Development of an Alternative Treatment Scheme for Sr/TRU Removal: Permanganate Treatment of AN-107 Waste

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July 2000

Prepared for BNFL, Inc.
under Contract W375-LC-98-4168
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Battelle, Pacific Northwest Division
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Summary

A number of Hanford tanks received waste containing organic complexants, which increase the solubility of Sr-90 and transuranic (TRU) elements. Wastes from these tanks require additional pretreatment to remove Sr-90 and TRU for immobilization as low activity waste (Waste Envelope C). The baseline pretreatment process for Sr/TRU removal was isotopic exchange and precipitation with added strontium and iron. However, studies at both Battelle and Savannah River Technology Center (SRTC) have shown that the Sr/Fe precipitates were very difficult to filter. This was a result of the formation of poor filtering iron solids. An alternate treatment technology was needed for Sr/TRU removal. Battelle had demonstrated that permanganate treatment was effective for decontaminating waste samples from Hanford Tank SY-101 and proposed that permanganate be examined as an alternative Sr/TRU removal scheme for complexant-containing tank wastes such as AN-107.

Battelle conducted preliminary small-scale experiments to determine the effectiveness of permanganate treatment with AN-107 waste samples that had been archived at Battelle from earlier studies. Three series of experiments were performed to evaluate conditions that provided adequate Sr/TRU decontamination using permanganate treatment. The final series included experiments with actual AN-107 diluted feed that had been obtained specifically for BNFL process testing. Conditions that provided adequate Sr/TRU decontamination were identified. A free hydroxide concentration of 0.5M provided adequate decontamination with added Sr of 0.05M and permanganate of 0.03M for archived AN-107. The best results were obtained when reagents were added in the sequence Sr followed by permanganate with the waste at ambient temperature. The reaction conditions for Sr/TRU removal will be further evaluated with a 1-L batch of archived AN-107, which will provide a large enough volume of waste to conduct crossflow filtration studies (Hallen et al. 2000a).
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1.0 Introduction

This report summarizes work performed for BNFL Inc. by Battelle in support of the Privatization Contract for treatment of Hanford underground storage tank wastes. The privatization work is part of the River Protection Project-Waste Treatment Plant (RPP-WTP). Under Part B-1 of the privatization effort, Battelle is conducting technology development and demonstration of various process flowsheet steps for BNFL with actual waste samples. Three candidate low-activity waste types have been identified for initial treatment: Envelope A, Envelope B, and Envelope C. Each of these represents compositional envelopes as defined in the privatization contract (Specification 7 of the TWRS privatization contract, http://www.hanford.gov/doe/contracts/de-ac06-96r13308/index.html). Before the liquid (supernatant) fraction of Envelope C wastes (e.g., Tank 241-AN-107 waste) can be disposed of as low-activity waste, pretreatment is required to remove transuranic (TRU) elements and radioactive strontium. Because of the high concentration of organic complexants in this waste, conventional separation processes (e.g., ion exchange) are not effective.

During Part A-1 of the privatization effort, the Savannah River Technical Center (SRTC) developed a Sr/TRU removal process involving isotopic dilution and precipitation with added strontium and iron (SRTC 1997a-d). While this treatment process provided the necessary supernatant decontamination, the resulting precipitate could not be filtered. Tests with waste simulants identified the iron precipitate as causing the difficulty with filtration (SRTC 2000). The search began for an alternative treatment process. Battelle proposed permanganate be examined as an alternative because it had been demonstrated to work with waste from Hanford Tank SY-101, which also contains high levels of organic complexants (Orth et al. 1995).

Orth et al. (1995) examined the removal of radioactive Sr and TRU from complexant-containing (citrate, glycolate, EDTA, HEDTA, and NTA) tank waste by the addition of metal cations and chemical oxidant. Permanganate was examined as a chemical oxidant to promote destruction/defunctionalization of the complexing agents and possible flocculation by the manganese solids. Permanganate was found to oxidize chromium first; then organic carbon; and last, nitrite. A sample of 3:1 diluted SY-101 waste was treated with 0.15M permanganate and decontamination factors (DFs)\(^{(6)}\) of >143 were obtained for Sr and 28.5 for Pu. Orth et al. recommended permanganate doses of 0.1M for treating complexant-containing wastes. For wastes such as in Tank SY-101, the chromium in the sludge consumes as much as half the permanganate. Waste in Tank AN-107 does not have the high chromium values in the sludge, so permanganate is expected to be effective at lower concentrations.

