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# Permanganate Reduction of Savannah River Site Actual Waste Samples for Strontium and Actinides Removal

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## 1.0 Summary

The authors investigated the performance of permanganate treatment for the removal of strontium and actinides from actual high-level waste. Researchers also conducted comparison tests with monosodium titanate (MST) as a means of evaluating the permanganate performance. Tests utilized a Tank 37H/44F composite waste solution. Personnel significantly increased the concentration of alpha emitting actinides in the waste by the addition of acidic americium/curium solution (F-Canyon Tank 17.1 solution), which contained a significant quantity of plutonium, and neptunium-237 stock solution. Tests examined three permanganate treatment options.

- Nominal Permanganate: sequential addition of 0.01 M non-radioactive Sr, 0.01 M permanganate, and 0.045 M formate
- Low Permanganate: sequential addition of 0.01 M non-radioactive Sr, 0.002 M permanganate, and 0.009 M formate to yield a lower solids concentration
- Permanganate without Strontium: sequential addition of 0.01 M permanganate and 0.045 M formate only

Results indicated the following.

- All three permanganate treatment options reduced the $^{90}Sr$ concentration below the Saltstone Waste Acceptance Criteria (WAC). After 24 hours of reaction, 99% of the initial level of 154 nCi/g of strontium was removed. The use of non-radioactive strontium to provide isotopic dilution provided no significant advantage in kinetics or ultimate capacity for strontium removal in the treatment of these wastes.
- Comparison monosodium titanate tests also reduced the $^{90}Sr$ concentration below the Saltstone WAC. After 24 hours of reaction, 90% of the initial level of 154 nCi/g of strontium was removed.
- Under the conditions tested (i.e., high Pu concentration), all three permanganate treatment options proved unsuccessful in reducing the Pu concentration below the Saltstone WAC. After 24 hours of reaction, only 23% of the initial level of 303 nCi/g of Pu was removed. The Nominal Permanganate option provided the best level of decontamination within 24 hours of treatment (41% of the initial level of 303 nCi/g of Pu was removed). The addition of non-radioactive Sr for isotopic dilution of Sr appeared to enhance Pu removal (41% of the initial level of 303 nCi/g of Pu was removed with the addition of Sr and no, 0%, of the Pu was removed without Sr addition). The data set is not sufficient to fully evaluate this observation.
- The MST tests, like permanganate, also proved unsuccessful in decontaminating Pu to the required concentration (after 24 hours of reaction, 58% of the initial level of 303 nCi/g of Pu was removed). Under the conditions tested, MST proved slightly more effective than permanganate in reducing the Pu concentration.
- Neptunium decontamination appeared roughly equivalent between both treatment methods (after 24 hours of reaction, both the nominal permanganate treatment and MST treatment removed 47% of the initial level of 0.0877 nCi/g of Np) with both methods failing to achieve required removal efficiency.
- The poor decontamination performance observed in both permanganate and MST treatments likely resulted due to the increased concentrations of plutonium present in the waste. Increased levels of either MST or permanganate are required to achieve the required level of decontamination.

Based on these findings, we recommend the following.

- Conduct additional tests with increasing concentrations of MST and permanganate. The proposed tests would use the residual solution from a small subset of the current tests. Personnel would incrementally increase the quantity of MST or permanganate added while monitoring removal efficiency for periods of 24 hours. Also, the use of hydrogen peroxide as a reductant in place of formate should be evaluated.

- Investigate the observation that the soluble Np concentration increased in most permanganate tests during the first few hours of testing. A possible cause for this is oxidation of the insoluble Np by permanganate.

**Keywords: Salt Disposition, Strontium, Plutonium, Uranium, Neptunium, HLW**

## 2.0 Introduction

The current baseline flowsheet for the Salt Waste Processing Facility (SWPF) at the Savannah River Site uses monosodium titanate (MST) for the removal of radioactive Sr, Pu and Np. Hobbs and Walker[1] studied the adsorption of plutonium and uranium onto MST in alkaline solutions. These tests showed that MST would remove the targeted radionuclides from simulated alkaline waste. Continued testing indicates that plutonium removal kinetics and neptunium capacity of the MST material impacts the size of equipment and waste blending plans for the SWPF. Additionally, calculations suggest the baseline MST process may not achieve the desired decontamination in wastes containing elevated concentrations of Pu and Np.[2] Consequently, the Department of Energy (DOE) requested that Savannah River Technology Center (SRTC) personnel investigate the ability to remove

strontium and actinides from high-level waste by addition of sodium permanganate in the presence of a reductant (e.g., formate) and with isotopic dilution by addition of strontium. This approach follows similar studies performed for Hanford waste.[3] If successful, this process offers increased throughput resulting in decreased equipment size. The DOE also requested that SRTC conduct tests evaluating the capability of MST in actual high-level waste that contained elevated concentrations of Pu and Np as a means of comparison to the permanganate treatment.

Previously, research identified the adsorption kinetics of actinides and strontium onto MST as a technical risk. Hobbs' MST tests examined the extent and rate of adsorption of strontium, uranium, neptunium and plutonium as a function of temperature, monosodium titanate concentration, and the concentrations of sodium and adsorbing species (Sr, Pu, Np and U).[4] Analysis of the testing indicated the need to perform additional kinetic testing with radioactive Savannah River Site (SRS) tank waste and with simulants at lower ionic strength and MST concentrations. Subsequent radioactive waste tests utilized a composite material prepared from archive samples from over twenty SRS tanks. Results indicated that the extent and rate of strontium, plutonium, neptunium and uranium removal with MST in radioactive waste agree with that previously measured with simulants.[5,6] Additional tests with simulated waste solutions measured the extent and rate of strontium, plutonium, neptunium and uranium removal at 25 ° C in the presence of 0.2 and 0.4 g/L MST at 4.5 and 7.5 M sodium concentration. Later testing measured removal characteristics of the MST testing using a simulated salt solution with a sodium concentration of 5.6M. Results indicated lower sorbate removal with increased sodium ion concentration.[7] Tests described in this document address the capability of permanganate treatment to remove Sr, Pu, and Np from actual high-level waste containing elevated concentrations of Pu. Additionally, the tests investigate MST (using two unique batches) performance with the same waste for direct comparison to the permanganate performance.

## 3.0 Experimental

The investigation consisted of 10 tests addressing either permanganate ($MnO_4^-$) or MST performance in actual high-level waste. Table 1 indicates the variations in the tests. Six of the 10 tests used permanganate with three tests as duplicates of the others. Three of the 10 tests used MST. A single control test contained neither permanganate nor MST. Nine of the tests used the same actual high-level waste stock solution containing elevated concentrations of Pu. One test used a similar, yet more dilute salt waste that omitted the additional Pu. Researchers performed this last test as a comparison to previous experiment with actual high-level waste, sodium tetraphenylborate, and MST in a continuous mode.[8]

**Table 1. Actual high-level waste test design**

| Test | Description | Salt Waste | Sr | $MnO4^-$ | Formate | MST |
|---|---|---|---|---|---|---|
| 1 | Control | 5.6 M $Na^+$ | not added | not added | Not added | not added |
| 2A | Nominal $MnO_4^-$ | 5.6 M $Na^+$ | 0.01 M | 0.01 M | 0.045 M | not added |
| 2B | Nominal $MnO_4^-$ | 5.6 M $Na^+$ | 0.01 M | 0.01 M | 0.045 M | not added |
| 3A | Low $MnO_4^-$ | 5.6 M $Na^+$ | 0.01 M | 0.002 M | 0.009 M | not added |
| 3B | Low $MnO_4^-$ | 5.6 M $Na^+$ | 0.01 M | 0.002 M | 0.009 M | not added |
| 4A | $MnO_4^-$ w/out Sr | 5.6 M $Na^+$ | not added | 0.01 M | 0.045 M | not added |

| | | | | | | |
|---|---|---|---|---|---|---|
| 4B | $MnO_4^-$ w/out Sr | 5.6 M $Na^+$ | not added | 0.01 M | 0.045 M | not added |
| 5A | MST: Lot 33180 | 5.6 M $Na^+$ | not added | not added | Not added | 0.4 g/L |
| 5B | MST: Lot TNX | 5.6 M $Na^+$ | not added | not added | Not added | 0.4 g/L |
| 6 | MST: Lot TNX | 4.7 M $Na^+$ | not added | not added | Not added | 0.4 g/L |

### 3.1 Preparation of Salt Solutions

The actual high-level waste solutions used in these tests came from archived waste samples. The waste solution identified as 5.6 M $Na^+$ came from D. D. Walker and consisted of a composite of Tank 37H/44F waste. Personnel used this same material in the previous actual waste solvent extraction demonstration.[9] Researchers prepared the waste for testing by analyzing the received waste solution for sodium. Once analyzed, they then diluted the waste with 1.6 M NaOH to the desired sodium concentration of 5.8 M. This permitted adding small aqueous aliquots of the treatment additives so that the final test solutions would contain 5.6 M $Na^+$. Two dilutions and analyses occurred to achieve the desired concentration. The resulting solution volume equaled 1000 mL. Researchers then added 6 mL of acidic Americium/Curium solution (obtained from T. B. Peters[10]) and 66 μ g (contained in 1 mL of 5 M $HNO_3$) of $^{237}Np$ (from D. T. Hobbs 2.68 mg/mL $^{237}Np$ stock solution) to the diluted waste solution. Operators shook the solution to mix and allowed it to equilibrate over 1 week. We did not filter the solution prior to use. Filtered and unfiltered aliquots received analysis by titration, atomic adsorption (AA), ion chromatography (IC), inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-emission spectroscopy (ICP-ES), gamma radiolysis, and plutonium triphenyltrifluoroacetone scintillation analysis (PuTTA). Appendix 1 contains the final diluted composition of the waste solution. Table 2 shows specific components of relevance.

