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Effects of Ammonium Molybdophosphate (AMP) on Strontium, Actinides, and RCRA Metals in SRS Simulated Waste

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LIST OF ACRONYMS

AA	Atomic Absorbtion
ADS	Analytical Development Section
AMP	Ammonium MolybdoPhosphate
DF	Decontamination Factor
HLW	High Level Waste
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
K_d	Distribution Coefficient
MDL	Method Detection Limit
NaOH	Sodium Hydroxide
Pu-TTA	Plutonium triphenoyltrifluoroacetone extraction
RCRA	Resource Conservation Recovery Act
SRS	Savannah River Site
SRTC	Savannah River Technology Center
WAC	Waste Acceptance Criteria

1.0 EXECUTIVE SUMMARY

High Level Waste samples contain elevated concentrations of radioactive cesium requiring marked dilution of the waste to facilitate handling in non-shielded facilities. The authors developed a sample treatment protocol, using ammonium molybdophosphate (AMP) to remove sufficient cesium to allow handling of the samples with minimal dilution. The sample treatment protocol includes the following steps.

- pH adjust the sample to the range of 0.01 to 1.0 M acidity
- Mix 30 mL of acidified sample with 40-60 mg of AMP
- Cap and shake the mixture for 30-60 seconds
- Filter AMP from the liquid using 0.45 µm PTFE syringe filters
- Send filtrate directly forward for analysis

To develop the method, SRTC performed a series of tests with three different salt solutions designed to determine the propensity of ammonium molybdophosphate (AMP) to bind some of the common analytes such as the actinides (Pu, Am, Np, U), strontium, or the metals (Ag, As, Ba, Cd, Cr, Hg, Pb, Se) regulated by the Resource Conservation Recovery Act (RCRA). SRTC also examined relevant literature to summarize reported interactions between AMP and other elements.

- Within the protocol conditions, AMP exhibited no appreciable affinity for plutonium, neptunium, uranium and strontium.

- In this testing, AMP showed a possible minor affinity for americium; however, the data is not as clear due to continued americium solubility changes during the duration of the experiment.

- Of the eight RCRA elements (Ag, As, Ba, Cd, Cr, Hg, Pb, Se) studied, AMP exhibited affinity for only silver under our experimental conditions.

- Under our experimental conditions, AMP has an affinity for rubidium.

- AMP showed no affinity for potassium under the test conditions, although the literature suggests that AMP does possess some affinity for potassium.

- The use of AMP can clearly provide a benefit for those analytical procedures that do not require dilution of the original sample. Radiochemistry is the prime beneficiary. The effect on ICPMS, ICPES, or AA, which require internal dilutions to reduce salt content, is harder to determine at this time. The results of real waste tests will allow a judgment in those cases.

- The AMP treatment protocol allowed accurate measurement of non-radioactive strontium at concentrations above 0.07 mg/L using inductively coupled plasma mass spectroscopy. The analytical method yielded a +15% bias when the concentration fell to 0.007 mg/l.

- Tests results found during the literature search indicate that cerium, yttrium, europium, thallium, americium, silver, and possibly mercury (I) have or may have some affinity for AMP. However, these tests allowed extended duration contact of the solution with AMP. For the shorter contact times inherent in this sample treatment protocol, the AMP may not significantly remove these species. As yttrium and europium are examples of rare earth and lanthanide elements, respectively, AMP likely has an affinity for other rare earths and lanthanides.

- We recommend examining the AMP protocols used in actual waste testing done in the EPC vendor support work when the results become available. At that time, the present work (simulant) and real waste results can be compared to insure there are no offsets. Furthermore, the actual waste testing will increase the number of elements for which we can explicitly determine for the presence of AMP affinity. We also recommend an additional small battery of simulant tests to confirm if AMP has an affinity for potassium or selenium.

2.0 INTRODUCTION

The work described in this document responds to a DOE request in support of technical needs expressed, in part, by the Engineering, Procurement, and Construction Contractors for the Salt Waste Processing Facility. The DOE authorized the work within Budget Change Package CR030338, “SPP Scope of Work: EPC Vendor Support” (April, 2003). The tasks specified by this request follow.

- AMP Method Development – For appropriate samples or programs, develop ammonium molybdophosphate as a cesium removal agent for high activity wastes to avoid large sample dilutions necessary for ALARA reasons when processing samples. Results from this task will be applied towards the following two tasks.

- Supernate Sample Analyses – Acquire and analyze supernate samples from seven High Level Waste tanks to better understand the composition of feed planned for the Salt Waste Processing Facility.

- MST Multi-strike Demonstration – Determine the effect of increased MST addition (up to 1.2 g/L) and benefit of extra filtration steps with multiple additions of MST to salt waste containing actinides and strontium.

Researchers wrote a task plan [1] that defined the scope of work for all three tasks. However, the work described in this document addresses only the first of the tasks above; the AMP Method Development.

Virtually all samples of high level radioactive waste require large dilutions before being handled outside of shielded cell facilities. This large dilution is a source of difficulty for obtaining accurate actinide analyses. This dilution proves necessary for ALARA reasons; undiluted samples usually exceed the 10 mrem at 30 cm administrative limit. In most cases, the high activity results from gamma activity of ^{137}Cs present in most SRS waste solutions. Recently, personnel began using AMP to remove cesium in an attempt to minimize the required dilution.[2,3,4] The initial attempts showed variable results but demonstrated the feasibility of the method. Personnel selected this approach due to the high selectivity of AMP resin for cesium with indications of negligible sorption of strontium and the actinides.[5] This work involved the use of AMP, although the other form, AMP-PAN (that is AMP bound in polyacrylonitrile) functions identically.

3.0 EXPERIMENTAL

3.1 Solution Preparation and Equilibrium

Researchers prepared three different salt solutions based on previous simulant recipes. The “High Hydroxide” and “High Nitrate” recipes derive directly from previous work, while the “High Potassium” recipe represents a variation of the “SRS Average Waste” recipe.[6] The amount of potassium (0.1 M) in the High Potassium recipe represents a challenge to solvent extraction because it can cause formation of a third phase.[7] Due to this effect, SRTC has value in determining if the use of AMP will improve measurements of potassium in tank waste. Table 1 lists the components of each of the three salt solutions. The order of the species listed in Table 1 is the order ion which personnel added the material to the salt solution during preparation. During the solution preparation technicians added the radionuclides along with the cold chemicals. Technicians omitted non-radioactive (i.e., “cold”) strontium since the bulk reagents typically contain enough tramp strontium for testing (with a target of 800 μg/L). Technicians made the solutions in 250 mL poly bottles and sealed them to prevent influx of atmospheric carbon dioxide. Once made, personnel allowed the solutions to equilibrate for seven weeks to insure that the actinides and strontium reached solubility equilibrium. Technicians filtered the solutions with a 0.1 μm PES filter cup after three weeks to eliminate any insoluble species. During six of the seven weeks, personnel collected samples from each bottle. They filtered the samples, acidified with nitric acid, and analyzed by radiocounting and Inductively Coupled Plasma Mass Spectroscopy (ICPMS) for the actinides and strontium to observe the approach to equilibrium.

3.2 General Procedure Using AMP

All work that used the AMP followed the same general procedure. For this work, personnel adjusted aliquots of salt solutions with nitric acid until reaching the requisite pH range (1-2), keeping track of the effective dilution. The molarity of acid used depended on the salt solution and the dilution (Table 2). Technicians allowed the samples to digest for at least two hours before proceeding. Then, they contacted 30 mL of the pH adjusted solution with a small quantity (~60 mg) of AMP. After vigorously shaking for 30 seconds, technicians filtered to remove the AMP using a 0.45 µm PTFE syringe filter and prepared the resulting filtrate for analysis. Personnel also analyzed control samples without AMP treatment (but still filtered) in the same exact manner as the other samples.

