/mL	212	206	181	190
Fe	µg/mL	34.4	36.6	28	26.4
K	µg/mL	392	375	291	298
Na	µg/mL	54300	53400	48200	49300
S	µg/mL	621	615	517	499
^{239/240} Pu	µCi/mL	0.00252	0.00251	0.00155	0.00149
²⁴¹ Am	µCi/mL	0.0109	0.0204	0.00488	0.00649
⁹⁰ Sr	µCi/mL	7.39	5.32	2.62	4.37

Table A5. Sample Results at 0.1 M Fe/0.1 M Sr/0.3 M Phosphate

		1.5 M NaOH		3.0 M NaOH	
		Cone 4A	Cone 4B	Cone 10A	Cone 10B
Customer ID Number		HE-38437D	HE-38437P	HE-38437J	HE-38437V
Lab Serial Number		J1642	J1657	J1649	J1664
TIC	μg/mL	3090	3020	1760	1800
OH ⁻	μg/mL	5730	5800	11300	11600
Al	μg/mL	196	204	170	176
Fe	μg/mL	39.2	46.7	32.8	32.9
K	μg/mL	372	350	309	282
Na	μg/mL	50800	50400	45600	46600
S	μg/mL	570	558	486	460
^{239/240} Pu	μCi/mL	0.00285	0.00355	0.00231	0.00256
²⁴¹ Am	μCi/mL	0.0195	0.0198	0.00846	0.0109
⁹⁰ Sr	μCi/mL	0.899	1.54	0.843	0.842

Table A6. Sample Results at 0.1 M Sr

		1.5 M NaOH		3.0 M NaOH	
		Cone 5A	Cone 5B	Cone 11A	Cone 11B
Customer ID Number		HE-38437E	HE-38437Q	HE-38437K	HE-38437W
Lab Serial Number		J1643	J1658	J1651	J1665
TIC	μg/mL	3550	3650	1920	2000
OH ⁻	μg/mL	7780	7690	11000	14000
Al	μg/mL	230	244	189	191
Fe	μg/mL	196	181	98.5	89.2
K	μg/mL	393	430	331	289
Na	μg/mL	56400	57400	52400	50300
S	μg/mL	650	649	519	503
^{239/240} Pu	μCi/mL	0.00847	0.00789	0.00626	0.00542
²⁴¹ Am	μCi/mL	0.107	0.0912	0.104	0.0553
⁹⁰ Sr	μCi/mL	1.47	0.894	0.864	1.45

Table A7. Sample Results after Second Strike at 0.1 M Iron

		1.5 M NaOH		3.0 M NaOH	
		Cone 6A'	Cone 6B'	Cone 12A'	Cone 12B'
Customer ID Number		HE-38437F	HE-38437R	HE-38437L	HE-38437X
Lab Serial Number		J1645	J1659	J1652	J1666
TIC	$\mu\text{g/mL}$	3480	3470	1950	1970
OH ⁻	$\mu\text{g/mL}$	5370	5200	10600	10900
Al	$\mu\text{g/mL}$	195	211	167	175
Fe	$\mu\text{g/mL}$	28.3	28.9	20.3	20.5
K	$\mu\text{g/mL}$	345	346	305	277
Na	$\mu\text{g/mL}$	47600	51000	46400	46000
S	$\mu\text{g/mL}$	582	582	469	467
^{239/240} Pu	$\mu\text{Ci/mL}$	0.00269	0.00275	0.00252	0.00255
²⁴¹ Am	$\mu\text{Ci/mL}$	0.00587	0.00757	0.00221	0.00505
⁹⁰ Sr	$\mu\text{Ci/mL}$	4.35	5.74	2.56	3.70

APPENDIX B

**ANALYTICAL RESULTS CONVERTED TO COMMON BASIS
(DILUTION FACTORS INCORPORATED)**

Analytical results for the diluted samples were converted to a "common basis" by multiplying by the appropriate dilution factors, as explained in Section 2.2.2. The "common basis" is defined as what the concentrations of the samples would have been if there had been no change in volume when (a) the caustic adjustment was made, (b) the precipitation reagents were added, or (c) the final decanted liquids were diluted with water for analysis. In other words, the "common basis" values can be compared directly with the concentrations in the tank 107-AN waste liquid before any reagents were added to the waste. The dilution factors used were as follows:

Baseline samples: multiply Appendix A values by $(12.005/2.005) = 5.99$

Cones 1A and 1B: multiply App A values by $(54/49) \times (14/4.0) = 3.86$

Cones 2A/B - 5A/B: multiply App A values by $(54/49) \times ((V_s + 10)/V_s) \times (4.4/4.0)$, where V_s is the volume of the supernatant liquid decanted from the centrifuge cone. These values are given in Table 2 of the report.

Cones 6A' and 6B': multiply by $(54/49) \times ((V_s + 10)/V_s) \times (4.8/4.0)$

Cones 7A and 7B: multiply by $(60/49) \times (12.1/2.1) = 7.06$

Cones 8A - 11B: multiply by $(60/49) \times ((V_s + 10)/V_s) \times (4.4/4.0)$

Cones 12A' and 12B': multiply by $(60/49) \times ((V_s + 10)/V_s) \times (4.8/4.0)$

The common-basis values are rounded off to the appropriate number of significant figures after averaging the duplicate sample results. The average value is recorded in Tables 3a - 3c in the report.