Permanganate is also used as a precursor to MnO\(_2\) and/or Mn(OH)\(_2\) coprecipitants via the “Method of Appearing Reagents” (Krot et al. 1996). This method requires that a chemical reductant such as formate or hydrdrazine be added to the waste to be treated. However, for Hanford wastes, it is not necessary because reductants are already present in the waste as organic complexing agents or their degradation products (e.g., formate). The resulting manganese solids are effective coprecipitants for Pu and other TRU elements, but generally not as effective as iron precipitates. Decontamination factors of >100 have been reported for various simulated waste streams.

The objective of the work reported here was to determine the potential use of permanganate for Sr/TRU removal and the optimal conditions for Sr/TRU decontamination. An archived sample of AN-107 waste was used because only a limited quantity of the actual AN-107 diluted feed was available. Tank waste simulants were not used for decontamination studies because the exact composition of organic complexants in the waste is not known, nor is the speciation of soluble Sr or TRU components. Proof-of-principle experiments were performed using approximately 20-mL samples of waste with

\(^{(6)}\) The decontamination factor is defined as the amount of the contaminant in the waste before treatment divided by the amount present after treatment.
various amounts of permanganate, strontium, and/or other metal ions. Supernatant decontamination data were obtained from the test data. The Sr and TRU DFs were compared to determine the efficiency of the Sr/TRU removal process. Preferred conditions were identified for larger-scale testing using 1-L samples.

The proof-of-principle experiments, three series of small-scale Sr/TRU decontamination tests, are described in this report. Test conditions and experimental procedures are described in Section 2.0. Experimental results from the three series of tests are described in Section 3.0. The major conclusion and recommendations that evolved from this work are given in Section 4.0. The appendices contain the test instructions, data sheets, logbook entries, analytical data, calculation, and staff role/responsibilities for this work.
2.0 Test Conditions and Experimental Procedures

2.1 Description of Archived AN-107 and AN-107 Diluted Feed Samples

Prior to its use for the BNFL project, the archived AN-107 material was diluted, decanted from the settled solids, and treated by ion exchange to remove cesium (Hendrickson 1997). It was collected as 45 grab samples in 125-mL bottles taken during January 1997. Approximately 5.4-L of tank waste were then transferred to Hanford’s 222-S Analytical Laboratory, and 0.53M sodium hydroxide was added to dilute the waste to 5M sodium and a free hydroxide concentration of 0.24M. The supernatant was not filtered prior to cesium ion exchange. Instead, the solids were allowed to settle, and the supernatant was decanted and sent through the crystalline silicotitanate loaded columns. Analysis of the waste after cesium removal indicated the free hydroxide was 0.126M. Following cesium removal the sample was transferred to Battelle in five 1-L plastic bottles where it has been stored in the Shielded Analytical Laboratory (SAL) hot cells in the Radiochemical Processing Laboratory (RPL).

In addition to the tests done with the archived AN-107 sample, three experiments were conducted with actual AN-107 diluted feed prepared specifically for the BNFL testing. The diluted feed was prepared and characterized at Battelle for integrated process testing (Urie et al. 1999). Waste samples retrieved from AN-107 were diluted and caustic adjusted to target concentrations of 7.7M sodium, and 1.0M free hydroxide for demonstration of the Sr/TRU removal process (Hallen et al. 2000b).

2.2 Development of Test Conditions

The Privatization Contract requires that the immobilized low-activity waste (ILAW) product contain less than 100 nCi/g TRU and less than 20 Ci/m³ Sr-90. Supernatant from Envelope C waste contains levels of Sr and TRU too high to meet ILAW requirements. For AN-107 waste, DFs of approximately 10 for Sr-90 (90% removal) and 5 for TRU (80% removal) are needed to meet the ILAW disposal requirements. Since over 90% of the TRU in AN-107 is Am-241, a target DF of 5 was established for Am-241.

Experimental conditions were defined using the results from the earlier studies by Orth et al. and limited studies conducted with AN-107 waste simulant at Battelle and SRTC (1999). Based on these studies a permanganate treatment level of 0.05M was expected to yield good decontamination results. Also results by Orth and others suggested that increased free hydroxide concentration, and/or addition of other metal salt precipitants (calcium, strontium, and europium) could improve decontamination of Sr-90 and TRU. This information was used to construct the first test matrix (see Section 3.1). The target concentrations listed in the test matrix are based on the final composition after addition of all reagents. The quantity of each reagent to add to the waste to achieve these values, as well as the actual quantities that were used, can be found in the test instructions included in Appendix A. Two controls were added for calculating the DFs, one unfiltered containing entrained solids and the other filtered to remove the entrained solids.