Table 1. Simulant Compositions

	Target Concentrations		
Species	High Hydroxide	High Nitrate	High Potassium
$NaAlO_2$	0.31	0.37	0.36
^{238}U	10 mg/L	10 mg/L	10 mg/L
$^{239/240}Pu$	200 ug/L	200 ug/L	200 ug/L
^{237}Np	500 ug/L	500 ug/L	500 ug/L
^{85}Sr	1.6E-03 ug/L	1.6E-03 ug/L	1.6E-03 ug/L
$NaNO_3$	1.2	3.3	2.5
NaCl	0.012	0.0460	0.029
NaF	0.012	0.0575	0.037
Na_2HPO_4	0.0092	0.0115	0.012
$Na_2C_2O_4$	0.0092	0.0092	0.0092
Na_2SiO_3	0.0046	0.00460	0.0046
Na_2MoO_4	0.00023	0.00023	0.00023
KNO_3	0.035	0.00472	0.10
NaOH	3.5	1.35	2.2
Na_2CO_3	0.20	0.184	0.18
Na_2SO_4	0.035	0.253	0.17
$NaNO_2$	0.85	0.426	0.60
CsCl	7.5E-05	7.50E-05	7.0E-05
^{241}Am	20 ug/L	20 ug/L	20 ug/L
Na^+ (calc)	6. 5	6. 5	6. 5

[a] Added as $Al(NO_3)_3 \cdot 9H_2O$.

Success for the treatment protocol is defined as proving minimal uptake of non-cesium elements, while at the same time demonstrating superior instrument sensitivity through the use of AMP. The literature indicates some uptake of plutonium and even greater sorption of americium; however, we minimized this sorption by limiting the contact time with the resin.[8]

Table 2. Acid Molarity Used in Each the Dilutions

Salt Solution	Acidity of HNO3 Used (M)	
	2× Dilution	10× Dilution
High Hydroxide	5.7	0.63
High Nitrate	3.5	0.40
High Potassium	4.4	0.49

3.3 Radionuclide Testing

The task used three simulated high-level waste solutions traced with the radionuclides of interest. As such, the development provided a limited investigation of the influence from variations in the radionuclide concentrations and the changes in bulk solution chemistry (e.g., High Hydroxide versus High Nitrate versus High Potassium). Researchers used solution recipes defined in previous work (Table 1). The simulant included radioisotope concentrations of ^{238}U (UO_2^{2+}) at 10 mg/L, $^{239/240}$Pu (IV) at 0.2 mg/L, ^{85}Sr at 0.6 mg/L, ^{237}Np (V) at 0.4 mg/L, and ^{241}Am (III) at 0.04 mg/L. Personnel also added non-radioactive cesium (7.5E-05 M).[9]

For this work, the experiments examined the effects of 2× and 10× dilutions as part of the AMP treatment. We based these dilutions on prior trials that indicated these are the minimal dilutions necessary for ALARA control and for adequate acidification of the samples. Measurements for each of these solutions and dilutions occurred in triplicate.

3.4 RCRA and Non-radioactive Element Testing

A risk, exists that AMP will show an affinity for elements in a RCRA or Waste Acceptance Criteria (WAC) analysis (i.e., Ag, As, Ba, Cd, Cr, Hg, Pb, Se). SRTC recommended performing Inductively Coupled Plasma-Emission Spectroscopy (ICP-ES), Atomic Absorption (AA), and Cold Vapor Mercury Atomic Absorption (CVHg-AA) on selected samples to mitigate this risk. For this work, researchers prepared a quantity of High Potassium simulant, but without the radioisotope spikes (so we could work with the materials in a chemical hood). They spiked the simulant with 100 mg/L each of the eight RCRA elements. Technicians allowed the solution to equilibrate for four weeks before filtering through a 0.2 μm filter cup to remove solids. Two weeks after filtration, researchers performed an AMP test in the same general manner described previously.

In addition to the RCRA element analysis, the authors decided to examine the effect of AMP on any other element measured in the requested analyses. The ICPES and ICPMS data allowed us to examine the effects of AMP on several elements besides the RCRA elements.

3.5 Strontium and Cesium Testing

For the final part of the AMP treatment, researchers examined a series of successive dilutions on one of the AMP-treated simulant solutions to determine the effects of dilutions on detection limits via ICP-MS for non-radioactive strontium. While the researchers did not explicitly add non-radioactive strontium, the simulated waste contained tramp strontium, allowing the researchers to examine isotopic determinations using ICPMS.

3.6 Literature Review of AMP Data

The authors performed a literature search to determined past evidence of AMP removing various elements. The results of this search are summarized in section 4.5.

4.0 EXPERIMENTAL RESULTS

4.1 Solution Preparation and Equilibrium

During the seven week equilibrium period, researchers sent filtered and acidified samples of each of the three simulant solutions for actinide and strontium analysis. $^{239/240}$Pu measurement occurred by plutonium triphenoyltrifluoroacetone extraction analysis (PuTTA) and counting; ^{238}U and ^{237}Np values come from ICPMS, while ^{241}Am and ^{85}Sr derive from gamma spectroscopy. We did not collect samples after six weeks of equilibration. Personnel filtered the bulk solutions after the third week's samples to ensure that no insoluble solids remained in solution. Table 3 and Figures 1-5 display the radioisotope content of filtered samples from each of the simulant solutions. Plutonium, neptunium, uranium, and strontium all showed comparable concentrations throughout the equilibration period. Neither time nor the filtration before the fourth weeks sample appeared to influence the concentrations in solution. For these radionuclides, equilibrium occurred quickly and the concentrations remains stable. Americium proved much less stable and soluble in solution. Each of the three solutions experienced a rapid decline in americium concentration in solution, which slowed during the last three samples. The last data point in the High Nitrate and two of the last data points in the High Potassium solutions had americium concentrations less than the method detection level (0.0821, 0.0503, 0.0829 nC/g, respectively). We originally estimated that the amount of americium added to the solution (8.05E-08 M) would fall below the solubility limit (1.6E-06 M [10]), and should not precipitate from solution. The High Hydroxide had a slightly lower ionic strength than the other two solutions; this may account for it's higher americium solubility.

Table 3. Radioisotope Values During Equilibrium for Each Simulant

Simulant Solution	Time (weeks)	Pu-239/240 Activity (nCi/g)	Np-237 Activity (nCi/g)	U-238 Activity (mg/L)	Sr-85 Activity (nCi/g)	Am-241 Activity (nCi/g)
High Hydroxide	1	1.14E+01	2.65E-01	1.01E+01	2.29E+01	1.30E+01
	2	1.19E+01	2.52E-01	1.00E+01	2.64E+01	4.03E+00
	3	1.14E+01	2.64E-01	1.03E+01	2.68E+01	3.82E+00
	4	1.22E+01	2.84E-01	1.06E+01	2.49E+01	2.78E+00
	5	1.18E+01	3.19E-01	1.15E+01	2.60E+01	1.78E+00
	7	1.22E+01	3.09E-01	1.10E+01	2.52E+01	1.13E+00
High Nitrate	1	1.10E+01	2.42E-01	1.05E+01	2.19E+01	9.91E+00
	2	1.14E+01	2.55E-01	1.04E+01	2.38E+01	2.00E+00
	3	1.12E+01	2.50E-01	1.04E+01	2.39E+01	7.73E-01
	4	1.17E+01	3.01E-01	1.15E+01	2.24E+01	4.22E-01
	5	1.19E+01	3.01E-01	1.14E+01	2.28E+01	2.89E-01
	7	1.15E+01	3.04E-01	1.12E+01	2.20E+01	<8.21E-02
High Potassium	1	1.11E+01	2.44E-01	9.98E+00	2.22E+01	5.33E+00
	2	1.10E+01	2.49E-01	1.10E+01	2.55E+01	9.63E-01
	3	1.13E+01	2.52E-01	1.02E+01	2.53E+01	4.71E-01
	4	1.14E+01	3.10E-01	1.10E+01	2.38E+01	2.23E-01
	5	1.18E+01	3.00E-01	1.16E+01	2.36E+01	<5.03E-02
	7	1.11E+01	2.87E-01	1.11E+01	2.39E+01	<8.29E-02

Figures 1-5 contain analytical error bars, but in the case of the plutonium, americium, and strontium, the errors are too small to easily see in the graphs (typically 2-5% uncertainty). The analytical uncertainty for the uranium and neptunium – via ICP-MS – analyses is a constant 15%. The red horizontal bars indicate the target concentrations for each radioisotope; prior solubility data prohibited making a reliable prediction for americium and hence Figure 4 does not include a target line.