**Table 2. Components of interest in actual high-level waste 5.6 $Na^+$ salt solution**

| Component | Unit | Target | Soluble Concentration | Total Concentration | Process Limit |
|---|---|---|---|---|---|
| Na | M | 5.6 | 5.8 | 5.8 | None |
| $OH^-$ | M | as received | 3.7 | not measured | None |
| $^{90}Sr$ | nCi/g | as received | 154 | 626 | 40 |
| $^{237}Np$ | nCi/g | 0.06 | 0.0877 | 0.118 | 0.03 |
| Total U | μ g/L | as received | 7820 | 8110 | None |
| $^{238}Pu$ | nCi/g | | 235 | 828 | 18 |
| $^{239/240}Pu$ | nCi/g | 882 | 68 | 235 | 18 |
| Total Pu | nCi/g | | 303 | 1063 | 18 |

(The specific activities of $^{90}Sr$, $^{237}Np$, $^{238}Pu$, $^{239}Pu$, and $^{240}Pu$ are 137.1, 0.000705, 17.12, 0.0614, and 0.228 Ci/g, respectively.)

The waste solution identified as 4.7 M $Na^+$ came from T. B. Peters and consisted of a Tank 37H/44F/multi-tank composite. This material remained from the previous actual waste Small Tank Tetraphenylborate Continuously Stirred Tank Reactor (CSTR) demonstration.[8] As with the other waste solution, researchers prepared this waste for testing by analyzing the received waste solution for sodium. Once analyzed, they diluted the waste with 1.6 M NaOH to the desired endpoint of 4.7 M. Again, personnel used two dilutions and analyses to achieve the desired concentration. Unlike the previous waste solution, personnel did not add other radionuclides to the diluted waste solution. They shook the solution to mix and allowed it to equilibrate over 2 weeks. We did not filter the solution prior to use. Personnel analyzed filtered and unfiltered aliquots by titration, AA, IC, ICP-MS, ICP-ES, gamma radiolysis, and PuTTA. Appendix 2 contains the final diluted composition of the waste solution. Table 3 shows specific components of relevance.

### 3.2 Sr and Actinides Removal Tests

Researchers performed testing with the radioactive waste solutions in the SRTC Shielded Cells Facility. All tests used 250-mL polyethylene (PE) bottles fitted with a cap. Researchers prepared each test by placing 114 mL of the appropriate waste solution in the bottle. The MST tests initiated with the addition of a pre-dosed aliquot of 0.048 g MST (contained in a 6 mL aqueous slurry). The MST used in the tests came from two different batches. One batch, Lot 33180, represents a "qualified" batch of MST. The other source, TNX MST, consisted of residual MST from the prior demonstrations with actual waste.[8,9] This material originally came from a composite from MST drums located at the former SRS TNX site.

**Table 3. Specific components of interest in actual high-level waste 4.7 M $Na^+$ solution**

| Component | Unit | Target | Soluble Concentration | Total Concentration | Process Limit |
|---|---|---|---|---|---|
| Na | M | 4.7 | 4.8 | 4.8 | None |
| $OH^-$ | M | as received | 3.5 | 3.5 | None |
| $^{90}Sr$ | nCi/g | as received | 569 | 634 | 40 |
| $^{237}Np$ | nCi/g | as received | < 0.010 | < 0.016 | 0.03 |
| Total U | µ g/L | as received | 1800 | 1800 | None |
| $^{238}Pu$ | nCi/g | as received | 3.4 | 3.4 | 18 |
| $^{239/240}Pu$ | nCi/g | as received | < 0.4 | < 0.6 | 18 |
| Total Pu | nCi/g | as received | < 4 | < 4 | 18 |

(The specific activities of $^{90}Sr$, $^{237}Np$, $^{238}Pu$, $^{239}Pu$, and $^{240}Pu$ are 137.1, 0.000705, 17.12, 0.0614, and 0.228 Ci/g, respectively.)

Permanganate tests initiated with addition of 2 mL pre-dosed aliquots of 0.60 M $Sr(NO_3)_2$ to each test that required Sr addition. Note that personnel added a 2 mL aliquot of water to the Permanganate without Strontium tests as well as the control to maintain the same level of dilution. Operators shook the bottles (by manipulator) to mix. Approximately, 30 minutes later  timing varied slightly from test to test as determined by amount of time required to perform additions to all tests  personnel added pre-dosed 2 mL aliquots of $NaMnO_4$ to each test. The Nominal Permanganate and Permanganate without Strontium tests used 0.60 M stock $NaMnO_4$ solution while the Low Permanganate tests used 0.12 M stock $NaMnO_4$ solution. The control received 2 mL of water. After each addition, operators shook the test bottles. Approximately 30 minutes later, personnel added pre-dosed 2 mL aliquots of sodium formate to each test; the Nominal Permanganate and Permanganate without Strontium tests used 2.72 M stock sodium formate solution while the Low Permanganate tests used 0.54 M stock formate solution. Again the control received 2 mL of water. This completed initiation of the tests.

Researchers placed the test bottles on a shaker table at ambient temperature (21°C) and agitated continuously at a rate capable of suspending solid materials as visually observed. Sampling occurred 2, 5, 24, 96, and 168 hours after addition of the MST or Sr aliquots. Sampling involved removing a test bottle from the shaker, manually shaking to produce a homogeneous mixture, and pulling approximately 4.5 mL of the test mixture into a disposable 10-mL syringe. Personnel filtered the sample mixture through a 0.45 μ m nylon syringe filter disk and into a PE sample bottle. They capped the original test bottle and replaced in the shaker, typically within 5 minutes the start of sampling. After sampling all tests, the operator pipetted 1-mL portions of each filtered sample into a second set of pre-weighed, PE sample bottles containing ~49.5 mL of 2 M nitric acid. They weighed the diluted samples to determine the mass of sample transferred into each bottle. They shook the diluted samples and submitted for analysis by ICP-MS, PuTTA, and radiochemistry for U, Np, Pu, and Sr concentrations.

### 3.3 Quality Assurance

Personnel prepared non-radioactive solutions from reagent grade chemicals using calibrated balances checked daily before use.[11] The weights used for balance checks received calibration by the SRTC Standards Laboratory. Personnel verified the accuracy of glassware and pipettes used to measure volumes by gravimetric methods using water as a standard.[12] All Measurement and test equipment (M&TE) used in this task received calibration or verification for accuracy prior to their use. The Analytical Development Section performed all chemical analyses.

## 4.0 Results and Discussion

### 4.1 Permanganate Tests

Figures 1 - 4 present the results of permanganate tests with respect to each sorbate (i.e., $^{90}Sr$, total Pu, $^{237}Np$, and total U). Appendix 3 contains the numerical data. Table 4 provides a summary of average decontamination factors (DFs) for each of the sorbates after 5 and 24 h of reaction. The table also provides data previously reported by M. C. Duff for comparison.[13] However, the Duff data came from simulated waste tests with significant differences in the initial sorbate concentrations. A comparison of the actual waste and Duff's simulant test solutions is shown in Table 2. Numerical kinetics data from the Duff test (referred to as Test #24 in Reference 13) is contained at the end of Appendix 3.

**Table 2. Comparison of test solutions used in the current actual waste tests and Duff's simulant Test #24.**

| Component | Duff's Test #24 | Current Testing[a] |
|---|---|---|
| $Na^+$ (M) | 5.6 | 5.8 |
| $OH^-$ (M) | 1.33 | 3.7 |
| $NO_3^-$ (M) | 2.6 | 0.86 |
| $NO_2^-$ (M) | 0.34 | 0.58 |
| $Al(OH)_4^-$ (M) | 0.43 | <0.2 |
| $SO_4^{-2}$ (M) | 0.52 | 0.004 |
| $CO_3^{-2}$ (M) | 0.026 | <0.2 |
| Sr (μ g/L) | 660 | 1.4 (5.6)[b] |
| $^{37}Np$ (μ g/L) | 423 | 153 (all soluble) |
| $^{239/240}Pu$ (μ g/L) | 181 | 1185 (4100) |
| $^{238}U$ (μ g/L) | 10880 | 7820 (8110) |

[a]Values shown in parentheses represent the total concentration of the analyte.

[b]Actual waste test values shown are for Sr-90. Non-radioactive Sr was below detection.

