Transuranium Removal from Hanford AN-107 Simulants using Sodium Permanganate and Calcium

by

W. Wilmarth

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

S. W. Rosencrance

C. A. Nash

D. P. DiPrete

C. C. DiPrete

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Westinghouse
Savannah River Company
Aiken, SC 29808

October 26, 1999

BNF-003-98-0160A

Mr. Mike Johnson
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2940 George Washington Way
Richland, WA 99352

Dear Mike:

RPP-WTP PRIVATIZATION PART B, SRTC WFO-98-003, SUBMITTAL OF "TECHNICAL REPORT: TRANSURANIUM REMOVAL FROM HANFORD AN-107 SIMULANTS USING SODIUM PERMANGANATE AND CALCIUM, BNF-003-98-160, REV. 0"

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Sincerely,

AT Wach for H F. Sturm

Harold F. Sturm, Jr.
BNFL RPP-WTP Project Manager
Savannah River Technology Center

hfs/sgm
Att.
c: W. R. Wilmarth, 773-42A
C. A. Nash, 773-42A
S. W. Rosencrance, 773-42A
D. P. DiPrete, 773-41A
C. C. DiPrete, 773-41A
C. T. Randall, 773-42A
B. J. Skwarek, 773-41A
H. F. Sturm, 773-A
S. T. Wach, 773-42A
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W. R. Wilmarth
C. A. Nash
S. W. Rosencrance
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C. C. DiPrete

September 22, 1999

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Table of Contents

List of Figures

List of Tables

Transuranium Removal from Hanford AN-107 Simulants Using Sodium Permanganate and Calcium

W. R. Wilmarth, C. A. Nash, S. W. Rosencrance
Waste Processing Technology Section
D. P. DiPrete and C. C. DiPrete
Analytical Development Section
Savannah River Technology Center

Summary

Removal of strontium from the complexant-containing wastes (AN-102 and AN-107) had previously been acceptably accomplished by isotopic dilution. Actinide removal using ferric co-precipitation, however, was very problematic from both a processing and a decontamination standpoint. Therefore, a series of tests were performed to identify other potential actinide removal agents and to test these agents at various concentrations. The results of this work provide the following conclusions.

- Addition of sodium permanganate to an AN-107 simulant produced a filterable precipitate in the presence of the strontium isotopic dilution precipitate. Actinide decontamination factors can approach design requirements at high permanganate concentrations.
- A synergism exists between additions of calcium nitrate and sodium permanganate to dramatically increase the strontium and actinide decontamination factors without adversely affecting filterability of the precipitates.
- The kinetics of the actinide and strontium removal of the permanganate decontamination process is rapid at moderate temperature (50 °C).
- The relative amounts of precipitating agents and the required decontamination factors may indicate little or no calcium will be required, thus, minimizing cold chemical loading in the glass.

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 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 Herting[6] 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.

Previous studies at the Savannah River Technology Center (SRTC)[8,9] examined various aspects of the precipitation process such as addition rate, reagent concentrations, free hydroxide level, organic complexant concentration, and temperature using simulated waste solution (see Appendix 1 for composition). The results of these studies gained insight into the chemistry affecting the effectiveness of the precipitation in terms of decontamination and filterability. However, the presence of the organic complexing agents renders the ferric precipitates poorly filterable.

SRTC has performed testing of alternative technologies for the removal of transuranium elements from the chelant-containing wastes. The basis for the work was predominately of Orth.[7] The decontamination agents used in these experiments were mixtures of ferric/ferrous, nickel (II), cobalt (II), cobalt (III), lanthanum,[8] and uranyl ion.

Additionally, the application of freshly prepared manganese oxide surfaces has been shown to selectively adsorb lanthanides,[11] actinides,[12] and other radionuclides.[13,14] The manganese dioxide produced by the reduction of permanganate has also been shown to adsorb organic molecules from aqueous solution.[15] This adsorption property could potentially be used in the decontamination of the chelant-containing wastes at Hanford. Of further interest is the synergistic effect of calcium on the adsorption of organic material on manganese dioxide in wastewater clarification processes.[16] Therefore, a series of experiments were conducted to examine these effects on transuranium decontamination of an AN-107 waste simulant.