Figure 1. Plutonium-239/240 in Solution in Each Simulant During Equilibrium

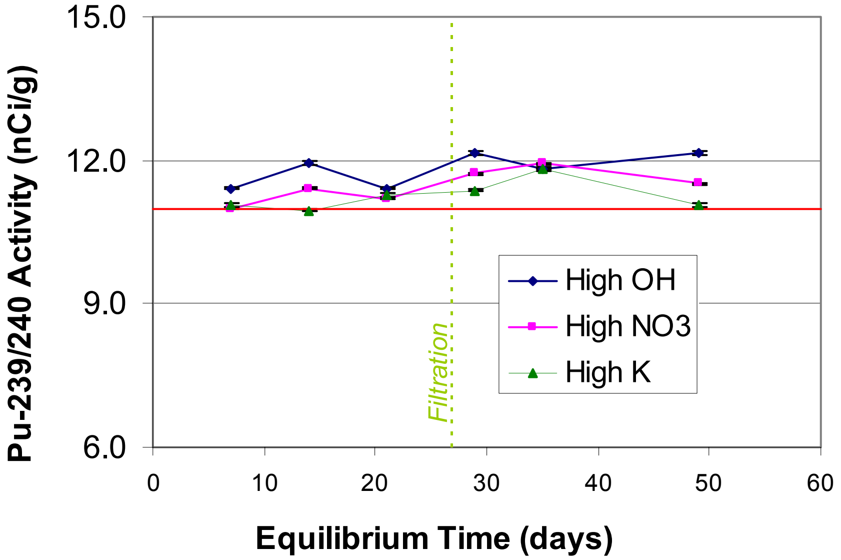


Figure 2. Uranium-238 in Solution in Each Simulant During Equilibrium

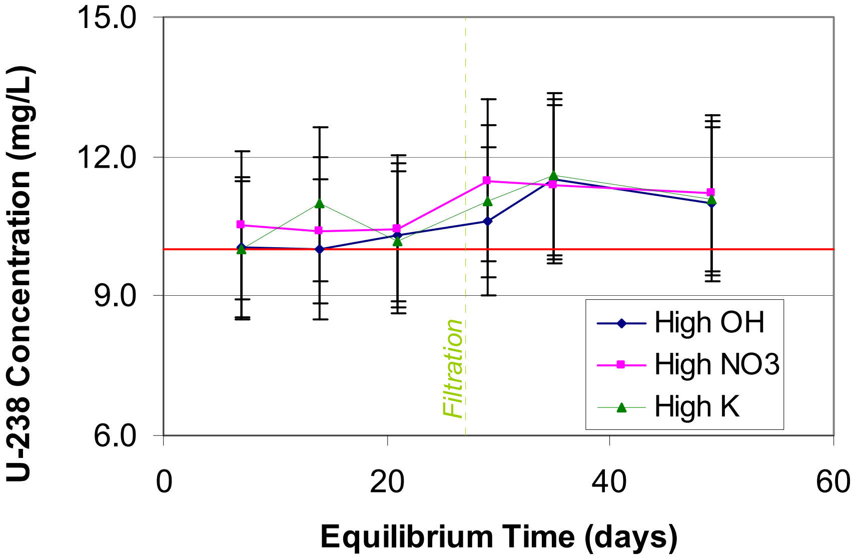


Figure 3. Neptunium in Solution in Each Simulant During Equilibrium

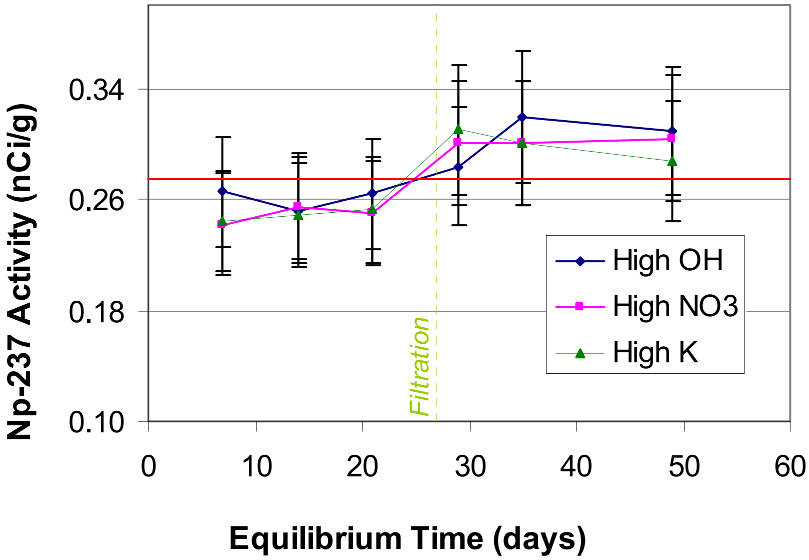
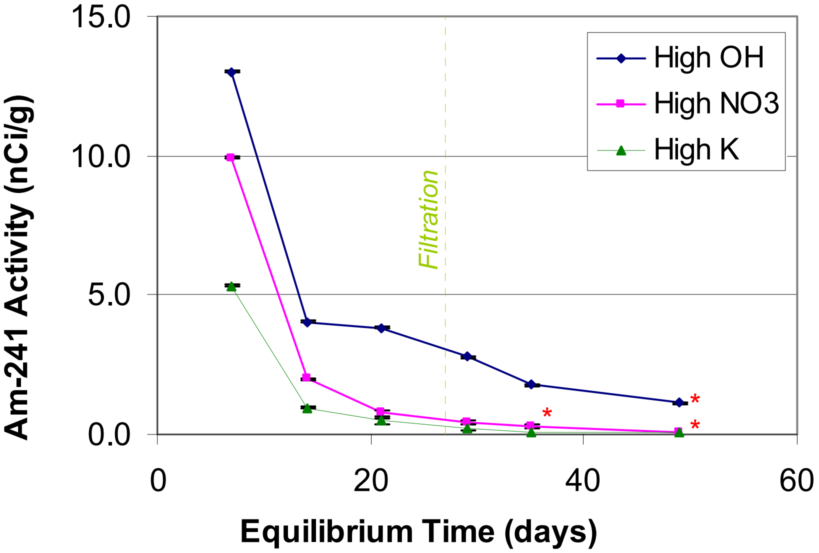
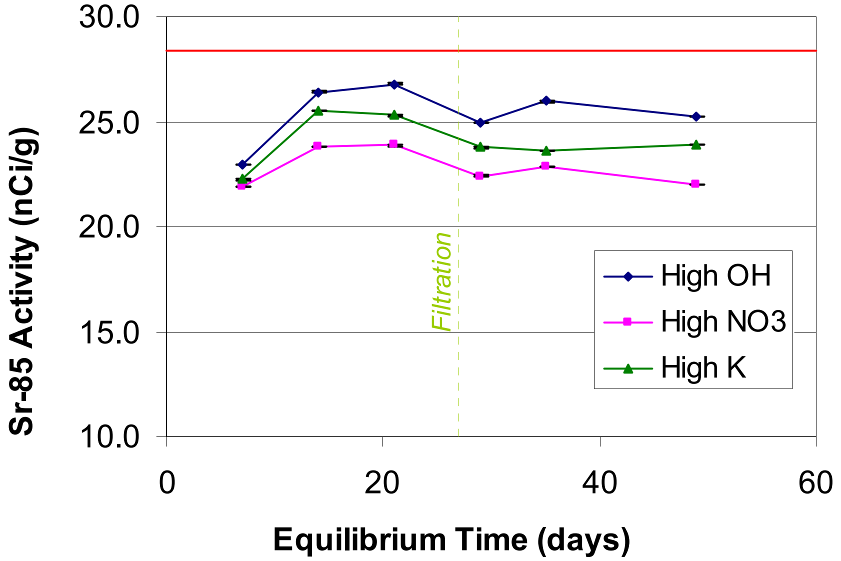


Figure 4. Americium in Solution in Each of the Simulants During Equilibrium



The red stars in the americium graph indicate those data points that fell below the method detection limit (MDL). Personnel added sufficient americium to reach 52.4 nCi/g if complete dissolution occurred.