The results of the first series of experiments were used to define the conditions for the second series of experiments (see Section 3.2). The most significant observation was the lack of free hydroxide in the starting waste; consequently, for the second series of experiments, sodium hydroxide was added to the waste before the experiments were started. Also, permanganate alone did not give as high as expected DF for Sr-90, so non-radioactive strontium addition at different concentrations was examined for isotopic dilution. The completed test instruction and experimental details of the second series of experiments are included in Appendix B.
Adequate Sr-90 and TRU decontamination was obtained for many conditions in the second series of experiments. The third series of experiments was conducted to verify these results, further optimize reagent concentration, and examine the preferred reaction conditions with samples of actual AN-107 diluted feed (see Section 3.3). The completed test instruction and experimental details of the third series of experiments are included in Appendix C.

The heater temperature, hold time, and addition sequence was varied for each series of experiments. The first series of experiments was heated to 40°C and held there for 1 hour after addition of all reagents. This was considered adequate for permanganate treatment, based on results of Orth et al. (1995) that permanganate oxidation was complete in a matter of minutes at 30°C. However, this was a lower temperature and shorter time than the conditions identified for Sr/Fe addition from Part A-1 experiments. As a result, the second series of experiments was heated to 50°C and held for 4 hours after reagent addition. The additional time was expected to allow more isotopic exchange of Sr-90 with added nonradioactive Sr. For the third series of experiments, the first reagent was added and the waste heated to 50°C and held for 2 hours. The second reagent was then added, and the waste heated to 50°C and held for an additional 2 hours, again to allow for more isotopic Sr exchange.

2.3 Experimental

Battelle had archived approximately 5 L of AN-107 waste that had previously been treated by ion exchange for cesium removal (Henderson 1997). The waste was reported to be 5M sodium and 0.1M hydroxide. Experiments were designed on the assumption that the composition had not changed. However, after the first series of experiments, no free hydroxide was found by titration of waste samples. Continued organic aging of this waste during storage most likely consumed the free hydroxide. Later tests all included the addition of hydroxide to ensure the presence of free hydroxide during treatment/precipitation. The composition of the other components in the archived AN-107 is reported in the literature; the key components of the initial waste were determined for each series of experiments as a control, and were used to calculate DFs for the treated waste.

The small-scale experiments were conducted in the SAL hot cells with samples of approximately 20 mL of tank waste. The reagents were added to the wastes with an adjustable pipette, in the order listed in the test matrix. The reagents were rapidly added to the waste at room temperature and mixed by swirling of the vials with the remote manipulator hand. For the first two series of experiments, the addition of all reagents was completed before the samples were heated in a heat block that had been preheated to the set temperature. The samples were held for the prescribed time at this temperature, removed, cooled, centrifuged, and filtered for analyses. Stock solutions of the reagents were prepared for addition to the waste. The first round of experiments used 0.4M potassium permanganate as the stock solution, and later experiments used 1M sodium permanganate as the stock solution. The metal addition solutions were made up as the nitrate salt in 1M concentration. Sodium hydroxide was added as solid pellets or 10-19M solution. The actual quantities of waste and reagents used are given in the test instructions included as Appendix A, B, and C of this report.

2.4 Chemical Analyses

All of the chemical analyses were conducted at Battelle. BNFL designated the analytes of interest and minimum reportable quantity in a test specification or guidance letter (BNFL 1999a,b). Because the archived AN-107 sample had most of the radioactive cesium removed, Am-241 concentration could be determined directly by gamma energy analysis (GEA), along with the Eu isotopes 154 and 155. Relatively high levels of Cs-137 raise the gamma background level in the detector through Compton scattering, thereby making it difficult to detect other, lower-level gamma emitters, especially those having gamma energies below that of Cs-137. For the AN-107 diluted feed samples, separation and alpha energy analysis (AEA) were required for Am-241 because of the high Cs-137 concentration.
concentration was determined by chemical separation followed by beta counting. Sodium concentration was determined by inductively couple plasma-atomic emission spectrometry (ICP-AES), as well as the other metals listed in the test instructions. Selected samples were also analyzed by titration to determine the free hydroxide concentration. All of the analytical results are included in Appendix D.
3.0 Results and Discussion