### *4.1.1 Strontium Decontamination*

All three permanganate test variations reduced the $^{90}Sr$ concentration below the Saltstone WAC (40 nCi/g). Examination of the Sr data indicates that the bulk of decontamination occurred within 2 h. This observation mimics that observed in prior studies using permanganate to treat Hanford high-level waste.[3] The data indicate that the Permanganate ($MnO_4$) without Strontium tests proved the most rapid and the Nominal Permanganate tests proved the slowest of all test sets. However, no conclusions on the rates of decontamination should be made given the small number of samples and the minor differences in data. Ultimately, all three permanganate test variations yielded nearly the same level of decontamination by the end of one week (see Appendix 3).

**Table 5. Comparison of average decontamination factors (DFs) for each permanganate test**

| | $^{90}$Sr DF | | Pu DF | | $^{237}$Np DF | | U DF | |
|---|---|---|---|---|---|---|---|---|
| **Test** | **5 h** | **24 h** | **5 h** | **24 h** | **5 h** | **24 h** | **5 h** | **24 h** |
| Control | 1.0 | 1.0 | 1.1 | 1.0 | 1.3 | 1.0 | 1.1 | 1.0 |
| Nominal $MnO_4$ | 47 | 64 | 1.6 | 1.7 | 0.9 | 1.9 | 1.0 | 1.1 |
| Low $MnO_4$ | 62 | 74 | 1.0 | 1.3 | 0.9 | 1.2 | 1.0 | 1.1 |
| $MnO_4$ without Sr | 110 | 94 | 1.0 | 0.9 | 0.8 | 1.2 | 1.0 | 1.0 |
| Duff's Test #24[13] | 14 | 78 | 4.2 | 3.5 | 1.2 | 1.2 | 1.2 | 1.3 |

### *4.1.2 Plutonium Decontamination*

Plutonium behaved differently than strontium. Figure 2 and Table 5 show that only the Nominal Permanganate tests showed significant decontamination within the planned 24 hour process cycle. In all cases, removal efficiency peaked within 5 hours and then declined over the remaining test period. All of the tests failed to reduce the Pu concentration below the Saltstone WAC (total alpha = 18 nCi/g). Duff's Test #24 showed similar kinetics (i.e., decontamination maximized very early in the test). However, Duff's test showed better DFs for simulant with approximately 4% as much Pu. All Pu remained soluble in Duff's test while only 29% of the Pu in the current tests remained soluble.

### *4.1.3 Neptunium Decontamination*

Figure 3 and Table 5 provide neptunium behavior for the conditions tested. The Np concentration data suggests that addition of permanganate resulted in dissolution of some Np solids present in the sludge solids. This may occur due to the presence of $MnO_4^-$, which is a strong oxidizer. Generally, higher oxidation states of actinides exhibit higher solubilities. The oxidation potential for $MnO_4^-$ under alkaline conditions is sufficiently high to oxidize Np(V) to Np(VI). Thus it is possible that the higher Np concentration may reflect oxidation of Np(V) to Np(VI). If true, this effect might produce a delay in decontamination, as observed with this data set. In the experiments using Permanganate without Strontium, the dissolution appeared complete within the accuracy of the analyses. Given the complexity of the tests due to the changing systems, comparison of the tests is difficult. The data do not indicate which condition offers the best decontamination. None of the tests achieved the required level of decontamination (Np = 0.03 nCi/g).