Figure 5. Strontium-85 in Solution in Each Simulant During Equilibrium



Given the relatively short half life of ^{85}Sr (65.2 days), Figure 5 contains strontium values corrected for decay; all data is increased against the first weeks equilibrium sample (time = 0).

4.2 Radionuclide Testing

After the equilibration period, researchers started the AMP testing. Researchers pulled four samples from each solution and prepared them for the AMP tests by acidifying to an acid concentration of 0.1 M (although acid concentrations of 0.01 to 1 M are acceptable). During acidification, personnel diluted two of the samples 2:1 during acidification, with the other two samples diluted 10:1. We subjected one of each of the diluted samples to the AMP treatment, and used the remaining sample as a control. The ADS personnel analyzed each of the experimental and control samples in triplicate.

Tables 3-7, list the averages of the triplicate analyses. In cases where the analytical uncertainties of the triplicate data points were different, the uncertainty (1σ) of the average of the triplicates was calculated using the following formula (equation 1).

$$\text{composite uncertainty} = \sqrt{\frac{\Sigma\,(\text{sample uncertainty})^2}{9}} \qquad \text{Equation 1}$$

For analyses performed by ICPMS, the uncertainty (1σ) of the average was calculated by the simple standard deviation (ICPMS has a fixed analytical uncertainty of 15% for each data point).

In cases in the americium data where an analytical result fell below the method detection limit, we did not use the individual value in determining the average. Values in Table 10 that contained such cases are footnoted for identification.

For each of the solutions and dilution, compare the experimental value *vs.* the control value. If the AMP shows no effect or affinity for an element, the two values should be identical. If the experimental result is less than the control value, this difference may indicate that AMP has an affinity for the element.

4.2.1 Plutonium Data

Table 4 lists the plutonium results of the AMP strikes and controls.

Table 4. Plutonium-239/240 Data for AMP Strikes

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (nCi/g)	Uncertainty (nCi/g)	Experimental (nCi/g)	Uncertainty (nCi/g)
High OH Experiment	11.4	0.377	13.3	0.478
High OH Control	12.9	0.408	12.1	0.391
High NO3 Experiment	12.3	0.507	11.7	0.398
High NO3 Control	12.9	0.492	12.5	0.438
High K Experiment	11.4	0.392	11.7	0.398
High K Control	11.2	0.411	10.9	0.460

A rigorous approach to examining the data is to perform an analysis of variance (F-test). Using this method we were able to explicitly determine if the dilution, salt solution, or the differences in the experiment/control ("type") were statistically relevant. Although a detailed explanation of the F-test is beyond the scope of this document, we summarize the results (Table 5) as well as present some of the statistical output in Appendix I. The statistical analyses were conducted using JMP® Version 5.0 from SAS Institute, Inc.

Table 5. Effect Test Results for Plutonium Data Analysis

Variable	Prob > F
Dilution	**0.9901**
Solution	**0.0524**
Type	**0.7318**

If the probability of the F-value (Prob > F column of Table 5) is less than 0.05, then the corresponding variable is statistically significant (at the 0.05 significance level[a]) in explaining the amount of plutonium in solution. The results of the F-test for plutonium indicate that the plutonium levels in solution in our tests were not dependent on the dilution, the solution composition, or whether the sample was an experimental or control (the "type" variable) sample at the 5% significance level (i.e., with at least 95% confidence). While the effects due to solution type are just above this significant level, remember that it is expected that differences in solution composition can cause small differences in species solubility.

The most important result is that there is no significant differences between the experimental and control data. From these data we concluded that AMP does not possess any affinity for plutonium under our experimental conditions.

4.2.2 Uranium Data

Table 6 lists the uranium results of the AMP experimental data and controls.

Table 6. Uranium Data for AMP Strikes

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (nCi/g)	Uncertainty (nCi/g)	Experimental (nCi/g)	Uncertainty (nCi/g)
High OH Experiment	10.2	0.524	10.9	0.436
High OH Control	8.03	1.71	10.9	0.208
High NO3 Experiment	11.9	0.370	11.8	0.170
High NO3 Control	11.9	0.186	11.9	0.221
High K Experiment	10.8	0.269	9.62	0.746
High K Control	10.2	0.326	9.56	0.815

The High Hydroxide 2× dilution data showed an offset between the experimental and control data. Examination of the uranium 2× dilution control raw data shows that one of

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[a] The 5% significance level is a 95% confidence that the variable is statistically significant.

the triplicate results is notably low (9.62, 8.24, 6.22 mg/L). However, a Q-test does not allow us to remove this data point from consideration. Accordingly, we consider this data point to be due to analytical or experimental variance which in turn biases the control data low. We do not consider this offset between the experimental and control data to be due to AMP.

We analyzed the uranium data in the same fashion as with the plutonium data (F-test). We summarize the results (Table 7) as well as present some of the statistical output in Appendix I.

Table 7. Effect Test Results for Uranium Data Analysis

Variable	Prob > F
Dilution	**0.3833**
Solution	**<.0001**
Type	**0.1897**

The results of the F-test for uranium indicate that the uranium levels in solution in our tests were not dependant on the dilution, or whether the sample was an experimental or control sample. The difference in solution composition produced a significant variance, indicating that the uranium levels in solution did depend on the solution composition.

The most important result is that there is no significant differences between the experimental and control data. From this data we concluded that AMP does not possess any affinity for uranium under our experimental conditions.

4.2.3 Neptunium Data

Table 8 lists the neptunium results of the AMP control and experimental data.

Table 8. Neptunium Data for AMP Strikes

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (nCi/g)	Uncertainty (nCi/g)	Experimental (nCi/g)	Uncertainty (nCi/g)
High OH Experiment	0.252	0.0103	0.276	0.0223
High OH Control	0.216	0.0318	0.289	0.00803
High NO3 Experiment	0.288	0.00205	0.286	0.0172
High NO3 Control	0.300	0.00921	0.295	0.00812
High K Experiment	0.294	0.00793	0.253	0.00952
High K Control	0.265	0.00940	0.255	0.0248

As with the uranium data, the High Hydroxide 2× dilution data showed an offset between the experimental and control data. Examination of the neptunium 2× dilution control raw data shows that one of the triplicate results is notably low (0.241, 0.226, 0.180 nCi/g); the same data point for the uranium data – obtained from the same ICP-MS analysis – is also low. However, a Q-test does not allow us to remove this data point from consideration. Accordingly, we consider this data point to be due to analytical or experimental variance which in turn biases the control data low. The fact that both the uranium and neptunium data points have the same data point that is low reinforces the notion that the offset between the experimental and control data is not due to AMP.

We analyzed the neptunium data in the same fashion as with the plutonium data (F-test). We summarize the results (Table 9) as well as present some of the statistical output in Appendix I.

Table 9. Effect Test Results for Neptunium Data Analysis

Variable	Prob > F
Dilution	**0.4290**
Solution	**0.0044**
Type	**0.5702**

The results of the F-test for neptunium indicate that the neptunium levels in solution in our tests were not dependant on the dilution, or whether the sample was an experimental or control sample. The difference in solution composition produced a significant variance, indicating that the neptunium concentration in solution did depend on the solution composition.

The most relevant result is that there is no significant differences between the experimental and control data. From this data we concluded that AMP does not possess any affinity for neptunium under our experimental conditions.