Each series of experiments involved multiple samples and each was analyzed to determine the change in waste composition upon treatment. Samples of the initial waste were analyzed with and without filtration (0.45 um) to examine the contribution of entrained solids to the overall treatment. The radionuclide composition of the treated samples was compared with the initial composition to determine the extent of decontamination. The initial waste composition varied for each series of experiments. The Decontamination Factor (DF) for a specific radionuclide is defined as the concentration of the component in the initial waste divided by the concentration after treatment, corrected by the amount of dilution that occurred:

\[ DF = \frac{[A]_i}{([A]_f \times MD)} \]

where \([A]_i\) is the concentration of component A per mass in the initial sample, \([A]_f\) is the concentration of component A per mass in the treated sample, and MD is the mass dilution, final mass of treated solution divided by the initial mass of solution. The final mass is determined by summing up the mass of initial waste and all dilution, adjustments, and/or reagent additions.

3.1 The First Series of Proof-of-Principle Experiments

The first series of experiments focused on treatment with permanganate alone and in combination with added metal cations (Ca, Sr and Eu) using the waste as received (see Table 3.1). The archived AN-107 was expected to contain 5M Na and 0.12M free hydroxide. However, analyses of the starting material showed that there was no free hydroxide remaining in this waste. Where specified, sodium hydroxide was added as a 10M stock solution; metals cations were added as 1M nitrate solutions \([\text{Ca(NO}_3\text{)}_2, \text{Sr(NO}_3\text{)}_2, \text{and Eu(NO}_3\text{)}_3]\); and permanganate was added as 0.4M KMnO₄. The reagents were added in the order listed in Table 3.1. After addition of all reagents, the samples were heated to 40°C for 1 hour. The samples were removed from the heat block, cooled, and centrifuged. The supernatant was decanted from the centrifuged solids and filtered through a 0.45-um filter disk. Sample MN-01 was the initial waste and was not heated or filtered. Sample MN-02 was heated and filtered along with the other samples but no chemical reagents were added. Samples were acid digested and analyzed (ICP-AES, total alpha, GEA, and separation-beta counting for \(^{90}\text{Sr}\)). The centrifuged solids from one experiment, MN-03, were acid digested and analyzed (results reported as MN-12 in Appendix D).
Table 3.1. Test Matrix for the First Series of Permanganate Addition Experiments.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>NaOH Addition</th>
<th>Metal Addition</th>
<th>Target ([\text{MnO}_4^-])</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>MN-01</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>initial waste-unfiltered</td>
</tr>
<tr>
<td>MN-02</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>initial waste-filtered</td>
</tr>
<tr>
<td>MN-03</td>
<td>none</td>
<td>none</td>
<td>0.05 M</td>
<td>base case test condition</td>
</tr>
<tr>
<td>MN-04</td>
<td>none</td>
<td>none</td>
<td>0.05 M</td>
<td>duplicate (of MN-03)</td>
</tr>
<tr>
<td>MN-05</td>
<td>none</td>
<td>none</td>
<td>0.03M</td>
<td>low ([\text{MnO}_4^-])</td>
</tr>
<tr>
<td>MN-06</td>
<td>none</td>
<td>none</td>
<td>0.08 M</td>
<td>high ([\text{MnO}_4^-])</td>
</tr>
<tr>
<td>MN-07</td>
<td>to 1M</td>
<td>none</td>
<td>0.05 M</td>
<td>high ([\text{OH}^-])</td>
</tr>
<tr>
<td>MN-08</td>
<td>to 1M</td>
<td>none</td>
<td>0.08 M</td>
<td>high ([\text{MnO}_4^+]), high ([\text{OH}^-])</td>
</tr>
<tr>
<td>MN-09</td>
<td>none</td>
<td>to 0.05M Ca</td>
<td>0.05 M</td>
<td>metal addition</td>
</tr>
<tr>
<td>MN-10</td>
<td>none</td>
<td>to 0.05M Sr</td>
<td>0.05 M</td>
<td>metal addition</td>
</tr>
<tr>
<td>MN-11</td>
<td>none</td>
<td>to 0.05M Eu</td>
<td>0.05 M</td>
<td>metal addition</td>
</tr>
<tr>
<td>MN-12</td>
<td></td>
<td></td>
<td></td>
<td>centrifuged solids from MN-03</td>
</tr>
</tbody>
</table>