Experimental

Approximately 50 mL of the simulant (Appendix 1) was placed into a 200-mL beaker or 125-mL serum vial. 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 was added. 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, heated for a specified time in either the water bath or in a temperature-controlled oven. The samples were 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. After the samples had cooled, the remaining slurry was filtered. Samples were submitted to the Analytical Development Section of SRTC for analyses.[17] The analyses included liquid scintillation analysis for strontium-90[18] and alpha pulse height analysis.[19]

In performing the plutonium analyses, the samples were subjected to a thenoyltrifluoroacetone (TTA) separation. An aliquot of the sample was initially spiked with a Pu-238 tracer. All of the plutonium in the sample was reduced, an anion complexing agent (aluminum nitrate) was then added. The plutonium in the sample was then oxidized to a +4 oxidation state. The plutonium (IV) was then extracted from the

matrix using a TTA solution. The TTA layer was mounted in a counting dish and analyzed by alpha spectroscopy. A blank sample was run with the batch of samples.

Table 1. Experimental Conditions

Sodium Ion Concentration	5.5 M
Hydroxide Ion Concentration	1.0 M
Aluminate Ion Concentration	0.4 M
Temperature	50 °C

The analysis results for the Pu-239 alpha peak were yielded using the Pu-238 recoveries from the Pu-238 tracer solution. As the Pu-239 spike solution used was a sample of weapons grade plutonium , a correction was required for the Pu-238 activity inherent to the weapons grade material. A sample of the stock weapons grade plutonium was analyzed for a Pu-239/240 to Pu-238 ratio. This ratio was used to correct the separation yields.

Results and Discussion

Addition Ferric and Ferrous Precipitation Studies

The results from the statistically designed experiments showed filterability and decontamination for the strontium nitrate/ferric nitrate to be too low for practical use. The BNFL Inc. basis of design for the filtration process assumes a filter flux rate of ≥ 1 gpm/ft^2 can be achieved using 0.1 μm sintered metal filter elements. The use of other iron precipitating agents may prove more useful. Therefore, a series of tests were conducted to determine if mixed valence precipitates of iron were conducive to use in the Sr/TRU removal step. Additionally, oxalic acid and lanthanum nitrate reagents were examined as possible replacement of the ferric nitrate. In these studies, the focus was actinide removal; therefore, no strontium isotopic dilution was performed.

Figure 1 contains the decontamination data calculated from the Sr-85 and Am-241 radiotracer measurements. The decontamination factor (DF) required for the flowsheet is 5. The ferric/ferrous mixture did not enhance the americium removal. The mixed valence precipitate was magnetic and did filter slightly better. The inability of iron (III) to affect an actinide DF directs the research to other agents. The inability of lanthanum

(III) to displace the americium ions from the complexing agents is a little surprising based on the higher charge density of La (III) vs. Am (III).
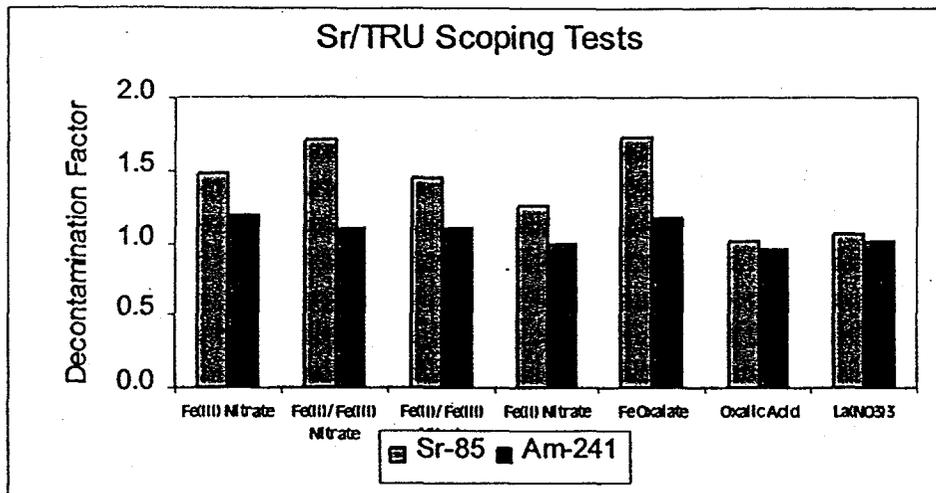
Figure 1. Decontamination Factors for Ferric/Ferrous Precipitations