4.2.4 Americium Data

Of all the radioisotope data, the americium information proves the hardest to interpret. Due to the lack of solubility displayed during the equilibrium period, some of the analyses of americium gave less than detectable concentrations. This issue continued during the AMP strikes. Table 10 shows the americium data. Of the 12 sets of data, three sets consist entirely of method detection limits (MDL), and thus provide no insight on the influence of the AMP treatment. The High Potassium 2× control data contained a single analysis below the MDL and we exclude that value from the calculations.

Table 10. Americium Data for AMP Strikes

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (nCi/g)	Uncertainty (nCi/g)	Experimental (nCi/g)	Uncertainty (nCi/g)
High OH Experiment	1.38	0.0229	1.25	0.0321
High OH Control	1.43	0.0238	1.48	0.0357
High NO3 Experiment	MDL [a]	MDL [a]	MDL [a]	MDL [a]
High NO3 Control	0.0223	0.00661	MDL [a]	MDL [a]
High K Experiment	0.0435	0.00552	0.0680	0.00722
High K Control	0.0368 [a]	0.00652	0.0736	0.00780

MDL = Method Detection Limit (varies for each data point)

[a] Indicates these values contain MDL data that were not used when calculating the average.

From all the data we conclude that AMP may possess a small affinity for americium under our experimental conditions.

We analyzed the americium data in the same fashion as with the plutonium data (F-test). We summarize the results (Table 11) as well as present some of the statistical output in Appendix I.

Table 11. Effect Test Results for Americium Data Analysis

Variable	Prob > F
Dilution	**0.6642**
Solution	**<.0001**
Type	**0.0156**

The results of the F-test for americium indicate that the americium levels in solution in our tests were not dependant on the dilution. The difference in solution composition produced a significant variance, indicating that the americium concentration in solution strongly depended on the solution composition. The difference between the experimental and control samples also was significant, indicating that AMP does have an affinity for americium.

As part of the F-test analysis, we examined the effect of one variable at a time. In the case of the experimental/control variable, we estimated the degree of difference in the behavior of americium in the experimental vs. control experiments. By comparing the least square mean results (Table 12), we estimated the DF caused by the presence of AMP in the experiment (Control÷Experimental) to be 1.11.

Table 12. Experimental vs. Control Data

Variable	Least Sq Mean
Control	**0.760**
Experimental	**0.686**

4.2.5 Strontium Data

The strontium data showed excellent agreement between the experimental results and control data (Table 13).

Table 13. Strontium-85 Data for AMP Strikes

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (nCi/g)	Uncertainty (nCi/g)	Experimental (nCi/g)	Uncertainty (nCi/g)
High OH Experiment	27.8	0.306	28.7	0.348
High OH Control	27.7	0.306	29.0	0.352
High NO3 Experiment	24.8	0.276	25.7	0.315
High NO3 Control	24.9	0.277	25.8	0.315
High K Experiment	26.5	0.295	27.5	0.308
High K Control	27.1	0.302	27.7	0.311

We analyzed the strontium data in the same fashion as with the plutonium data (F-test). We summarize the results (Table 14) as well as present some of the statistical output in Appendix I.

Table 14. Effect Test Results for Strontium Data Analysis

Variable	Prob > F
Dilution	**<.0001**
Solution	**<.0001**
Type	**0.0607**

The results of the F-test for strontium indicate that the strontium levels in solution in our tests were dependant on the dilution, as well as the difference in solution composition.

Finally, whether the sample was an experimental or control sample had no effect on the strontium in solution.

The primary result is that there is no significant differences between the experimental and control data. From this data we concluded that AMP does not possess any affinity for strontium under our experimental conditions.

4.3 RCRA and Non-radioactive Element Testing

In addition to determining the effect of AMP on soluble radionuclides, SRTC tested the effect of AMP on other elements, particularly the RCRA elements (i.e., Ag, As, Ba, Cd, Cr, Hg, Pb, Se). The experiments followed the same general procedure as used in the radionuclide testing, although the solution volumes and amount of AMP used differed. The researchers used a High Potassium salt solution that did not contain radionuclides. Table 15 lists the data for the experimental and control samples.

Table 15. ICPES, AA, and CVHg-AA Data

Element	Analytical Method	Control Concentration (mg/L)	Experimental Concentration (mg/L)	DF
Ag	ICPES	26.0	11.2	2.32
Al	ICPES	8020	8040	1.00
As	AA	119	133	0.895
Ba	ICPES	0.600	0.580	1.03
Ca	ICPES	1.76	2.24	0.786
Cd	ICPES	3.40	4.00	0.850
Cr	ICPES	83.6	81.6	1.02
K	ICPES	3400	3280	1.04
Hg	CVHg	61.2	64	0.956
Mo	ICPES	10.8	11.1	0.973
Na	ICPES	124000	125000	0.992
P	ICPES	310	302	1.03
Pb	ICPES	84.6	83.8	1.01
S	ICPES	4800	4800	1.00
Se	AA	30.8	59	0.522
Si	ICPES	59.2	56.6	1.05

Analytical uncertainty is 10%. Bold elements are RCRA.

Of all 16 elements that we could qualify from the data, AMP showed an affinity for only silver (i.e., significantly more silver reported in the control than in the experiment). This agrees with data from the literature (see section 4.5, below). While we expect AMP to have an affinity for potassium, the experimental and control values agreed within analytical uncertainty. The selenium result is noticeably higher in the experimental data (than in the control), which indicates that AMP treatment may give a false positive high result.

While elemental sulfur and phosphorus are listed in the table, the sulfur in the study came from sulfate anion, and the phosphorus came from phosphate anion. Therefore, while we can make no conclusions about AMP affinity for atomic sulfur or phosphorus, we can say that AMP does not have any affinity for sulfate or phosphate anions.

From ICP-MS fission product analysis, a limited amount of data can be retrieved from the analysis. Non-radioactive strontium, cesium, and rubidium data can be assessed. The strontium and cesium data is discussed in section 4.4, below. The rubidium data (Table 16) clearly indicates that AMP has an affinity for Rb. This behavior is expected, as Rb is the element above Cs in the periodic table; the affinity for AMP for alkali metals is well known.

Table 16. Rubidium Concentration in the Salt Solutions (2× Dilution)

Simulant	Experimental (mg/L)	Uncertainty (mg/L)	Control (mg/L)	Uncertainty (mg/L)	DF
High Hydroxide	4.55E-02 [a]	6.83E-03	9.17E-02	4.82E-03	2.02
High Nitrate	2.03E-02	3.88E-03	6.44E-02	6.26E-03	3.17
High Potassium	4.24E-02	9.88E-04	1.25E-01 [b]	1.14E-02	2.95

[a] While most values in this table were derived from triplicate samples, this data point was from a single result, and the uncertainty is just the 15% analytical uncertainty.

[b] This value and its uncertainty were derived from two results.

4.4 Strontium and Cesium Testing

The last set of tests involved examination of varying concentrations of strontium in the three salt solutions. The test design examines the effect of large dilutions on the non-radioactive strontium in solution, and the ability of ICP-MS to reliably analyze those solutions. When preparing the three solutions, personnel did not explicitly add strontium since "tramp" strontium is present in the reagent chemicals. ICP-MS analysis of the non-radioactive strontium in each of the three solutions during the equilibration period showed less cold strontium than desired (Figure 6). At the end of the radionuclide testing, the researchers added strontium to the remainder of the High Hydroxide simulant sufficient to add another ~900 μg/L of strontium (in the form on strontium nitrate). This solution equilibrated for two additional weeks while stirring. At the end of two weeks, the researchers performed a series of AMP strikes on aliquots of the salt solution. Aside from the control sample (no AMP), researchers performed an AMP strike on solutions after dilution 2:1, 4:1, 10:1, and 100:1 with nitric acid (the samples were allowed to acidify for at least two hours before use). Personnel collected a final sample one week later to confirm that the strontium concentration remained stable (i.e., reach equilibrium by the time of the AMP test) and to provide an additional measure of variability for the AMP treatment method. Researchers used the same general procedure detailed in section 3.2 except adding ~ 40mg of AMP instead of 60 mg. Table 17 lists the calculated and measured values. The measured value was derived by applying the appropriate known dilution to the control sample measured value (the control sample itself was diluted 2:1).