The effectiveness of permanganate treatment for TRU removal can be seen by examining the DFs for total alpha and Am-241 shown in Figure 3.1. The target DF of 5 was obtained for many of the samples. Decontamination was much higher with added hydroxide, MN-07 and MN-08, and added calcium, MN-09. This is expected because of the high carbonate and lack of any free hydroxide in the starting waste (bicarbonate/carbonate complexes of TRU). The addition of strontium with permanganate increased the DF slightly over permanganate alone. Increasing the permanganate level from 0.03M to 0.05M to 0.08M continued to increase the DF in a near linear relationship. The addition of Eu was evaluated to help increase the TRU DF. Unfortunately, because the \(\text{Eu(NO}_3\text{)}_3\) was acidic and the waste had no free hydroxide, the Eu addition just increased the conversion of carbonate to bicarbonate, decreasing the TRU DF compared with permanganate addition alone. Addition of free hydroxide had a greater effect on DF values, suggesting that 0.03M permanganate would be adequate for TRU decontamination with added free hydroxide.

Since over 90% of the TRU is Am-241, the good correlation between total alpha and Am-241 was expected. Decontamination factors for Eu-154 and Eu-155 are also shown in Figure 3.1 and varied similarly to Am-241 DFs, but were somewhat lower. Both Am and Eu were expected to be predominantly in the +3 oxidation state, and to have similar chemistries. No significant decontamination occurred for CO-60 or Cs-137.

Permanganate treatment of the waste removed iron from solution. Am-241 removal correlated with iron removal, as shown in Figure 3.2. Iron is known to be a good co-precipitant/flocculent for TRU, so the iron removal/precipitation may contribute to the high TRU removal. Data are also shown for MN-02, the filtered initial waste. This sample shows that approximately 10% of the Am-241 (and iron) removal was associated with filtration of the sample and removal of entrained solids. Manganese removal from solution showed a similar trend to Fe with the exception of MN-11, Eu addition. Because of the lack of free hydroxide, addition of the acidic Eu solution lowered the pH of the waste, and increased the soluble Mn.
The decontamination factors for Sr-90 are shown in Figure 3.3. Very little Sr decontamination occurred with permanganate addition, and little improvement occurred in going from 0.03M to 0.05M to 0.08M permanganate addition. The added free hydroxide only slightly improved the Sr DF. Calcium addition provided the highest Sr DF, but was still below the target DF of 10. Isotopic exchange with addition of nonradioactive strontium approximately doubled the Sr DF. This may be caused by the lower temperature and digest time used in these studies compared to the Part A-1 studies, which found Sr addition at 0.075M to be effective for decontamination of AN-107 (SRTC). Higher Sr DFs were expected for permanganate treatment based on the results from Orth et al. (1995). The much higher organic complexant concentration in AN-107 than SY-101 must be a significant factor in the low values. These tests indicate that different conditions for increased strontium decontamination are needed.

The ICP data in Appendix D for MN-09 and MN-10 indicate that the initial waste is below saturation in calcium and strontium. For MN-09, the calcium concentration increased from an initial value of 250 ug/g to 665 ug/g after addition of the Ca(NO₃)₂ and permanganate solutions. For MN-10, the total strontium concentration increased from an initial value of 1.2 ug/g to 125 ug/g after addition of the Sr(NO₃)₂ and permanganate solutions.

A comparison of MN-03 and MN-04, repeat experiments at the same conditions, shows that the procedure and analyses are reproducible; and the variability noted is caused by differences in reagent addition. This comparison is important since these experiments are difficult to conduct with actual waste in the hot cell; are conducted by remote manipulator; and reagent addition and stirring are difficult to repeat identically for every sample.

![Graph showing decontamination factors for different samples](image)

**Figure 3.1.** Decontamination Factors for Total Alpha, Am-241, Eu-154, and Eu-155.
3.2 The Second Series of Proof-of-Principle Experiments

Because the first series of experiments showed the importance of free hydroxide for TRU decontamination, the second series of experiments was conducted with archived AN-107 that was adjusted to a calculated free hydroxide concentration of 1 M. The second series of experiments, shown in Table 3.2, was designed to determine conditions for increased Sr DF. One test was added with permanganate at 0.16 M, twice the highest concentration from the first series. A series of tests were also added with various combinations of Sr and permanganate addition. Calcium addition at very low concentration was examined at or below the predicted solubility limit. The calcium addition was kept low because simulant tests had shown that calcium precipitate greatly reduced the filterability of the waste. Tests were added to determine the effect of higher sodium concentration and reduced carbonate.
A major change for all these experiments was the digest temperature, which was increased to 50°C, and the digest time, which was increased to 4 hours. These conditions were identified by SRTC for Sr/TRU removal with Sr and Fe addition, and were expected to increase the Sr decontamination by isotopic exchange/dilution and precipitation as SrCO₃.