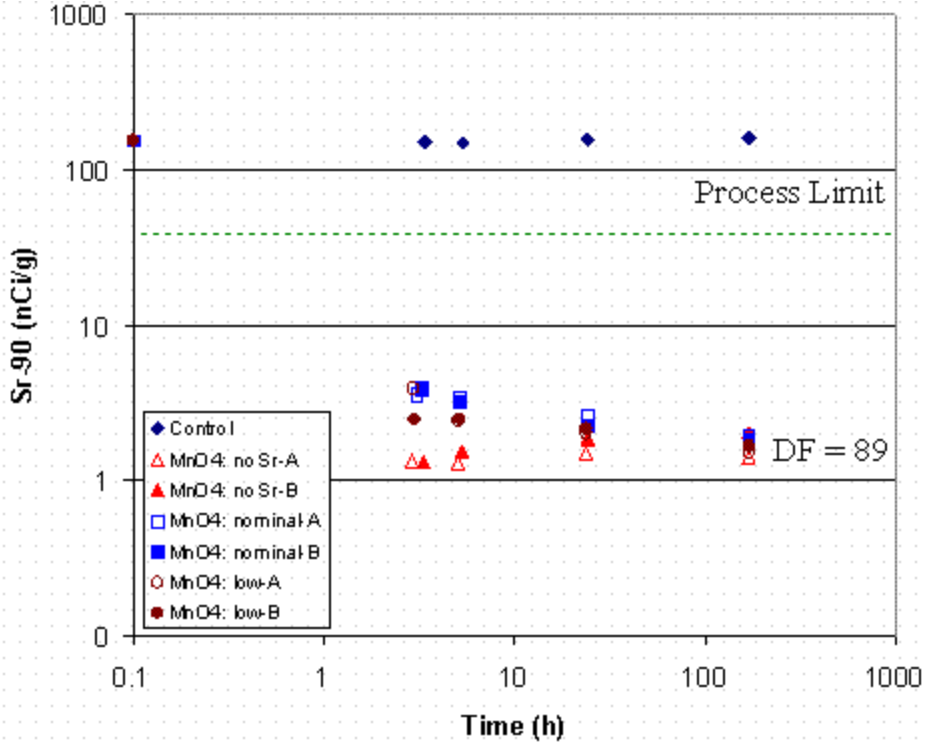


**Figure 1. Concentration of $^{90}$Sr during the permanganate tests**

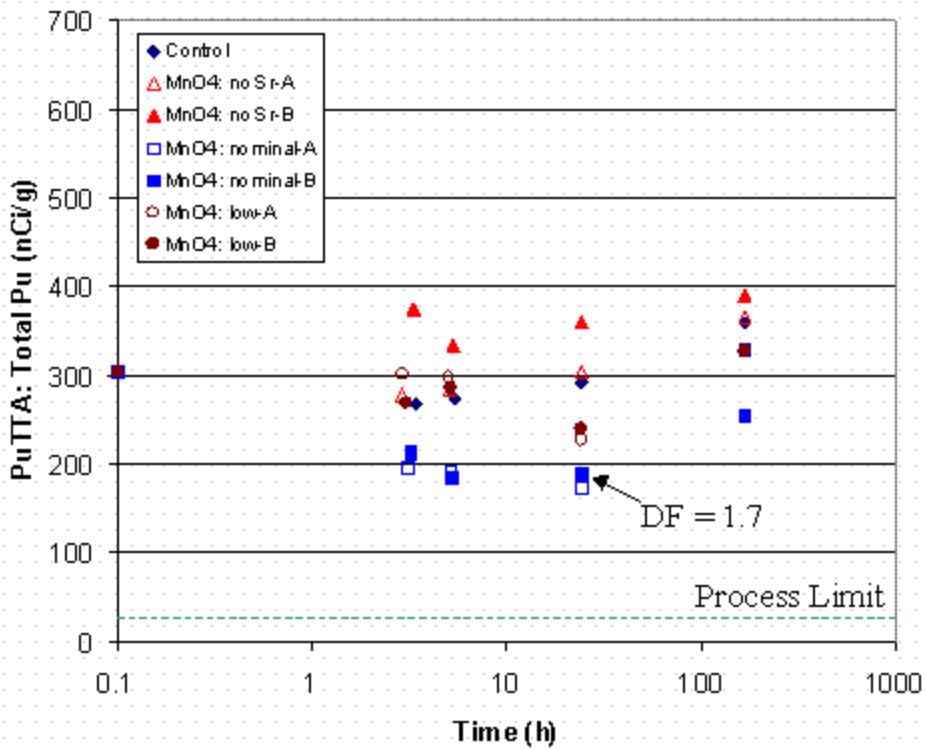


**Figure 2. Concentration of plutonium during the permanganate tests**

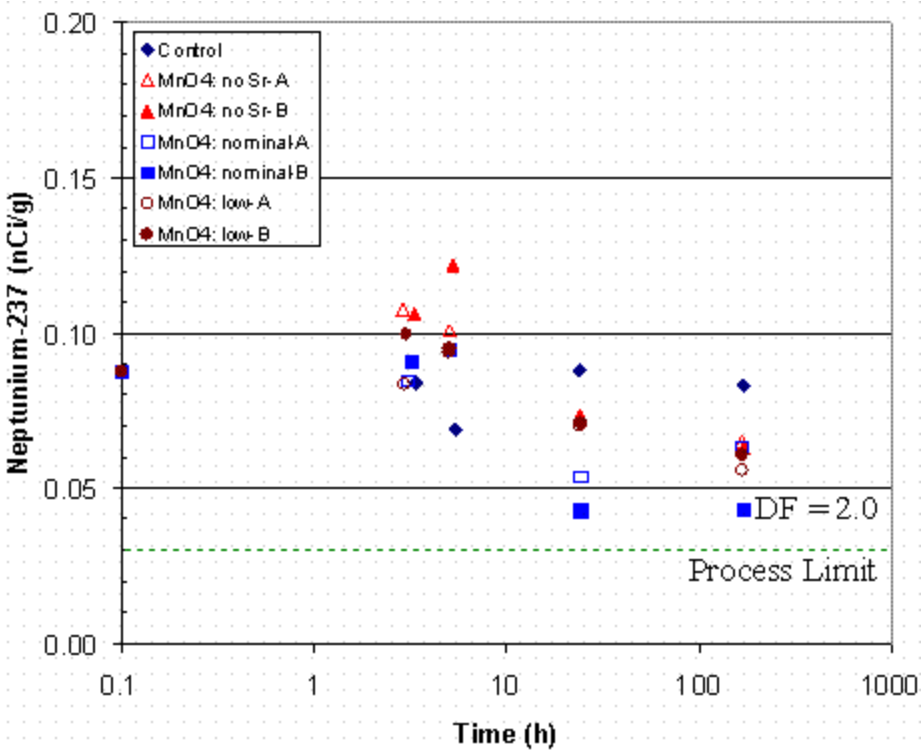


**Figure 3. Concentration of $^{237}$Np during the permanganate tests**

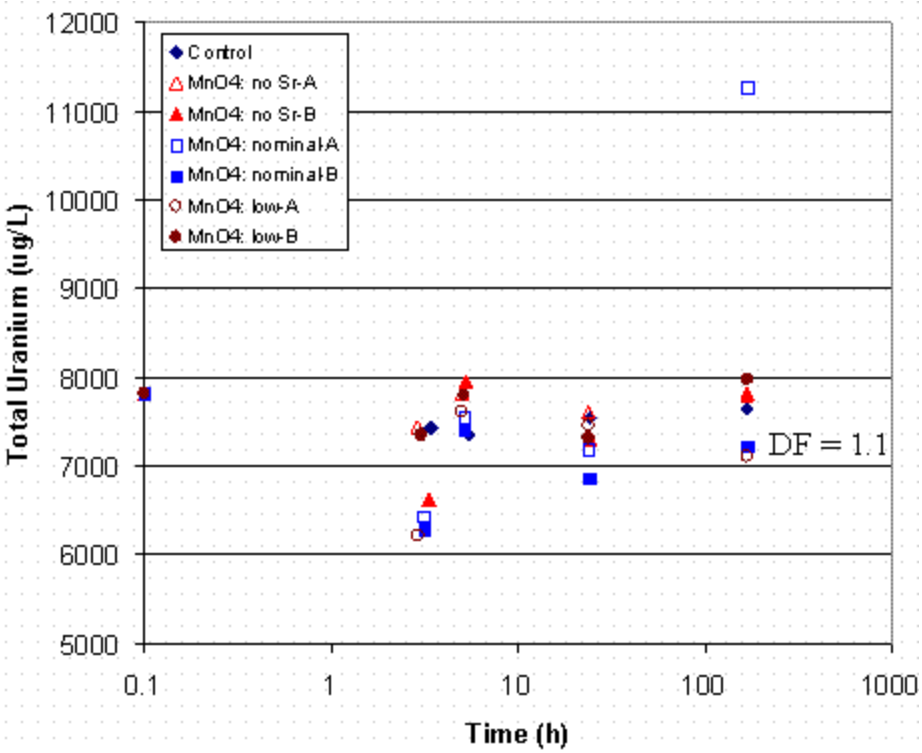


**Figure 4. Concentration of uranium during the permanganate test**

Given the complexity of the tests due to the changing systems, comparison of the tests is difficult. The data do not indicate which condition offers the best decontamination. None of the tests achieved the required level of decontamination (Np = 0.03 nCi/g).

### *4.1.4 Uranium Decontamination*

Figure 4 provides the data for uranium removal by permanganate treatment. Uranium behaved similarly to that reported by Duff.[13] Less than 10% of the uranium was removed in any of the tests within 24 or 168 hours. Note that the waste does not require uranium removal. However, we monitor its removal efficiency since it competes with the other sorbates in MST pre-treatment and is removed in the $MnO_4^-$ treatment process (based on simulant tests). None of the tests distinguished themselves from the others. The low DF values indicate little removal of uranium in these tests.

In summary, all three permanganate test variations reduced Sr to a concentration below the Waste Acceptance Criteria. Neither Pu nor Np decontamination proved sufficient to reduce their concentrations below their respective process limits. One possible cause for poor performance is the increased concentration of plutonium present in these tests.

## 4.2 MST Tests with 5.6 M $Na^+$ Solution

Figures 5 - 8 present monosodium titanate removal efficiency data for $^{90}Sr$, total Pu, $^{237}Np$, and total U, respectively. Appendix 3 contains numerical test data. Table 4 provides a summary of DFs for each of the sorbates, with respect to each MST lot, after 24 and 168 h of reaction. The table also provides data previously reported by K. M. Marshall for comparison.[14] This data came from a simulant test with the same batch of MST and with a similar salt composition (see Table 3 for the solution comparison).

**Table 3. Comparison of test solution compositions for actual waste used in current testing and simulant used by Marshall.[14]**

| Component | Marshall | Current Testing[a] |
|---|---|---|
| $Na^+$ (M) | 5.6 | 5.8 |
| $OH^-$ (M) | 3.0 | 3.7 |
| $NO_3^-$ (M) | 1.0 | 0.86 |
| $NO_2^-$ (M) | 0.50 | 0.58 |
| $Al(OH)_4^-$ (M) | 0.10 | <0.2 |
| $SO_4^{-2}$ (M) | 0.49 | 0.004 |
| $CO_3^{-2}$ (M) | 0.02 | <0.2 |
| Sr (μ g/L) | 621 | 1.4 (5.6)[b] |
| $^{37}Np$ (μ g/L) | 241 | 153 (all soluble) |
| $^{239/240}Pu$ (μ g/L) | 231 | 1185 (4100) |
| $^{238}U$ (μ g/L) | 7050 | 7820 (8110) |

[a]Values shown in parentheses represent the total concentration of the analyte.
[b]Values shown are for Sr-90. Non-radioactive Sr was below detection.

The MST reduced the concentration of Sr below the process limit. The two different lots behaved very similarly. However, Marshall's comparative data with simulants showed significantly better DFs for Sr. The cause of the low DFs observed in these tests remains undetermined. The MST failed to remove either Pu or Np to a concentration below the Saltstone WAC. Again, both MST lots behaved similarly in case. Pu DFs were slightly lower in

the current tests than that observed by Marshall. Differences in Np and U DFs in the data sets were negligible. An increased MST concentration is suggested to decontaminate this waste, with respect to Pu and Np, to a satisfactory level.

**Table 4. Comparison of DFs for each MST test with 5.6 M $Na^+$ solution**

| | $^{90}Sr$ DF | | Pu DF | | $^{237}Np$ DF | | U DF | |
|---|---|---|---|---|---|---|---|---|
| **Test** | **24 h** | **168 h** | **24 h** | **168 h** | **24 h** | **168 h** | **24 h** | **168 h** |
| Control | 1.0 | 1.0 | 1.0 | 0.8 | 1.0 | 1.1 | 1.0 | 1.0 |
| MST: Lot 33180 | 10 | 10 | 2.4 | 3.1 | 2.0 | 2.1 | 1.2 | 1.2 |
| MST: TNX | 11 | 12 | 2.5 | 2.4 | 1.9 | 1.9 | 1.2 | 1.2 |
| K. Marshall's data[14] | 123 | 155 | 4.1 | 9.1 | 1.5 | 2.0 | 1.4 | 1.6 |