Figure 6. Non-radioactive Strontium present in Original Solutions

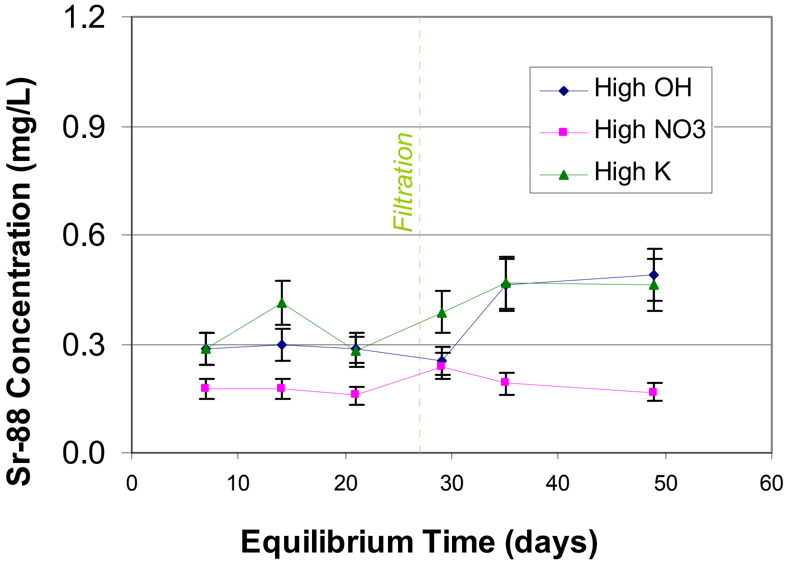


Table 17. Non-radioactive Strontium (^{88}Sr) Results (High OH Simulant)

Sample Dilution	Measured Cold Strontium (mg/L)	Calculated Cold Strontium (mg/L)
Control	0.366	NA
2:1	0.376	0.366
2:1*	0.367	0.366
4:1	0.186	0.183
10:1	0.0713	0.0732
100:1	0.00846	0.00732

"*" Repeat analysis one week after initial samples.
Analytical uncertainty is 15%.

The data confirms that the presence of AMP has no effect on the strontium concentration in solution through a range of concentrations. Also note that we are losing instrument linear response somewhere below 0.0713 mg/L.

SRTC also examined the effect of AMP on the cesium. While the affinity of AMP for cesium is well known, the researchers decided to quantify the effect under our reaction conditions. The cesium data comes from the radionuclide testing samples. During the equilibrium period, the non-radioactive cesium concentration (Figure 7) remained relatively constant. The differences in the cesium concentrations in each of three solutions are most likely attributable to varying amounts of tramp cesium introduced from the three different chemical recipes. Table 18 shows the data for the experimental and control samples for the AMP strike. In each case the AMP exhibited a strong affinity for cesium.

Figure 7. Non-radioactive Cesium Concentrations in Solution

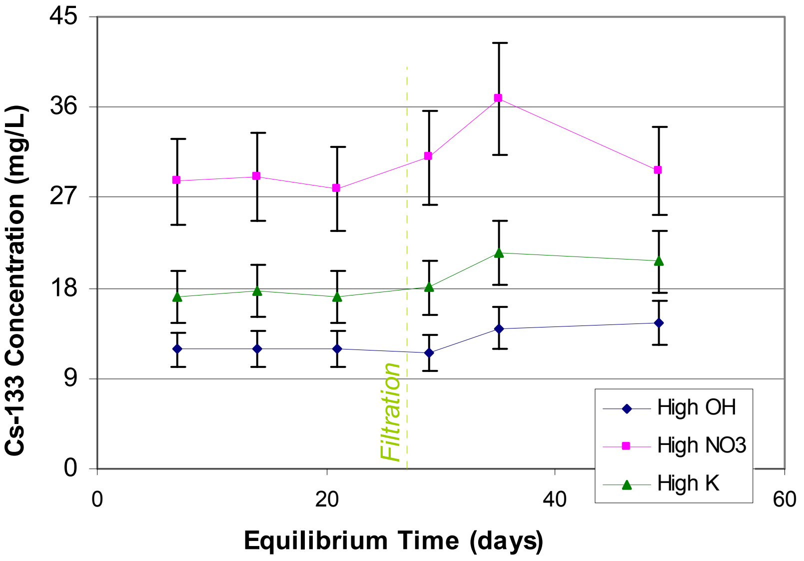


Table 18. Non-radioactive Cesium (^{133}Cs) Concentrations in Solution

Simulant Solution	2× Dilution		10× Dilution	
	Experimental (mg/L)	Uncertainty (mg/L)	Experimental (mg/L)	Uncertainty (mg/L)
High OH Experiment	0.358	0.0310	0.096	0.00830
High OH Control	10.1	0.875	12.4	1.07
High NO3 Experiment	0.519	0.0449	0.742	0.0643
High NO3 Control	35.6	3.08	35.8	3.10
High K Experiment	0.579	0.0501	0.146	0.0130
High K Control	27.0	2.34	24.9	2.16

The decontamination factor (DF) varied between the tests and even varied between the dilutions. Table 19 lists the DF values for each data set.

Table 19. Cesium Decontamination Factors from Each Solution

Data Set	DF Factor
High Hydroxide, 2× Dilution	28.2
High Hydroxide, 10× Dilution	129
High Nitrate, 2× Dilution	65.6
High Nitrate, 10× Dilution	48.2
High Potassium, 2× Dilution	46.6
High Potassium, 10× Dilution	171

Under a variety of conditions, the minimal DF equals ~30, which is adequate to reduce exposure for sample removal from the cells. The range of DF values is roughly comparable to the cesium DF quoted by Marsh (i.e., K_d of 2133 @ 30 minutes = DF of 84.5).

4.5 Literature Review of AMP Data

The literature contains numerous studies of the affinity of AMP for various elements.[5,8,11,12,13,14,15,16,17] M. J. Barnes and M. E. Stallings of SRTC are currently performing additional tests with Savannah River Site waste to increase the understanding of elemental affinities for AMP.

One of the most comprehensive single documents on element sorbtion is by Marsh *et al.*[8] Marsh tested a large number of sorbents (including AMP-PAN) with acidified simulant (Hanford SY-102) supernate solutions. Marsh tested the ability of AMP-PAN to sorb any of 14 different elements over three contact time periods (0.5, 2 and 6 hours). For comparison, our work used a contact time of 30 seconds. Table 20 provides the distribution coefficients (K_d) and decontamination factors (DF) from these tests.

In addition to cesium, it appears that AMP-PAN has measurable affinity for cerium, yttrium, and americium at contact times of 30 minutes or greater. SRTC work reported in this document indicates that AMP possibly has a small affinity for americium over short (30 second) contact times. These data sets clearly illustrate that longer contact time with AMP increases uptake for those elements that the material does sorb.

Table 20. Marsh *et al.,*[8] AMP-PAN Results

Element	30 minute K_d (ml/g)	30 minute DF	2 hour K_d (ml/g)	2 hour DF	6 hour K_d (ml/g)	6 hour DF
Ce	12	1.5	22	1.9	28	2.1
Cs	2133	84.55	4636	182.6	> 8000	314.4
Sr	< 0.1	1.0	< 0.1	1.0	< 0.1	1.0
Tc	0.8	1.0	1.2	1.0	1.4	1.1
Y	15	1.6	27	2.1	34	2.3
Cr	0.5	1.0	1.3	1.1	1.3	1.1
Co	1.5	1.1	1.8	1.1	1.7	1.1
Fe	0.5	1.0	1.1	1.0	1	1
Mn	< 0.1	1.0	0.1	1.0	< 0.1	1.0
Zn	0.5	1.0	0.4	1.0	0.5	1.0
Zr	0.4	1.0	1.1	1.0	0.9	1.0
U	1.6	1.1	2.1	1.1	2.5	1.1
Pu	0.5	1.0	0.8	1.0	1.2	1.1
Am	24	2.0	50	3.0	86	4.4

The 30 minute contact time data point from the Marsh work for americium translates to a DF of 1.94. Our 30 second contact time work gives an (composite) americium DF of 1.11. While this may indicate a minor AMP affinity for americium, the differences in the two DF values may be attributed to the contact times for the two sets of work.