The DFs for Am and Eu are presented in Figure 3.4. Results from duplicate experiments, PR-06 and PR-07, show good reproducibility. Samples PR-03 and PR-08 were sampled and analyzed twice; the duplicate results indicate good reproducibility of the analytical procedure. The differences between experiments were a result of the change in reagents and reaction conditions. The Am DFs were above the target value of 5 for all conditions except PR-08, high Sr and low permanganate test. The high concentration of Sr actually decreased the Am DF; compare PR-08 to PR-10. The large adverse impact of carbonate on Am (TRU) decontamination can be seen by comparing the results from PR-14 to PR-03, where the carbonate concentration had been reduced by approximately 50%. Results from this series of experiments confirm the requirement for free hydroxide to obtain adequate Am DF. This is likely related to the shift in carbonate/bicarbonate equilibrium caused by increasing the free hydroxide concentration. The small amount of Ca addition increased the Am DF, but the Am DFs were well above the target of 5 without the addition of Ca.

Table 3.2. Test Matrix for the Second Series of Permanganate Addition Experiments.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Other Addition</th>
<th>Target [Sr]</th>
<th>Target [MnO₄⁻]</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>PR-01</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>initial waste-unfiltered</td>
</tr>
<tr>
<td>PR-02</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>initial waste-filtered</td>
</tr>
<tr>
<td>PR-03</td>
<td>none</td>
<td>none</td>
<td>0.05 M</td>
<td>repeat of MN-7</td>
</tr>
<tr>
<td>PR-04</td>
<td>none</td>
<td>none</td>
<td>0.08 M</td>
<td>repeat of MN-8</td>
</tr>
<tr>
<td>PR-05</td>
<td>none</td>
<td>none</td>
<td>0.16 M</td>
<td>2X [MnO₄⁻]</td>
</tr>
<tr>
<td>PR-06</td>
<td>none</td>
<td>0.075 M</td>
<td>0.05 M</td>
<td>base case</td>
</tr>
<tr>
<td>PR-07</td>
<td>none</td>
<td>0.075 M</td>
<td>0.05 M</td>
<td>duplicate of base case</td>
</tr>
<tr>
<td>PR-08</td>
<td>none</td>
<td>0.075 M</td>
<td>0.03 M</td>
<td>low [MnO₄⁻]</td>
</tr>
<tr>
<td>PR-09</td>
<td>none</td>
<td>0.05 M</td>
<td>0.05 M</td>
<td>low [Sr]</td>
</tr>
<tr>
<td>PR-10</td>
<td>none</td>
<td>0.05 M</td>
<td>0.03 M</td>
<td>low [MnO₄⁻] and [Sr]</td>
</tr>
<tr>
<td>PR-11</td>
<td>0.01 M Ca</td>
<td>0.05 M</td>
<td>0.03 M</td>
<td>Ca effect</td>
</tr>
<tr>
<td>PR-12</td>
<td>0.01 M Ca</td>
<td>0.075 M</td>
<td>0.05 M</td>
<td>Ca effect</td>
</tr>
<tr>
<td>PR-13</td>
<td>1.5 M Na*</td>
<td>0.075 M</td>
<td>0.05 M</td>
<td>Na effect</td>
</tr>
<tr>
<td>PR-14</td>
<td>1 M H**</td>
<td>none</td>
<td>0.05 M</td>
<td>CO₃⁻ effect</td>
</tr>
<tr>
<td>PR-15</td>
<td></td>
<td></td>
<td></td>
<td>centrifuged solids from PR-06</td>
</tr>
</tbody>
</table>

* Sodium nitrate was added to increase sodium concentration.
** The initial waste was added to concentrated nitric acid to neutralize and reduce the carbonate concentration in half.
The Sr-90 decontamination factors are presented in Figure 3.5. These results confirm that permanganate alone, even at a concentration as high as 0.16 M, will not provide adequate Sr decontamination. The changes made for the combined Sr and permanganate addition increased the Sr DF above the target of 10. The Sr DFs appear to be much more sensitive to reaction conditions; note the variability between PR-06 and PR-07, which are results for the same treatment conditions. Similar Sr-90 DFs results for PR-06, 07, 10, 11, 12, and 13 suggest that the Sr and permanganate mole ratio needs to be between 1.5 and 1.7 Sr/Mn, that lower concentration of reagents are effective, and Ca addition has no benefit. Decreasing the carbonate in the waste did not significantly improve the Sr DF; compare PR-03 to PR-14.