Effect of Other Transition Metal Agents and Calcium

The ethylenediaminetetraacetic acid (EDTA) complexing agent is well known in the inorganic literature to bind to many different transition metals such as Ca (II), Fe (II), Ni (II) and Co(III). If the actinide is complexed by EDTA these metals could potentially be effective decontamination agents. Therefore, metals having high affinity for EDTA may remove the actinides from solution by substitution. A series of tests were performed to measure actinide and strontium DFs for a variety of agents. The filterability of the resultant precipitates was also measured. These tests used a strontium nitrate addition to aid the Sr-85 decontamination.
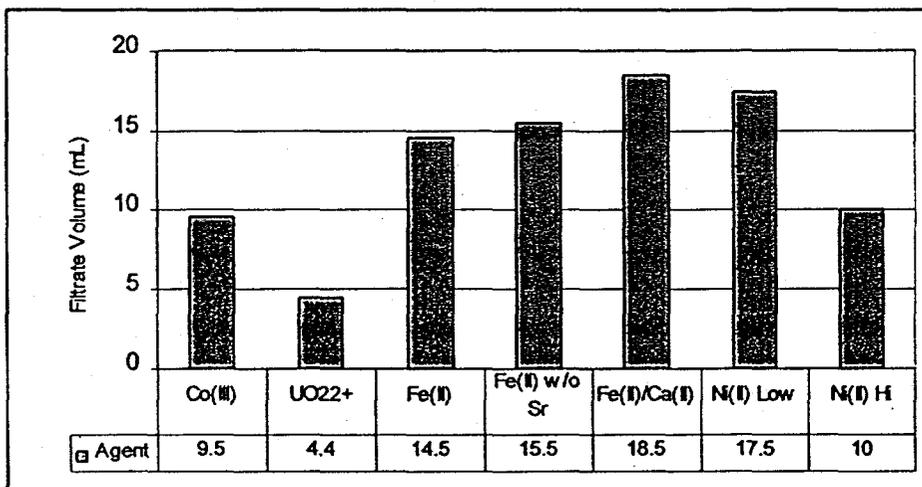
Figure 2. Filtration Volumes from Transition Tests

Figure 2 shows the filtration data collected in a one minute interval from the tests employing Fe (II), Co (III), Ni (II) and uranyl ion. Filtration volumes ranged from 4.4 mL to 18.5 mL. These fluxes are low and probably unsuitable for use in the decontamination process. However, the ferrous tests show that the addition of calcium (0.0375 M) does not significantly decrease the filtration flux, when combined with the ferric nitrate.

The decontamination factors for Sr-85, Pu-239 and Am-241 are shown in Figure 3. Strontium decontamination was successfully attained by isotopic dilution for all of the samples. The Sr DFs for the nickel experiments were multiplied by 0.1 to fit on the scale. The actual DFs were 97.7 and 98 for the low concentration (0.0375 M) and the high concentration (0.075 M), respectively. The actinide factors were low with the exception of the calcium (0.0375 M) and ferrous (0.0375 M) mixture. The calcium addition increased the DF for the actinide from 1.2 to 8 – 10. Both americium and plutonium were almost equally affected by this addition. Because of the low filterability of this precipitate the use of this mixture is not very attractive. However, the use of calcium addition to sequester the chelant may have other uses.
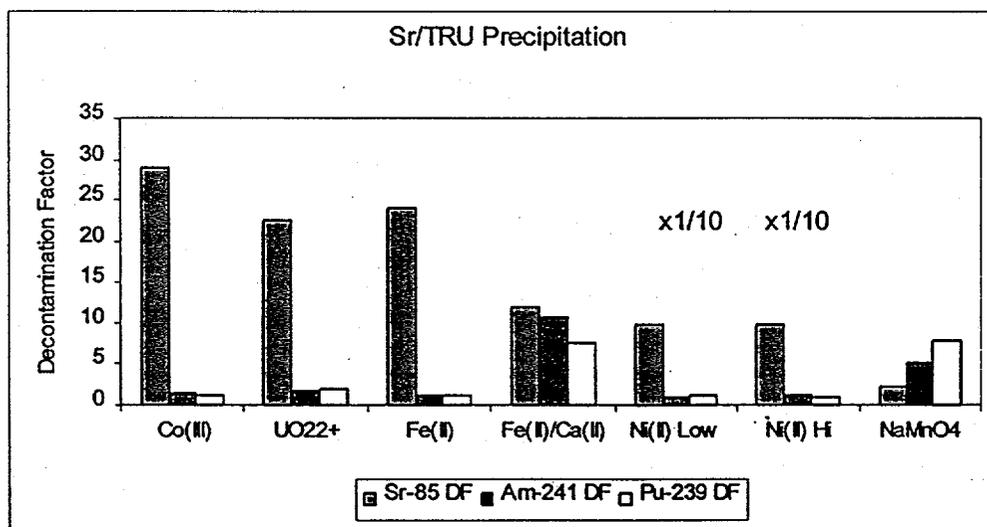
Figure 3. Decontamination Factors for Other Transition Metals

Initial Testing with Sodium Permanganate and Calcium Decontamination

Orth[7] had originally examined the use of potassium permanganate for strontium decontamination of a 101-SY simulant and found decontamination factors on the order of 10 – 20. A permanganate test with an actual sample from 101-SY showed plutonium decontamination in addition to the strontium removal. The applicability of this process to the complexant concentrated waste (AN-102 and AN-107) was worthy of testing.