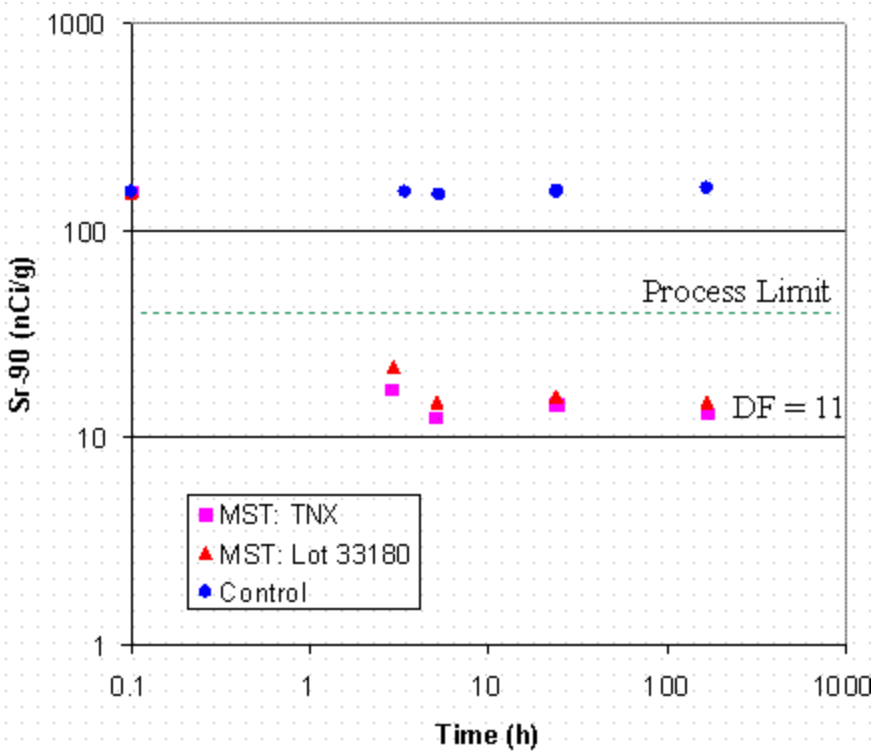


**Figure 5. Concentration of $^{90}Sr$ during the MST test with 5.6 M $Na^+$ solution.**

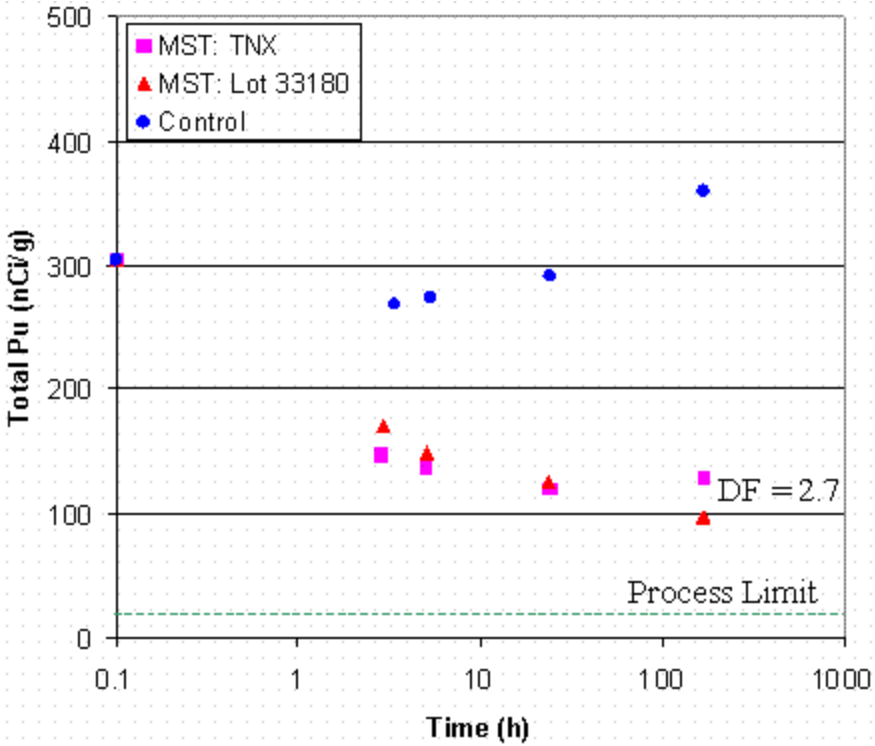


**Figure 6. Concentration of plutonium during the MST test with 5.6 M $Na^+$ solution**

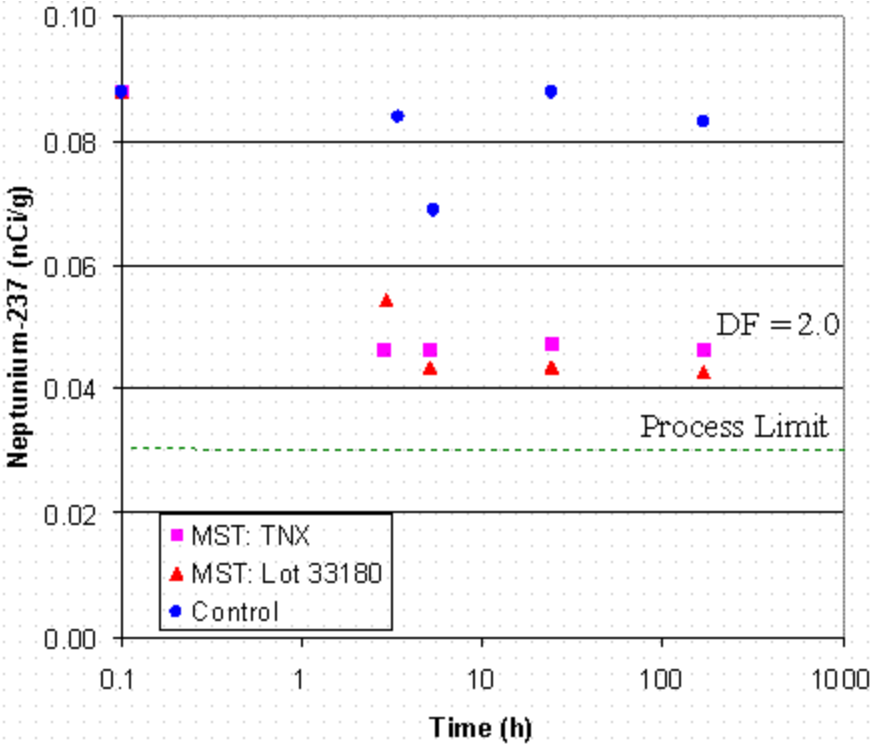


**Figure 7. Concentration of $^{237}Np$ during the MST test with 5.6 M $Na^{+}$ solution**

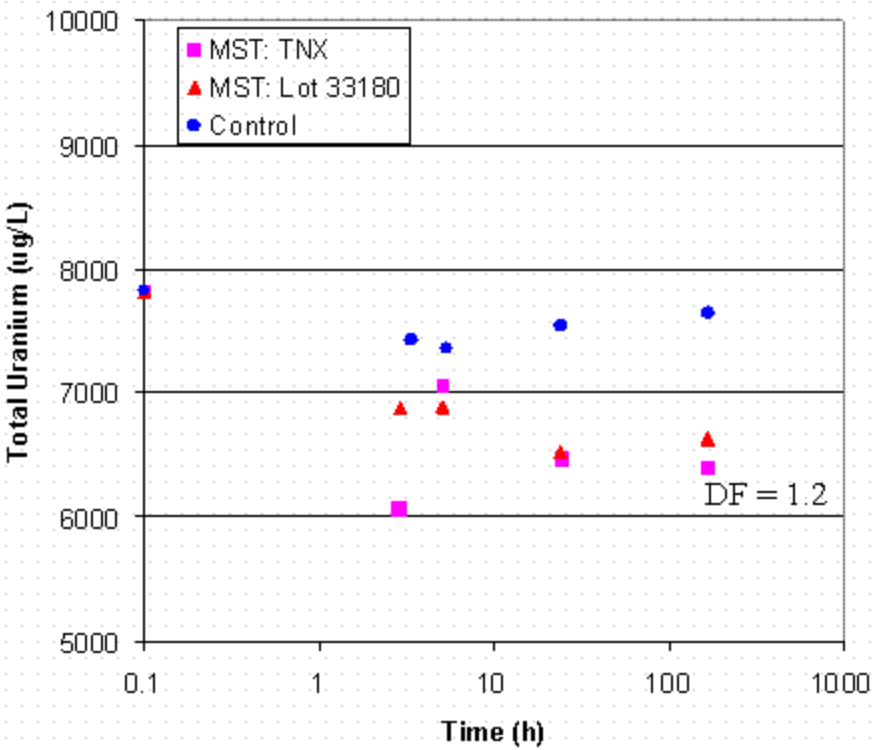


**Figure 8. Concentration of uranium during the MST test with 5.6 M $Na^+$ solution**

### 4.3 MST Test with 4.7 M $Na^+$ Solution

The purpose of this test was to provide a measure of the effect of mixing on MST treatment. Minimal information is available to assess the influence of mixing on sorbtion behavior. Comparison of the solvent[9] extraction and tetraphenylborate CSTR[8] demonstrations with actual waste showed a marked loss in efficiency as mixing performance decreased. Hence, we added this test to provide a more reliable examination of the influence of mixing.

Figures 9 - 12 provide the removal efficiency data for $^{90}Sr$, total Pu, $^{237}Np$, and total U, respectively, obtained by contacting with MST in a 4.7 M $Na^+$ solution. Appendix 3 contains numerical test data. Table 5 provides a summary of DFs for each of the sorbates after 24 and 168 h of reaction. The table also provides data previously obtained from the Small Tank Tetraphenylborate Precipitation Actual waste demonstration for comparison. Testing, in both cases, used the same waste salt solution.

Strontium decontamination in the current test satisfied the Saltstone WAC. The observed DF after 168 hours closely matched the steady state DF obtained in the CSTR demonstration. Plutonium and neptunium concentrations fell below the process limit prior to the start of testing. The CSTR demonstration achieved significantly better Pu decontamination than observed in the current test. The earlier demonstration did not track neptunium removal efficiency. Uranium DF in the current test proved slightly poorer than observed in the continuous test. The data suggest that the different

levels of agitation between the continuous precipitation process and that of the orbital shakers used in the current batch tests provided similar decontamination results (i.e., use of the orbital mixer in the current tests provided an adequate level of mixing).