Figure 3.4. Decontamination Factors for Americium (241) and Europium (155).

Figure 3.5. Strontium-90 decontamination factors for the second series of experiments.
In contrast to the Fe precipitation for TRU removal, permanganate treatment consumed very little of the added free hydroxide. Whereas iron consumed 3 mole of hydroxide per mole of added iron, permanganate consumed less than 1. Examining the titration data shows that Sr addition reduced the carbonate on a basis of 1 mole of carbonate per mole of added Sr, i.e., precipitation of SrCO₃.

### 3.3 The Third Series of Proof-of-Principle Experiments

The third series of experiments used samples of both archived AN-107 and AN-107 diluted feed. These tests, shown in Table 3.3, were conducted as a final confirmation of reaction conditions prior to conducting large-scale precipitation tests. The Sr and permanganate additions were at the lower concentration, and one experiment, OP-02, was a repeat of the earlier concentrations used PR-10. Reversing the order of addition, permanganate first followed by Sr, was examined (OP-05), as well as reduced free hydroxide concentration (LH-07). Because the change in reaction conditions was so successful for the second series, BNFL requested the conditions be changed for these tests also. After each chemical addition was completed, the samples were heated to 50°C and held for 2 hours. Before the second reagent was added, the samples were removed from the heater block and allowed to cool to make weighing easier (less balance drift). Then the second reagent was added, and the vials returned to the heating block for an additional 2 hours digestion at 50°C.

### Table 3.3. Test Matrix for the Third Series of Permanganate Addition Experiments.

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>AN-107 Waste ID</th>
<th>Target [Sr]</th>
<th>Target [MnO₄⁻]</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>OP-01</td>
<td>Archived, 1M OH + 1.5M Na</td>
<td>none</td>
<td>none</td>
<td>initial waste-filtered</td>
</tr>
<tr>
<td>OP-02</td>
<td>Archived, 1M OH</td>
<td>0.05</td>
<td>0.03</td>
<td>Repeat of PR-10</td>
</tr>
<tr>
<td>OP-03</td>
<td>Archived, 1M OH + 1.5M Na</td>
<td>0.05</td>
<td>0.03</td>
<td>low [Sr] and [MnO₄⁻]</td>
</tr>
<tr>
<td>OP-04</td>
<td>Archived, 1M OH + 1.5M Na</td>
<td>0.05</td>
<td>0.03</td>
<td>Duplicate of OP-03</td>
</tr>
<tr>
<td>OP-05</td>
<td>Archived, 1M OH + 1.5M Na</td>
<td>0.05</td>
<td>0.03</td>
<td>Reverse addition order</td>
</tr>
<tr>
<td>LH-06</td>
<td>Archived, 0.5M OH + 1.5M Na</td>
<td>none</td>
<td>none</td>
<td>initial waste-filtered</td>
</tr>
<tr>
<td>LH-07</td>
<td>Archived, 0.5M OH + 1.5M Na</td>
<td>0.05</td>
<td>0.03</td>
<td>Low hydroxide</td>
</tr>
<tr>
<td>RW-08</td>
<td>Diluted feed*</td>
<td>none</td>
<td>none</td>
<td>initial waste-filtered</td>
</tr>
<tr>
<td>RW-09</td>
<td>Diluted feed*</td>
<td>0.05</td>
<td>0.03</td>
<td>low [Sr] and [MnO₄⁻]</td>
</tr>
<tr>
<td>RW-10</td>
<td>Diluted feed*</td>
<td>0.05</td>
<td>0.03</td>
<td>Duplicate of RW-09</td>
</tr>
<tr>
<td>RW-11</td>
<td>Diluted feed*</td>
<td>0.075</td>
<td>0.05</td>
<td>High [Sr] and [MnO₄⁻]</td>
</tr>
</tbody>
</table>

*See Urie et al. (1999), 7.5M sodium and 0.71M free hydroxide.*

The Am-241 DFs are shown in Figure 3.6 for the third series of experiments. The DFs exceeded the target value of 5 for all of the archived AN-107 experiments. However, the DFs for Am-241 in the low concentration tests with AN-107 diluted feed, RW-09 and RW-10, were below 5. The AN-107 diluted feed is much more concentrated than the archived AN-107 which had been treated by ion exchange, and the higher reagent addition was necessary to obtain adequate Am decontamination. Examining the results from the experiments with archived AN-107, reversing the order of reagent addition, permanganate then Sr, appears to reduce the Am-241 decontamination. Reducing the free hydroxide addition from 1M to 0.5M had no impact on Am decontamination.