Shown in Figure 4 are the filtrate volumes (mL) obtained after four separate tests of adding various levels of calcium and sodium permanganate to the AN-107 simulant along with the 0.075 M strontium nitrate isotopic dilution. These volumes were obtained after one minute of filtering through a 0.45-micron cellulose nitrate filter. Filtrate volumes were high and range from 9 to 25 mL. The precipitate prepared by the addition of 0.2 M $Ca(NO_3)_2$ filtered the best. Addition of 0.075 M $NaMnO_4$ reduced the filterability (16 mL) in reference to the calcium precipitate. Addition of 0.0375 M calcium and 0.075 M permanganate exhibited the same filterability as the 0.075 M permanganate alone. Ironically, dramatically reducing the calcium and permanganate with the 0.075 M strontium caused the solution to filter relatively poorly.

	0.075 NaMnO4	0.0375 M Ca + 0.075 M NaMnO4	0.2 M Ca	0.004 M Ca + 0.01 M NaMnO4
Series1	16	15.5	25	9
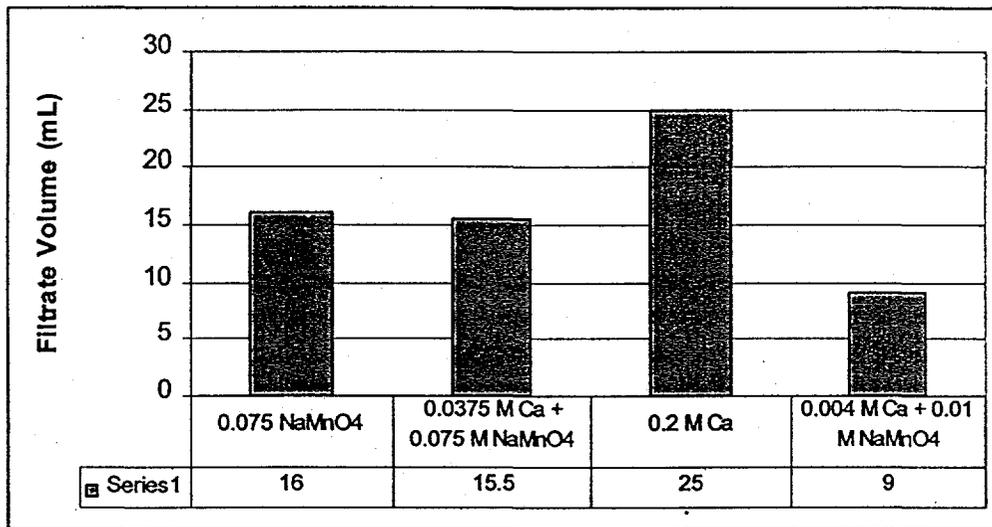
Figure 4. Filterability of Permanganate and Calcium Precipitates

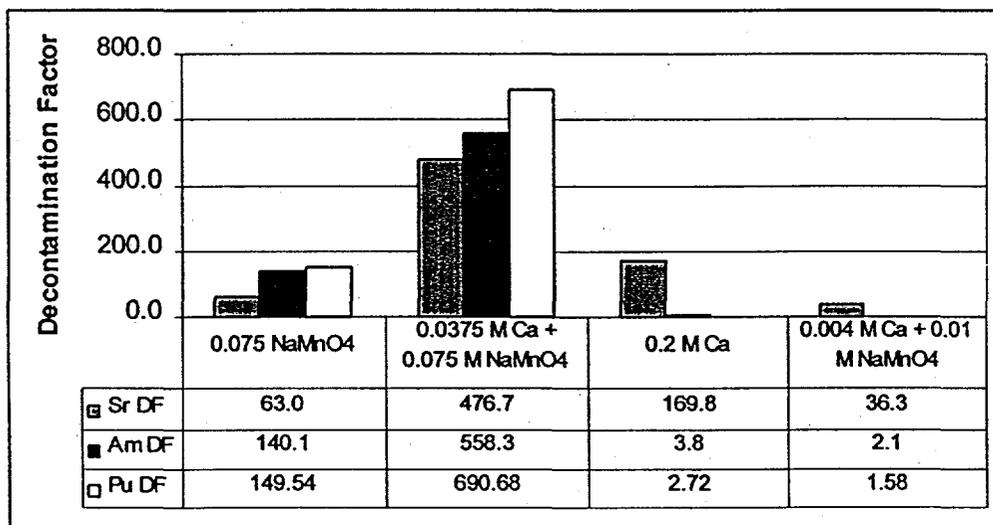|	0.075 NaMnO4	0.0375 M Ca + 0.075 M NaMnO4	0.2 M Ca	0.004 M Ca + 0.01 M NaMnO4
Sr DF	63.0	476.7	169.8	36.3
Am DF	140.1	558.3	3.8	2.1
Pu DF	149.54	690.68	2.72	1.58