**Table 5. Batch and Continuous Reaction DFs for MST with 4.7 M $Na^+$ solution**

| | $^{90}Sr$ DF | | Pu DF | | $^{237}Np$ DF | | U DF | |
|---|---|---|---|---|---|---|---|---|
| **Test** | **24 h** | **168 h** | **24 h** | **168 h** | **24 h** | **168 h** | **24 h** | **168 h** |
| MST: TNX - Batch | 23 | 39 | 7.1 | 5.9 | > 1.5 | 4.1 | 1.5 | 1.5 |
| MST: TNX - CSTR Demo[8] (steady state values) | 46 | | 155 | | not measured | | 2.4 | |

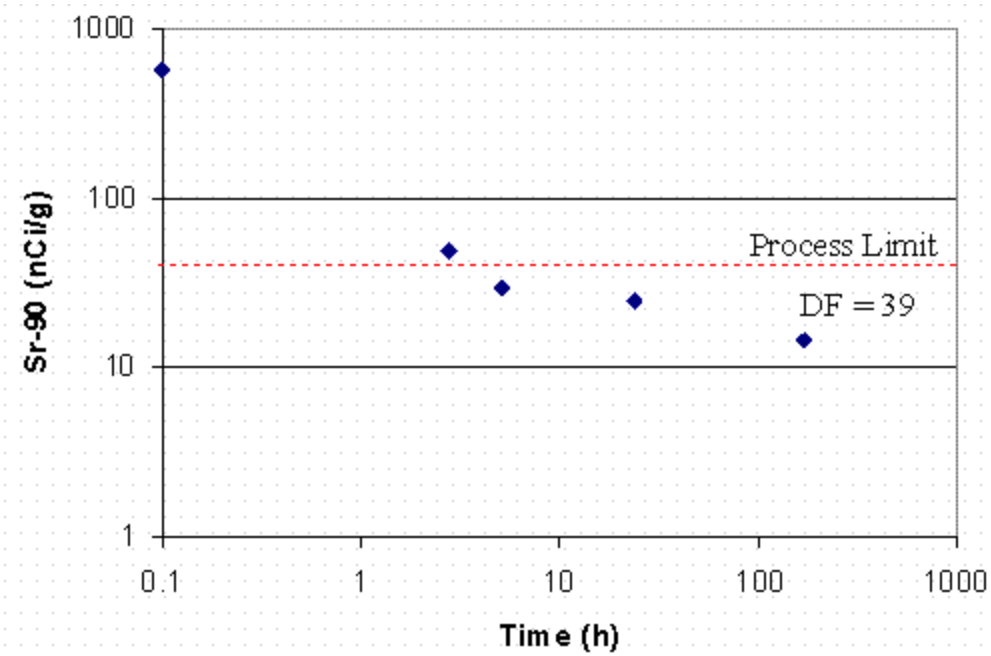


**Figure 9. Concentration of $^{90}Sr$ during the MST test with 4.7 M $Na^+$ solution**

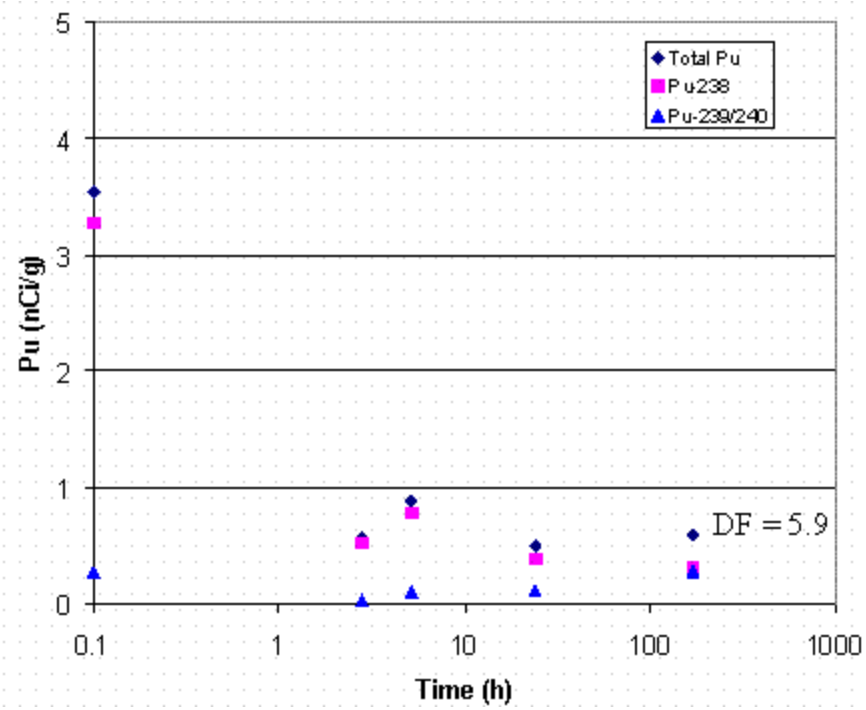


**Figure 10. Concentration of plutonium during the MST test with 4.7 M $Na^+$ solution**

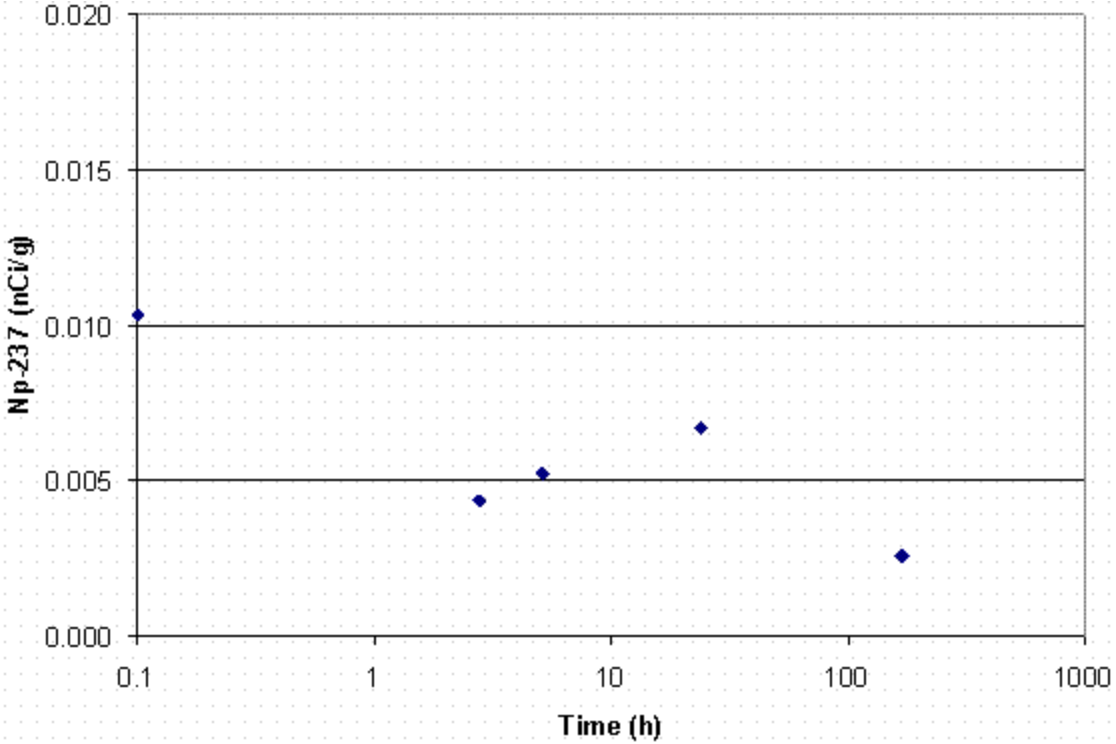


**Figure 11. Concentration of $^{237}Np$ during the MST test with 4.7 M $Na^{+}$ solution**

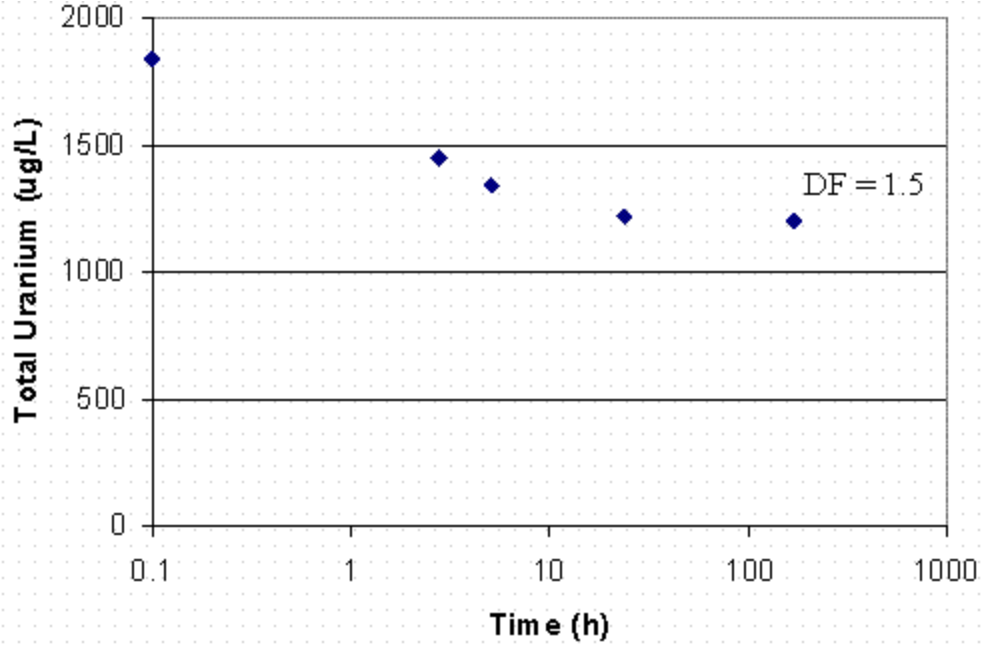


**Figure 12. Concentration of uranium during the MST test with 4.7 M $Na^+$ solution**

## 4.4 Comparison of Treatment Options

The primary objective of this test program was to investigate the ability of permanganate treatment to remove Sr, Pu, and Np to satisfactory levels. The performance of permanganate relative to the current baseline process, MST adsorption, provides the most direct assessment. Table 9 compares the average DFs obtained from the Nominal Permanganate tests with that of the two MST tests. Both test sets used the same waste solution, thereby negating concentration effects. The table contrasts the 24-h DFs since that sampling tie matches the cycle time in the proposed facility. The comparison shows that Nominal Permanganate provided superior $^{90}Sr$ decontamination while MST gave only better Pu decontamination. Neptunium and uranium DFs proved nearly identical in both test sets. Since, neither MST nor permanganate successful achieved Pu and Np decontamination, we recommend further tests to determine the amount of added sorbent required in each process option to successfully treat this waste.

**Table 9. Comparison of average 24 hour DFs for Nominal $MnO_4$ and MST with 5.6 M $Na^+$ solution**

| | $^{90}Sr$ | Pu | $^{237}Np$ | U |
|---|---|---|---|---|
| **Test** | **24 h DF** | **24 h DF** | **24 h DF** | **24 h DF** |
| Nominal $MnO_4$ | 64 | 1.7 | 1.9 | 1.1 |
| MST | 10.2 | 2.5 | 1.9 | 1.2 |

## 5.0 Quality Assurance

The following documents govern the work reported in this document.

- D. T. Hobbs, T. B. Peters, M. J. Barnes, M. C. Duff and K. M. Marshall, "Task Technical and Quality Assurance Plan for FY01 Strontium and Actinide Removal Testing," WSRC-RP-2001-00188, Rev. 1, July 31, 2001.
- Savannah River Site Salt Processing Project Research and Development Program Plan, PNNL-13253, Rev. 1, November 2000.
- Notebook WSRC-NB-2001-00168 (M. J. Barnes) contains the experimental data obtained from this work.

## 6.0 References

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2. H. H. Elder, "Salt Blending Basis for Revision 12 of the HLW System Plan", HLW-SDT-2001-00146, Rev. 0, April 26, 2001.
3. V. F. Peretrukin, V. I. Silin, A., V. Kareta, A. V. Gelis, V. P. Shilov, K. E. German, E. V. Firsova, A. G. Maslennikov and V. E. Trushina, "Purification of Alkaline Solutions and Wastes from Actinides and Technetium by Coprecipitation with Some Carriers Using the Method of Appearing Reagents: Final Report", Report PNNL-11988, UC-2030, Pacific Northwest National Laboratory, September 1998.