A second report by Todd, *et al.*[5] discussed the affinity of AMP-PAN for mercury, americium and plutonium. These tests contacted an acidic salt solution simulant containing ^{203}Hg, ^{238}Pu, and ^{241}Am for 24 hours with AMP-PAN. Table 21 lists Todd's distribution constants and decontamination factors, and comparative DF values from this work.

Table 21. Todd *et al.*,[5] AMP-PAN Results

Element	24 hour K_d	24 hour DF	30 second DF, this work [a]
Hg	1.41	1.01	0.956
Pu	5.12	1.05	1.01
Am	6.03	1.06	1.09

[a] The Pu and Am DF values were derived by a comparison of the total of the control results vs. the total of the experimental results. Our work also used a different volume/mass ratio than Todd, which causes some differences between the DF values.

The Todd work shows a minimal affinity of AMP-PAN for mercury, plutonium or americium.

An earlier study by R. Smit, *et al.*[15] studied the effect of AMP on sodium, potassium, rubidium, thallium, and silver. Smit used acidified (pH 2) solutions of ammonium nitrate with the appropriate radioisotope contacted with AMP for a period of ~8 hours ("overnight"). Table 22 lists the distribution coefficients and decontamination factors from Smit's work, as well as DF values from this work.

Table 22. Smit *et al.*,[15] Using AMP Results

Element	~8 hour K_d	~8 hour DF	30 second DF, this work
Rb	192	4.20	3.29 [b]
K	4	1.07	1.04
Na	0	1.00	0.992
Tl(I)	4295	72.58	NA
Ag	25.9	1.43	2.32

NA = not analyzed in this work

[b] The Rb DF was a composite value from data in Table 10.

AMP appears to have a strong affinity for thallium, a strong affinity for rubidium, and a moderate affinity for silver. Although not tested in his work, Smit declared that AMP should also have an affinity for mercury (I).

Work by W. Faubel *et al.*[17] declared that antimony, ruthenium/rhodium (Faulbel used a mixed ^{106}Ru/Rh tracer) and europium were not sorbed from a 1.9 M nitric acid solution after a 10 minute contact with AMP. The report did not provide any distribution coefficients.

Finally, D. DiPrete of SRTC , although not published, studied the effect AMP had on cobalt and europium. The analytical section at SRTC examined the spike recoveries of radiocobalt and –europium when samples were treated with AMP (Table 23).

Table 23. Recoveries of Radiocobalt and –Europium

Species	Before AMP (dpm/mL)	After AMP (dpm/mL)	% Difference	Analytical Uncertainty %
Co	1.82E+03	1.74E+03	4.4	5.31
Eu	3.64E+04	3.42E+04	6.0	0.98

In the case of cobalt, the difference between the before (before treating the solution with AMP) and after (after treating the solution with AMP) was 4.4%, which was less than the analytical uncertainty of the measurement. This means that AMP has no discernable effect on cobalt. With europium, the difference before and after was 6.0%, which was larger than the analytical uncertainty. From this we conclude that AMP does have an effect on europium, although not a large one.

In total, literature references declare that AMP has a minor affinity for americium, cerium, europium and yttrium after extended contact (i.e., longer than 30 seconds). AMP also has an affinity for silver, thallium, and rubidium. Mercury (II); the most common form of mercury in strongly acidic solution, does not readily sorb on AMP, although mercury (I) might. The effect of AMP on yttrium and europium (both group III elements) suggests that AMP likely has a small affinity for the rest of the rare earths or lanthanides.

5.0 CONCLUSIONS

- Researchers developed a sample treatment protocol, using ammonium molybdophosphate (AMP) to remove sufficient cesium to allow handling of the samples with minimal dilution. While the protocol conditions can vary somewhat, SRTC specifically used the following treatment steps.
    - pH adjust the sample to the range of 0.01 to 1.0 M acidity
    - Mix 30 mL of acidified sample with 40-60 mg of AMP
    - Cap and shake the mixture for 30-60 seconds
    - Filter AMP from the liquid using 0.45 μm PTFE syringe filters
    - Send filtrate directly forward for analysis

- We consider these steps subject to some variation as circumstances determine. For example, using 0.45 µm cellulose nitrate cup filters in the place of the PTFE syringe filters is perfectly acceptable.

- Within the protocol conditions, AMP exhibited no appreciable affinity for plutonium, neptunium, uranium and strontium.

- AMP showed a possible minor affinity for americium; however, the data is not as clear due to continued americium concentration changes during the length of the experiment and proximity of the MDL.

- Of all eight RCRA elements (Ag, As, Ba, Cd, Cr, Hg, Pb, Se), AMP exhibited affinity for only silver under our experimental conditions.

- SRTC determined that under our experimental conditions, AMP has an affinity for rubidium.

- While our data did not indicate that AMP has an affinity for potassium at our experimental conditions, the literature contains examples where AMP showed some affinity for potassium.

- The use of AMP can clearly provide a benefit for those analytical procedures that do not require dilutions. Radiochemistry is the prime beneficiary. The effect on ICPMS, ICPES, or AA, which require dilutions to reduce salt content, is harder to judge at this time. The results of the real waste tests will be required before we can make a judgment in those cases.

- The AMP treatment protocol allowed accurate measurement of non-radioactive strontium at concentrations above 0.07 mg/L using inductively coupled plasma mass spectroscopy. The analytical method yielded a +15% bias when the concentration fell to 0.007 mg/l.

- From similar tests reported in the literature, it appears that cerium, yttrium, europium, thallium, americium, silver, and possibly mercury (I) have or may have some affinity for AMP. However, differences in the testing methods do not make this an absolute conclusion. As yttrium and europium are either rare earth or lanthanide elements, AMP will likely show an affinity for other rare earths or lanthanides.

- We recommend the following actions.
  - Examine the AMP results from actual waste testing done in the EPC vendor support work when they become available. At that time, this work (simulant) and real waste results can be compared to insure there are no offsets. Furthermore, the actual waste testing will increase the number of elements for which we can explicitly determine AMP affinity.
  - Perform a small set of further simulant reactions to confirm whether or not AMP has an affinity for potassium or selenium.
  - Study ways to improve and simplify the experimental procedure. The protocol would be easier to perform if the researcher did not have to precisely determine the amount of acid required.

6.0 ACKNOWLEDGEMENTS

We would like to acknowledge and thank Mona Blume for the excellent technical work, ADS for the quality and rate of sample results, and Tommy Edwards for the statistical analysis of the data.

Appendix I. Statistical Results from Data Analysis of Variance

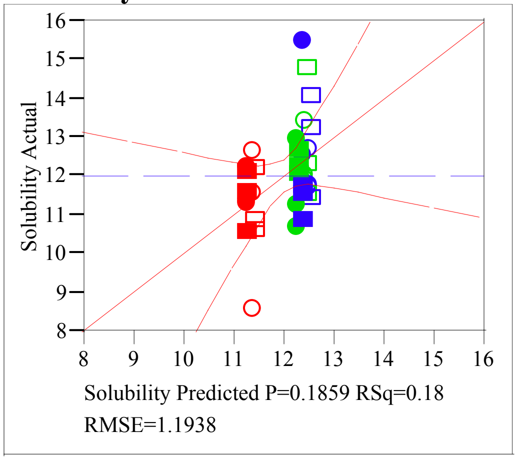
Using the JMP software package, all of the data points (triplicates, not the averages) were entered into an analysis of variance study. The effect of three variables; dilution, solution type and difference between experimental and control ("type") were studied to determine which, if any, were significant.