Figure 5. Decontamination Factors for Permanganate and Calcium Additions

The decontamination factors calculated from the measured Sr-85, Am-241 and Pu-239 concentrations are shown in Figure 5. Remembering the required DFs are 10 for Sr and 5 for Am and Pu, the DFs obtained from the addition of 0.075 M sodium permanganate far exceed the requirements. The DFs obtained were 60, 140 and 150 for Sr, Am and Pu, respectively. The addition of 0.2 M calcium nitrate to the solution along with the strontium isotopic dilution gave acceptable DF for Sr (170) but unacceptable DF for Am (3) and Pu (2.7). However, the combination of calcium and permanganate along with the strontium dilution exhibited a very large DF for each species. This synergism between Ca, Sr and MnO_4^- gave DFs of 470 for Sr, 550 for Am and 690 for Pu. A subsequent test at one tenth the addition level for calcium (0.004 M) and permanganate (0.01 M) showed greater than an order of magnitude reduction in the actinide DF. These results along with the good filterability data suggest the addition of calcium and permanganate along with the strontium isotopic dilution could be a potential replacement for the strontium and ferric nitrate flowsheet for treating the complex concentrate waste (AN-102 and AN-107 tanks).

Additional Calcium/Permanganate Tests

The results of the initial tests on calcium and permanganate addition for actinide removal and strontium addition for isotopic dilution for strontium removal were very encouraging. Therefore, additional tests were conducted at two calcium levels (0.0375 M and 0.02 M) along with three permanganate levels (0.02 M, 0.04 M, and 0.06 M) to examine filterability and decontamination levels in order to begin to optimize the minimum cold chemical feed. The results of the filtration data are shown in Figure 6. The filtrate volumes obtained after one minute through a 0.45 micron filter are the highest obtained during the Sr/TRU precipitation testing. The volumes were very near 30 mL or approximately 60 % of the liquid that was clarified in one minute.