4. D. T. Hobbs, M. G. Bronikowski, T. B. Edwards and R. L. Pulmano, "Final Report on Phase III Testing of Monosodium Titanate Adsorption Kinetics", WSRC-TR-99-00134, Rev. 0, May 28, 1999.
5. D. T. Hobbs and R. L. Pulmano, "Phase IV Simulant Testing of Monosodium Titanate Adsorption Kinetics", WSRC-TR-99-0219, Rev. 0, June 29, 1999.
6. D. T. Hobbs and R. L. Pulmano, "Phase IV Testing of Monosodium Titanate Adsorption Kinetics with Radioactive Waste", WSRC-TR-99-00286, Rev. 0, September 3, 1999.
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10. T. B. Peters, D. T. Hobbs, D. P. Diprete, C. C. Diprete and S. D. Fink, "Demonstration of Disposal of Americium and Curium Legacy Material Through the High-level waste System: Results from Baseline Flowsheet Studies", WSRC-TR-2001-00503, Rev. 0, October 23, 2001.
11. D. D. Walker, "Balance Check" (U), Manual L12.1, Procedure IWT-OP-011, Rev. 3, December 6, 1994.
12. D. D. Walker, "Calibration of Laboratory Glassware" (U), Manual L12.1, Procedure IWT-OP-009, Rev. 3, May 8, 1995.
13. M. C. Duff, D. T. Hobbs, and J. Siler, "Permanganate Treatment Optimization Studies for Sr and Actinide Removal from HLW Simulants", WSRC-TR-2002-00027, January 14, 2002.
14. M. J. Barnes, T. B. Edwards, D. T. Hobbs, and K. M. Marshall, "Strontium and Actinide Removal Testing with Monosodium Titanate and Other Sorbents", WSRC-TR-2001-00436, Rev. 0, October 29, 2001.

## Appendix 1. Composition of diluted 5.6 M $Na^+$ actual high-level waste

| Analytical Method | Analyte | Units | Soluble Concentration | Total Concentration |
|---|---|---|---|---|
| Titration | hydroxide | M | 3.7 | Not measured |
| IC | nitrate | M | 0.864 | Not measured |
| IC | nitrite | M | 0.584 | Not measured |
| IC | sulfate | M | 0.004 | Not measured |
| IC | chloride | M | 0.005 | Not measured |
| IC | formate | M | < 0.03 | Not measured |
| IC | oxalate | M | < 0.01 | Not measured |
| Titration | carbonate | M | < 0.2 | Not measured |
| Titration | aluminate | M | < 0.2 | Not measured |
| AA | sodium | M | 5.38 | Not measured |
| AA | potassium | M | 0.041 | Not measured |
| ICP-ES | Al | mg/L | 6069 | 6054 |
| ICP-ES | B | mg/L | 78.9 | 78.5 |
| ICP-ES | Cr | mg/L | 104 | 102 |
| ICP-ES | Fe | mg/L | 17 | 56.7 |
| ICP-ES | Mg | mg/L | < 2 | < 2 |
| ICP-ES | Mn | mg/L | < 2 | 2.31 |
| ICP-ES | Mo | mg/L | 88.8 | 91.6 |
| ICP-ES | Na | M | 5.79 | 5.77 |
| ICP-ES | P | mg/L | 233 | 211 |
| ICP-ES | Si | mg/L | 156 | 137 |
| ICP-ES | Zn | mg/L | 9.68 | 10.3 |
| Gamma | $^{137}Cs$ | dpm/mL | 1.89E+09 | Not measured |
| PuTTA | $^{238}Pu$ | dpm/mL | 6.43E+05 | 2.26E+06 |
| PuTTA | $^{239/240}Pu$ | dpm/mL | 1.85E+05 | 6.41E+05 |
| $^{90}Sr$ | $^{90}Sr$ | dpm/mL | 4.20E+05 | 1.71E+06 |
| ICP-MS | Mass 232 | ug/L | 217.6 | 158.6 |
| ICP-MS | Mass 234 | ug/L | 38.10 | 44.77 |
| ICP-MS | Mass 235 | ug/L | 84.65 | 105.7 |
| ICP-MS | Mass 236 | ug/L | 44.43 | 42.76 |
| ICP-MS | Mass 237 | ug/L | 153.0 | 137.4 |
| ICP-MS | Mass 238 | ug/L | 7653 | 7961 |

| | | | | |
|---|---|---|---|---|
| ICP-MS | Mass 239 | ug/L | < 18 | 40.94 |
| ICP-MS | Mass 240 | ug/L | 371.5 | 1088 |
| ICP-MS | Mass 241 | ug/L | < 18 | 154.46 |
| ICP-MS | Mass 242 | ug/L | < 18 | 70.57 |
| ICP-MS | Mass 243 | ug/L | 30.44 | 3236 |
| ICP-MS | Mass 244 | ug/L | < 18 | 676.8 |
| ICP-MS | Mass 245 | ug/L | < 18 | 42.29 |
| ICP-MS | Mass 246 | ug/L | < 18 | 39.23 |