Analyte=Plutonium

Response Solubility

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.175845
RSquare Adj	0.069502
Root Mean Square Error	1.193774
Mean of Response	12.02502
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	9.425993	2.35650	1.6536
Error	31	44.178002	1.42510	Prob > F
C. Total	35	53.603995		0.1859

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	7	9.699662	1.38567	0.9645
Pure Error	24	34.478340	1.43660	Prob > F
Total Error	31	44.178002		0.4784
				Max RSq
				0.3568

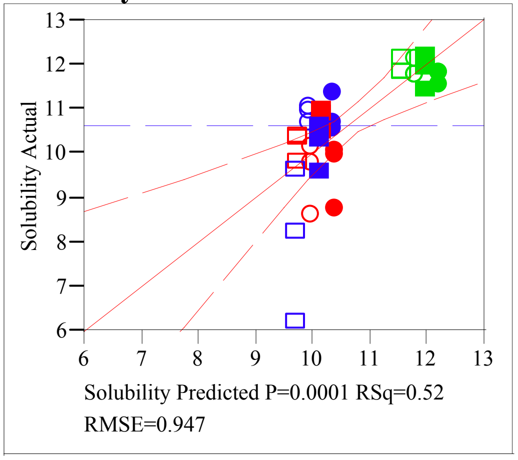
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	12.025019	0.198962	60.44	<.0001
Dilution[10x]	0.002485	0.198962	0.01	0.9901
Solution[K]	-0.715183	0.281375	-2.54	0.0162
Solution[NO3]	0.312614	0.281375	1.11	0.2751
Type[Control]	0.0688118	0.198962	0.35	0.7318

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dilution	1	1	0.0002223	0.0002	0.9901
Solution	2	2	9.2553079	3.2473	0.0524
Type	1	1	0.1704623	0.1196	0.7318

Analyte=Uranium
Response Solubility
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.515478
RSquare Adj	0.452959
Root Mean Square Error	0.947007
Mean of Response	10.64236
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	29.577693	7.39442	8.2451
Error	31	27.801508	0.89682	Prob > F
C. Total	35	57.379200		0.0001

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	7	17.637924	2.51970	5.9500
Pure Error	24	10.163583	0.42348	Prob > F
Total Error	31	27.801508		0.0004
				Max RSq
				0.8229

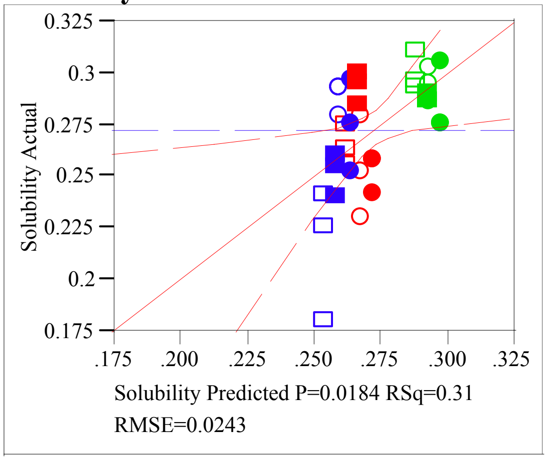
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	10.642361	0.157835	67.43	<.0001
Dilution[10x]	0.1395833	0.157835	0.88	0.3833
Solution[K]	-0.598194	0.223212	-2.68	0.0117
Solution[NO3]	1.2305556	0.223212	5.51	<.0001
Type[Control]	-0.211639	0.157835	-1.34	0.1897

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dilution	1	1	0.701406	0.7821	0.3833
Solution	2	2	27.263810	15.2002	<.0001
Type	1	1	1.612477	1.7980	0.1897

Analyte=Neptunium
Response Solubility
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.310191
RSquare Adj	0.221183
Root Mean Square Error	0.02426
Mean of Response	0.27238
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.00820434	0.002051	3.4850
Error	31	0.01824496	0.000589	Prob > F
C. Total	35	0.02644930		0.0184

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	7	0.01227075	0.001753	7.0421
Pure Error	24	0.00597421	0.000249	Prob > F
Total Error	31	0.01824496		0.0001
				Max RSq
				0.7741

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2723799	0.004043	67.37	<.0001
Dilution[10x]	0.0032406	0.004043	0.80	0.4290
Solution[K]	-0.005759	0.005718	-1.01	0.3216
Solution[NO3]	0.020001	0.005718	3.50	0.0014
Type[Control]	-0.002321	0.004043	-0.57	0.5702

Effect Tests

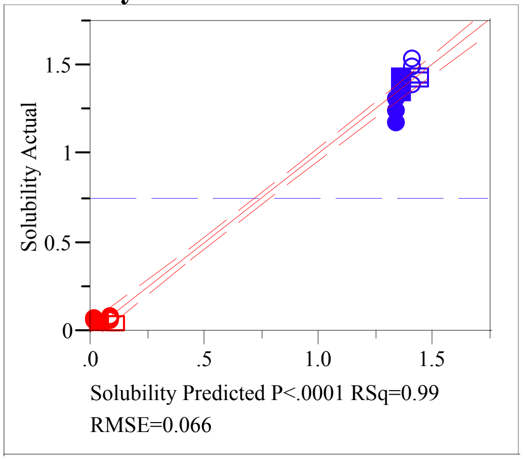
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dilution	1	1	0.00037805	0.6423	0.4290
Solution	2	2	0.00763243	6.4841	0.0044
Type	1	1	0.00019385	0.3294	0.5702

Analyte=Americium

Response Solubility

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.991898
RSquare Adj	0.990619
Root Mean Square Error	0.066041
Mean of Response	0.749774
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	10.144912	3.38164	775.3500
Error	19	0.082867	0.00436	Prob > F
C. Total	22	10.227779		<.0001

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.05777883	0.014445	8.6363
Pure Error	15	0.02508840	0.001673	Prob > F
Total Error	19	0.08286723		0.0008
				Max RSq
				0.9975

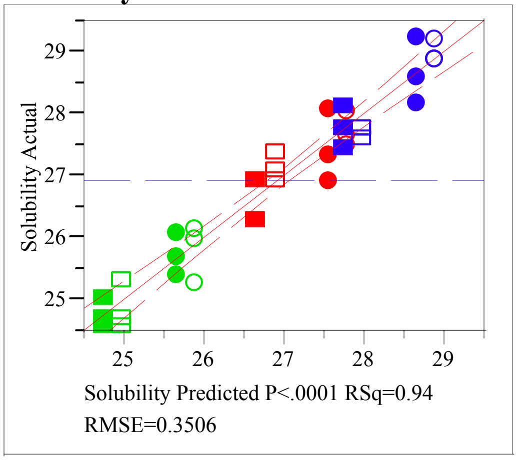
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.7228613	0.013813	52.33	<.0001
Dilution[10x]	-0.006092	0.013813	-0.44	0.6642
Solution[K]	-0.6618	0.013813	-47.91	<.0001
Type[Control]	0.0367065	0.013813	2.66	0.0156

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dilution	1	1	0.000848	0.1945	0.6642
Solution	1	1	10.010959	2295.337	<.0001
Type	1	1	0.030797	7.0612	0.0156

Analyte=Strontium
Response Solubility
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.943851
RSquare Adj	0.936606
Root Mean Square Error	0.350598
Mean of Response	26.94065
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	64.053216	16.0133	130.2753
Error	31	3.810487	0.1229	Prob > F
C. Total	35	67.863703		<.0001

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	7	0.5952997	0.085043	0.6348
Pure Error	24	3.2151870	0.133966	Prob > F
Total Error	31	3.8104868		0.7227
				Max RSq
				0.9526

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	26.940652	0.058433	461.05	<.0001
Dilution[10x]	0.4733531	0.058433	8.10	<.0001
Solution[K]	0.2700091	0.082637	3.27	0.0027
Solution[NO3]	-1.637903	0.082637	-19.82	<.0001
Type[Control]	0.1137448	0.058433	1.95	0.0607

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Dilution	1	1	8.066273	65.6227	<.0001
Solution	2	2	55.521179	225.8447	<.0001
Type	1	1	0.465763	3.7892	0.0607

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