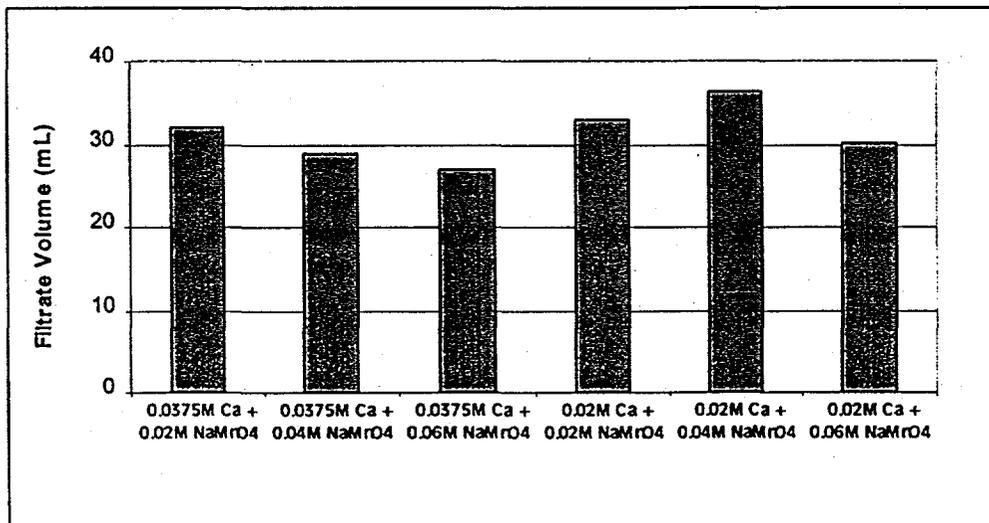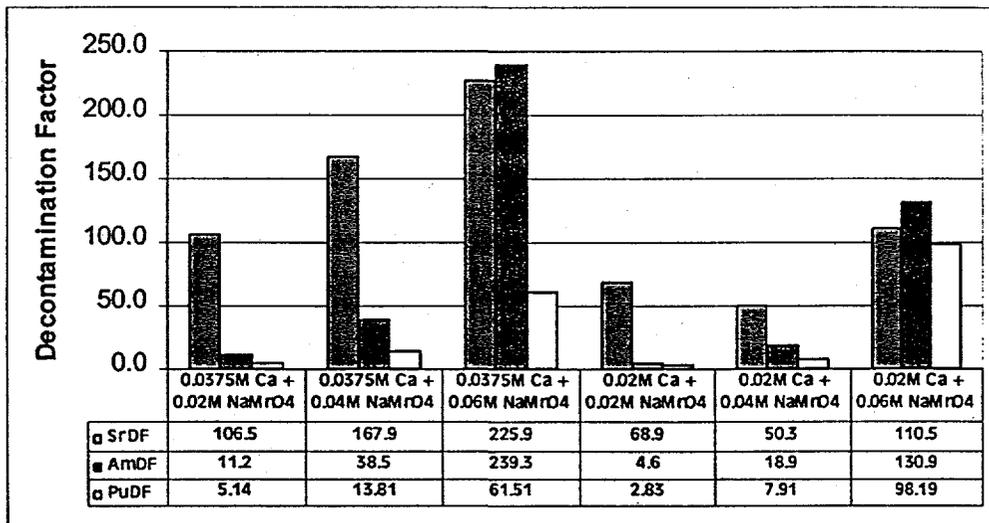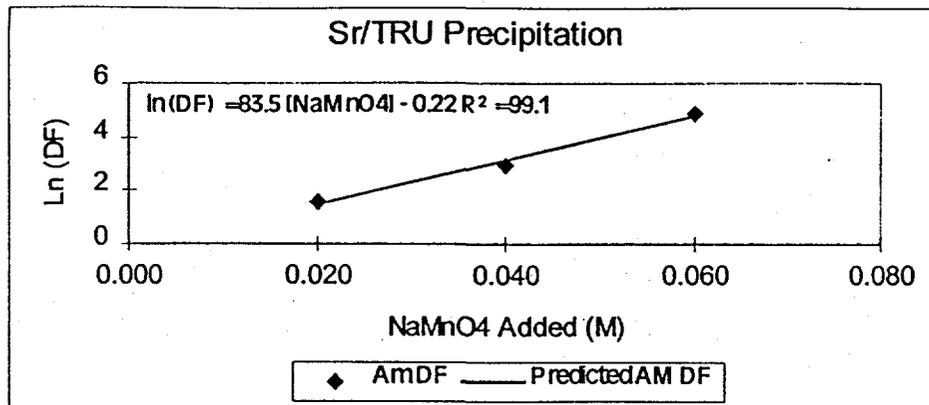

Figure 6. Filtrate Volume (mL) from 2 Level Calcium Tests

Figure 7 contains the decontamination data for the addition calcium and permanganate tests. The tests were conducted at the high calcium level (0.0375 M) exhibit sufficient DF for all of the species (Sr, Am, and Pu). There is a direct relationship between the DF observed for each species with increasing permanganate. Increasing the permanganate concentration to 0.06 M raised the americium DF to ~240 and the plutonium DF to ~60. The strontium DF increased with increasing permanganate concentration; however, the effect was not as large. At the lower calcium level (0.02 M), the same effects are observed. Actinide DFs increase with increasing permanganate concentration. Acceptable DFs for each species were obtained at mid permanganate concentration (0.02 M Ca and 0.04 M MnO_4^-). Shown in Figure 8 is a statistical fit of the of the ln (DF) for americium as a function of permanganate concentration. Using this equation, the permanganate concentration needed to obtained the required americium DF (5) would be 0.022 M sodium permanganate at the 0.02 M calcium concentration.

	0.0375M Ca + 0.02M NaMnO4	0.0375M Ca + 0.04M NaMnO4	0.0375M Ca + 0.06M NaMnO4	0.02M Ca + 0.02M NaMnO4	0.02M Ca + 0.04M NaMnO4	0.02M Ca + 0.06M NaMnO4
□ SrDF	106.5	167.9	225.9	68.9	50.3	110.5
▪ AmDF	11.2	38.5	239.3	4.6	18.9	130.9
□ PuDF	5.14	13.81	61.51	2.83	7.91	98.19

Figure 7. Decontamination Factors for Additional Calcium and Permanganate Tests

These tests clearly show the positive effect of increased permanganate addition for the removal of actinide species from the AN-107 simulant. Additional examination of the data shows a direct influence of the calcium concentration. For each permanganate level (0.02, 0.04 and 0.06 M), the calculated ratio of the actinide DF at 0.0375 M Ca to the DF at 0.02 M Ca is shown in Table 2. With the exception of the plutonium ratio at the very high permanganate level (0.06 M), reducing the calcium level by 2 reduced the actinide DF by ~2.