Table B1. Baseline Common-Basis Results

		Bottle A	Bottle B
TIC	$\mu\text{g/mL}$	14789	14789
OH ⁻	$\mu\text{g/mL}$	< 748	< 748
Al	$\mu\text{g/mL}$	838	856
Fe	$\mu\text{g/mL}$	778	790
K	$\mu\text{g/mL}$	1605	1653
Na	$\mu\text{g/mL}$	184416	188607
S	$\mu\text{g/mL}$	2605	2652
^{239/240} Pu	$\mu\text{Ci/mL}$	0.038	0.036
²⁴¹ Am	$\mu\text{Ci/mL}$	0.635	0.713
⁹⁰ Sr	$\mu\text{Ci/mL}$	359	54

Table B2. Control Sample Common-Basis Results

1.5 M NaOH		3.0 M NaOH	
Cone 1A		Cone 7A	
Cone 1B		Cone 7B	
TIC	µg/mL	15351	15120
OH ⁻	µg/mL	30510	29893
Al	µg/mL	918	930
Fe	µg/mL	1045	1061
K	µg/mL	1597	1605
Na	µg/mL	224100	225257
S	µg/mL	2588	2623
^{239/240} Pu	µCi/mL	0.040	0.015
²⁴¹ Am	µCi/mL	0.544	0.521
⁹⁰ Sr	µCi/mL	96.4	76.8
		59.0	59.0
		0.200	0.200
		2766	2766
		256816	256816
		1616	1616
		345	450
		1037	967
		72671	75493
		7006	7197

Table B3. Common-Basis Results at 0.1 M Iron

1.5 M NaOH		3.0 M NaOH	
Cone 2A		Cone 8A	
Cone 2B		Cone 8B	
TIC	µg/mL	15360	14795
OH ⁻	µg/mL	26648	26727
Al	µg/mL	891	875
Fe	µg/mL	304	295
K	µg/mL	1685	1576
Na	µg/mL	227372	220922
S	µg/mL	2600	2564
^{239/240} Pu	µCi/mL	0.012	0.021
²⁴¹ Am	µCi/mL	0.127	0.098
⁹⁰ Sr	µCi/mL	51.6	42.3
		51.2	51.2
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.
		I.S.	I.S.

All analytes reported as "insufficient sample"

Table B4. Common-Basis Results at 0.3 M Iron

		1.5 M NaOH		3.0 M NaOH	
		Cone 3A	Cone 3B	Cone 9A	Cone 9B
TIC	μg/mL	15165	15105	12431	11201
OH ⁻	μg/mL	19222	19432	57000	58394
Al	μg/mL	869	874	983	1009
Fe	μg/mL	141	155	152	140
K	μg/mL	1607	1591	1580	1582
Na	μg/mL	222551	226569	261657	261710
S	μg/mL	2545	2609	2807	2649
^{239/240} Pu	μCi/mL	0.010	0.011	0.008	0.008
²⁴¹ Am	μCi/mL	0.045	0.087	0.026	0.034
⁹⁰ Sr	μCi/mL	30.3	22.6	14.2	23.2

Table B5. Common-Basis Results at 0.1 M Fe/0.1 M Sr/0.3 M Phosphate

		1.5 M NaOH		3.0 M NaOH	
		Cone 4A	Cone 4B	Cone 10A	Cone 10B
TIC	μg/mL	14448	14121	10545	10506
OH ⁻	μg/mL	26792	27120	67705	67706
Al	μg/mL	916	954	1019	1027
Fe	μg/mL	183	218	197	192
K	μg/mL	1739	1637	1851	1646
Na	μg/mL	237531	235660	273215	271992
S	μg/mL	2665	2609	2912	2685
^{239/240} Pu	μCi/mL	0.013	0.017	0.014	0.015
²⁴¹ Am	μCi/mL	0.091	0.093	0.051	0.064
⁹⁰ Sr	μCi/mL	4.2	7.2	5.1	4.9

Table B6. Common-Basis Results at 0.1 M Sr

		1.5 M NaOH		3.0 M NaOH	
		Cone 5A	Cone 5B	Cone 11A	Cone 11B
TIC	$\mu\text{g/mL}$	14312	14715	9975	10617
OH ⁻	$\mu\text{g/mL}$	31364	31002	57149	74319
Al	$\mu\text{g/mL}$	927	984	982	1014
Fe	$\mu\text{g/mL}$	790	730	512	474
K	$\mu\text{g/mL}$	1584	1734	1720	1534
Na	$\mu\text{g/mL}$	227372	231403	272236	267019
S	$\mu\text{g/mL}$	2620	2616	2696	2670
^{239/240} Pu	$\mu\text{Ci/mL}$	0.034	0.032	0.033	0.029
²⁴¹ Am	$\mu\text{Ci/mL}$	0.431	0.368	0.540	0.294
⁹⁰ Sr	$\mu\text{Ci/mL}$	5.9	3.6	4.5	7.7

Table B7. Common-Basis Results after Second Strike at 0.1 M Iron

		1.5 M NaOH		3.0 M NaOH	
		Cone 6A'	Cone 6B'	Cone 12A'	Cone 12B'
TIC	$\mu\text{g/mL}$	15560	15261	11293	11408
OH ⁻	$\mu\text{g/mL}$	24010	22869	61386	63123
Al	$\mu\text{g/mL}$	872	928	967	1013
Fe	$\mu\text{g/mL}$	127	127	118	119
K	$\mu\text{g/mL}$	1543	1522	1766	1604
Na	$\mu\text{g/mL}$	212826	224293	268708	266391
S	$\mu\text{g/mL}$	2602	2560	2716	2704
^{239/240} Pu	$\mu\text{Ci/mL}$	0.012	0.012	0.015	0.015
²⁴¹ Am	$\mu\text{Ci/mL}$	0.026	0.033	0.013	0.029
⁹⁰ Sr	$\mu\text{Ci/mL}$	19.5	25.2	14.8	21.4