## Appendix 2. Composition of diluted 4.7 M $Na^+$ actual high-level waste

| Analytical Method | Analyte | Units | Soluble Concentration | Total Concentration |
|---|---|---|---|---|
| Titration | Hydroxide | Molar | 3.5 | not measured |
| IC | Nitrate | mg/L | 0.854 | not measured |
| IC | Nitrite | mg/L | 0.605 | not measured |
| IC | Sulfate | mg/L | 0.006 | not measured |
| IC | Halides | mg/L | 0.003 | not measured |
| IC | Formate | mg/L | < 0.03 | not measured |
| IC | Oxalate | mg/L | < 0.01 | not measured |
| Titration | Carbonate | Molar | < 0.2 | not measured |
| Titration | Aluminate | Molar | < 0.2 | not measured |
| AA | Sodium | Molar | 4.96 | not measured |
| AA | Potassium | Molar | 0.031 | not measured |
| ICP-ES | Al | mg/L | 6672 | 6661 |
| ICP-ES | B | mg/L | 69.1 | 73.2 |
| ICP-ES | Cr | mg/L | 134 | 137 |
| ICP-ES | Fe | mg/L | 31.7 | 19.4 |
| ICP-ES | Mg | mg/L | < 2 | < 2 |
| ICP-ES | Mn | mg/L | < 2 | < 2 |
| ICP-ES | Mo | mg/L | 95.6 | 106 |
| ICP-ES | Na | M | 4.86 | 4.75 |
| ICP-ES | P | mg/L | 234 | 243 |
| ICP-ES | Si | mg/L | 158 | 177 |
| ICP-ES | Zn | mg/L | 5.85 | 6.26 |

| | | | | |
|---|---|---|---|---|
| Gamma | $^{137}Cs$ | dpm/mL | 2.53E+09 | not measured |
| PuTTA | $^{238}Pu$ | dpm/mL | 8.72E+03 | 8.18E+03 |
| PuTTA | $^{239/240}Pu$ | dpm/mL | 7.07E+02 | 4.83E+03 |
| $^{90}Sr$ | $^{90}Sr$ | dpm/mL | 1.52E+06 | 1.69E+06 |
| ICP-MS | Mass 232 | ug/L | 93.48 | 62.60 |
| ICP-MS | Mass 234 | ug/L | 29.35 | 31.65 |
| ICP-MS | Mass 235 | ug/L | 86.47 | 119.0 |
| ICP-MS | Mass 236 | ug/L | 48.66 | 64.77 |
| ICP-MS | Mass 237 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 238 | ug/L | 1673 | 1581 |
| ICP-MS | Mass 239 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 240 | ug/L | < 18 | 73.92 |
| ICP-MS | Mass 241 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 242 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 243 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 244 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 245 | ug/L | < 18 | < 28 |
| ICP-MS | Mass 246 | ug/L | < 18 | < 28 |

## Appendix 3. Sorbate data tables

**Test 1: Control**

| Time | $^{238}Pu$ | $^{239/240}Pu$ | Total Pu | $^{90}Sr$ | $^{237}Np$ | Total U |
|---|---|---|---|---|---|---|
| (h) | (nCi/g) | (nCi/g) | (nCi/g) | (nCi/g) | (nCi/g) | (ug/L) |
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 3.4 | 208.6 | 59.6 | 268.20 | 153.3 | 0.08389 | 7430 |
| 5.4 | 211.2 | 62.4 | 273.57 | 148.8 | 0.06901 | 7354 |
| 24.0 | 226.9 | 64.6 | 291.53 | 156.7 | 0.08788 | 7546 |
| 168.3 | 279.7 | 79.6 | 359.34 | 160.1 | 0.08298 | 7642 |

**Test 2A: Nominal Permanganate**

| Time | $^{238}Pu$ | $^{239/240}Pu$ | Total Pu | $^{90}Sr$ | $^{237}Np$ | Total U |
|---|---|---|---|---|---|---|
| (h) | (nCi/g) | (nCi/g) | (nCi/g) | (nCi/g) | (nCi/g) | (ug/L) |
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 3.1 | 154.5 | 41.8 | 196.27 | 3.5 | 0.08490 | 6428 |
| 5.2 | 147.3 | 44.8 | 192.08 | 3.4 | 0.09464 | 7550 |
| 24.0 | 135.5 | 38.1 | 173.65 | 2.6 | 0.05363 | 7181 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 168.2 | 257.3 | 70.8 | 328.11 | 1.9 | 0.06326 | 11256 |

**Test 2B: Nominal Permanganate**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 3.2 | 162.7 | 51.3 | 213.99 | 4.0 | 0.09091 | 6287 |
| 5.2 | 143.7 | 41.6 | 185.28 | 3.2 | 0.09518 | 7395 |
| 24.0 | 147.4 | 42.5 | 189.84 | 2.3 | 0.04242 | 6869 |
| 168.3 | 196.9 | 58.1 | 255.05 | 1.9 | 0.04304 | 7221 |

**Test 3A: Low Permanganate**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 2.9 | 235.2 | 67.1 | 302.28 | 4.0 | 0.08368 | 6208 |
| 5.0 | 235.7 | 62.5 | 298.23 | 2.5 | 0.09382 | 7618 |
| 23.9 | 177.0 | 52.0 | 229.02 | 2.0 | 0.07016 | 7463 |
| 168.1 | 248.9 | 77.9 | 326.75 | 1.5 | 0.05560 | 7116 |

**Test 3B: Low Permanganate**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 3.0 | 213.9 | 54.7 | 268.64 | 2.5 | 0.09950 | 7356 |
| 5.1 | 223.9 | 63.1 | 287.03 | 2.5 | 0.09448 | 7806 |
| 23.9 | 186.6 | 53.7 | 240.29 | 2.1 | 0.07109 | 7312 |
| 168.1 | 257.5 | 70.3 | 327.80 | 1.7 | 0.06086 | 7986 |

**Test 4A: Permanganate without Strontium**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 2.9 | 215.8 | 61.8 | 277.60 | 1.3 | 0.10728 | 7449 |
| 5.0 | 219.8 | 64.0 | 283.72 | 1.3 | 0.10102 | 7815 |
| 23.9 | 235.7 | 67.7 | 303.43 | 1.5 | 0.07221 | 7604 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 168.0 | 280.2 | 85.9 | 366.12 | 1.4 | 0.06483 | 7800 |

**Test 4B: Permanganate without Strontium**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 3.3 | 291.4 | 82.9 | 374.26 | 1.3 | 0.10620 | 6622 |
| 5.3 | 261.8 | 72.2 | 334.01 | 1.5 | 0.12186 | 7955 |
| 24.0 | 278.8 | 81.0 | 359.83 | 1.8 | 0.07374 | 7298 |
| 168.3 | 306.9 | 83.7 | 390.61 | 2.0 | 0.06299 | 7824 |

**Test 5A: MST: Lot 33180**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 2.9 | 134.5 | 36.4 | 170.94 | 21.7 | 0.05430 | 6878 |
| 5.1 | 115.1 | 33.4 | 148.52 | 14.8 | 0.04332 | 6892 |
| 24.0 | 98.2 | 27.2 | 125.39 | 15.8 | 0.04360 | 6524 |
| 168.1 | 75.0 | 22.6 | 97.57 | 14.9 | 0.04276 | 6632 |

**Test 5B: MST: Lot TNX**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 235.4 | 67.9 | 303.27 | 153.7 | 0.08771 | 7820 |
| 2.9 | 111.5 | 35.8 | 147.30 | 17.0 | 0.04619 | 6067 |
| 5.1 | 106.0 | 31.7 | 137.70 | 12.5 | 0.04637 | 7070 |
| 24.0 | 92.9 | 26.9 | 119.76 | 14.4 | 0.04729 | 6480 |
| 168.1 | 101.2 | 27.8 | 128.99 | 13.1 | 0.04635 | 6402 |

**Test 6: MST: Lot TNX (4.7 M $Na^+$)**

| Time (h) | $^{238}Pu$ (nCi/g) | $^{239/240}Pu$ (nCi/g) | Total Pu (nCi/g) | $^{90}Sr$ (nCi/g) | $^{237}Np$ (nCi/g) | Total U (ug/L) |
|---|---|---|---|---|---|---|
| 0 | 3.28 | 0.27 | 3.54 | 569.2 | 0.01031 | 1837 |
| 2.8 | 0.53 | 0.03 | 0.56 | 48.7 | 0.00434 | 1452 |
| 5.1 | 0.79 | 0.11 | 0.89 | 29.4 | 0.00519 | 1343 |
| 24.0 | 0.38 | 0.12 | 0.50 | 24.6 | 0.00670 | 1216 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 168.0 | 0.32 | 0.28 | 0.60 | 14.5 | 0.00254 | 1204 |

**Duff's Permanganate Test #24**

| Time (h) | $^{239/240}Pu$ (ug/L) | $^{85}Sr$ (ng/L) | $^{237}Np$ (ug/L) | Total U (ug/L) |
|---|---|---|---|---|
| 0 | 181 | 1.108 | 423 | 10880 |
| 4.6 | 43.2 | 0.082 | 365 | 8952 |
| 24.7 | 52.1 | 0.014 | 359 | 8545 |
| 166.1 | 19.7 | 0.009 | 303 | 9244 |