Sr/TRU Precipitation

$$\ln(DF) = 83.5\,[NaMnO4] - 0.22 \quad R^2 = 99.1$$

Ln (DF) vs NaMnO4 Added (M)

AmDF ——— Predicted AM DF

Note: Calcium level is 0.02 M.

Figure 8. Statistical Fit of Am DF Data

Table 2. Comparison of Actinide DFs at Two Calcium Levels
DF Ratio for 0.0375 M Ca to 0.02 M Ca

Permanganate Level	Am	Pu
0.02 M	2.4	1.8
0.04 M	2.0	1.7
0.06 M	2.2	0.6

Rate of Actinide Removal in Calcium/Sodium Permanganate Decontamination

The rate of removal of the actinides is important to understand to support engineering and design of the plant. All of the pervious tests were conducted for 4 hours with sampling only at the end of the experiment. Therefore, a kinetic study was performed using the 5.5 M sodium AN-107 simulant. The level of strontium, calcium and permanganate additions was 0.075 M, 0.0375 M and 0.04 M, respectively. The temperature was 50 °C.

Shown in Figure 9 are the Sr-85 and Am-241 concentrations in the liquid phase plotted as a function of time. There is a very rapid drop in the radionuclide concentration in the

first 0.5 hours. A slower, steady reduction in the radionuclide concentration is then observed for the remainder of the test. There is no visible evidence of permanganate existing after addition. It appears as though the permanganate reacts and the freshly produced solids continue to absorb the radionuclides. The adsorption is very linear as can be seen in Figure 10. An additional order of magnitude of the DF is obtained for each additional hour at 50 °C. This behavior is typical of an adsorption process. Serrano and Garcia[11] had experimentally measured Ce-141 Freundlich adsorption isotherms. The Sr-85 and Am-241 behavior is probably very similar.
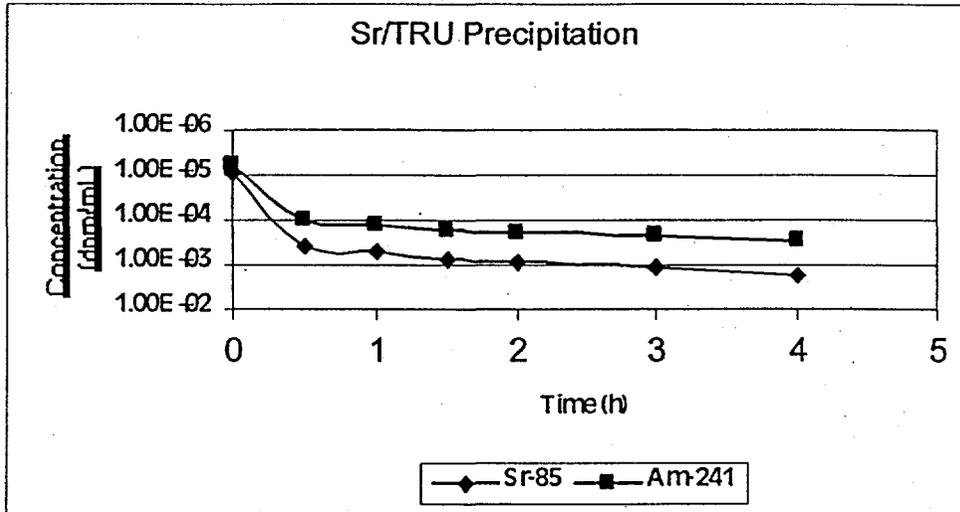
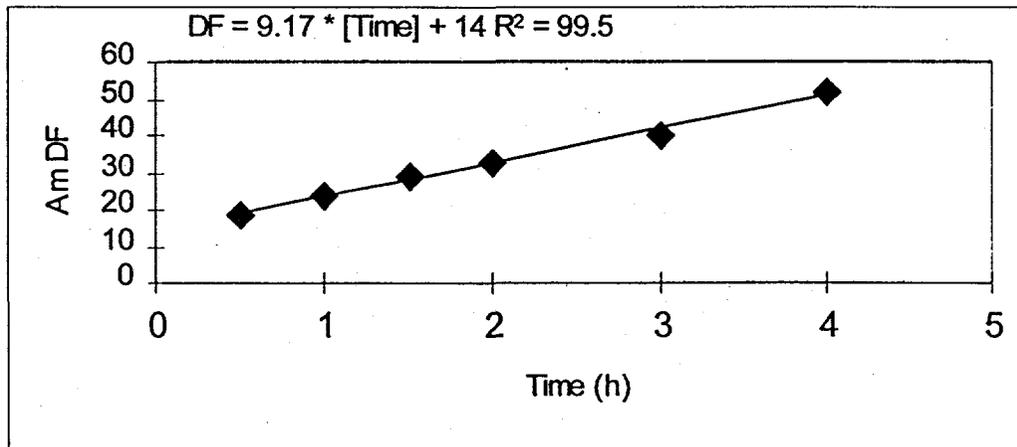Figure 9. Kinetic Data for Permanganate Decontamination

Figure 10. Statistical Analysis of Am DF

Conclusions

Removal of strontium from the complexant-containing waste (AN-102 and AN-107) had previously been acceptably accomplished by isotopic dilution. Actinide removal using ferric co-precipitation, however, was very problematic from both a processing and a decontamination standpoint. Various other transition metals were examined for replacement of the ferric hydroxide precipitation.

This work has identified and tested a sodium permanganate reaction that produces a precipitate that is readily filterable and provides sufficient decontamination of the actinides from an AN-107 simulant at a modest operating temperature (50 °C). Additionally, the work has identified a positive synergist effect of adding calcium nitrate prior to the addition of sodium permanganate. This synergism can increase both the actinide DF and strontium DF. Optimization of this process could potentially lead to a reduced cold chemical feed while meeting decontamination requirements and within the flowsheet time restraints.

The following recommendations arise from the results of this work:
- Testing with actual tank waste samples should be conducted to verify observations/test results with simulated waste solutions.
- Effects on glass composition should be evaluated for this precipitation process.
- BNFL Inc. must store the Sr/TRU precipitate for a period of time before blending with the high level waste sludge. Therefore, the long-term stability of the SR/TRU precipitate should be evaluated.

Acknowledgements

The authors wish to acknowledge the help and assistance provided by the Analytical Development Section in their analysis of the many samples submitted during this work. Additionally, Vicki Dukes in the preparation and handling of the samples performed an excellent job.
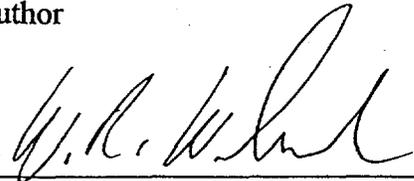
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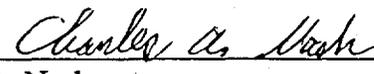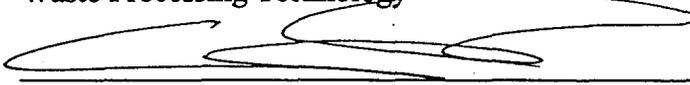
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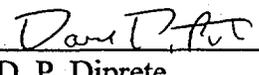_____   _____
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W. D. King   Date
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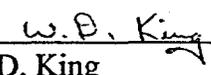
Management
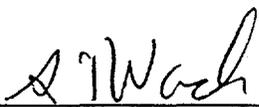
signature   10/25/99
_____   _____
S. T. Wach, Manager   Date
External Programs

signature   _____
_____
M. E. Johnson, Technical Representative   Date
BNFL, Inc.
                                    ATW 10/25/99

Appendix 1. AN-107 Simulant Composition

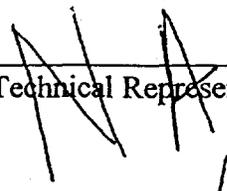| Component	Molecular weight	Concentration	Units	Concentration	Units
Acetate	59.04462	1100	mg/Liter	1.86E-02	M
Aluminum	26.98154	386	mg/Liter	1.43E-02	M
Ammonium	18.03846	22	mg/Liter	1.22E-03	M
Barium	137.33	7	mg/Liter	5.42E-05	M
Boron	10.81	35	mg/Liter	3.24E-03	M
Bromide	79.904	1150	mg/Liter	1.44E-02	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
Ethylenediaminete traacetic 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

W. R. Wilmarth, et al.

BNF-003-98-0160

BNF-98-003-160

Page 20 of 20

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 | 39265 mg/Liter | 0.018 M |