The strontium-90 DFs are shown in Figure 3.7. The Sr-90 DFs were all below the target values of 10. The lower DFs appear to be a result of the change in treatment conditions used for these experiments since OP-02, a repeat of the reagent concentrations used in experiment PR-10, had a lower DF. The reversed reagent addition order, OP-05, improved the Sr-90 value for these reaction conditions.
The decreased addition of free hydroxide from 1M to 0.5M had no effect on the Sr-90 DFs. The Sr-90 DFs for the AN-107 diluted feed were less than for the archived AN-107 samples, as was the case for Am-241 DFs. Sr-90 decontamination appears to be extremely sensitive to reaction conditions, and adequate Sr-90 decontamination is more difficult to obtain than Am-241 (TRU) decontamination.

**Figure 3.6.** Americium-241 Decontamination Factors for the Third Series of Experiments.

**Figure 3.7.** Strontium-90 Decontamination Factors for the Third Series of Experiments.

The AN-107 diluted feed samples were analyzed for Am-241 by separation followed by AEA. This analysis method also detects curium isotopes 242 and 243+244. The DFs for Cm were calculated and found to be equal to those obtained for Am-241.
4.0 Conclusion and Recommendations

The results of the proof-of-principle experiments showed that free hydroxide was needed for high TRU (Am-241) removal using permanganate. Similar results were obtained for calculated free hydroxide levels of 0.5 and 1.0M. Addition of permanganate alone could not achieve adequate Sr-90 decontamination, and isotopic dilution/precipitation with added nonradioactive strontium was required. Adequate levels of Sr-90 decontamination were more difficult to attain and more sensitive to treatment conditions. The highest strontium decontamination was obtained when reagents were added to the waste at ambient temperature in the order strontium then permanganate. Adequate Sr/TRU removal was obtained with addition of 0.05M strontium and 0.03M permanganate to samples of archived AN-107. However, because the AN-107 diluted feed is more concentrated, large-scale Sr/TRU removal tests should be conducted with 0.075M strontium and 0.05M permanganate.

The addition of low concentrations of calcium increased the Am DF, but had no impact on the Sr-90 decontamination. Since calcium was found to decrease the filterability of waste simulants, it is recommended that no calcium addition be used for the large-scale tests.

Additional tests should be run with AN-107 diluted feed on a larger scale with reduced reagent addition; free hydroxide 0.5M, Sr = 0.05M and permanganate = 0.03M. These tests should examine in detail the effects of temperature and digest time. Since SrCO$_3$ has retrograde solubility (decreases with increasing temperature), the digest temperature of 50°C may reduce the Sr-90 isotopic exchange because of the lower Sr solubility. The permanganate reaction is also rapid, complete in a manner of minutes, so the 4-hour digest time may not be required.
5.0 References


5.1
Appendix A: Test Instruction TI-037 and Data Sheets
**Title:** Sr/TRU Removal from AN-107, Permanganate Addition Scoping Studies  

**Work Location:** RPL SFO HLRF  

**Author:** SA Bryan  

**Use Category Identification:** Reference  

**Effective Date:** New  

**Supersedes Date:** New  

**Identified Hazards:**
- [x] Radiological  
- [ ] Hazardous Materials  
- [ ] Physical Hazards  
- [ ] Hazardous Environment  
- [ ] Other:  

**Required Reviewers:**
- [x] Author  
- [x] Technical Reviewer  
- [ ] RPL Manager  
- [ ] Project Manager  
- [ ] RPG Quality Engineer  
- [ ] BNFL (not required for scoping studies)  

**Are One-Time Modifications Allowed to this Procedure?**  
- [x] Yes  
- [ ] No  

**NOTE:** If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.  

**On-The Job Training Required?**  
- [ ] Yes  
- [x] No  

**FOR REVISIONS:**
- [ ] Is retraining to this procedure required?  
  - [ ] Yes  
  - [x] No  

- [ ] Does the OJT package associated with this procedure require revision to reflect procedure changes?  
  - [ ] Yes  
  - [ ] No  
  - [x] N/A  

**Approval**

<table>
<thead>
<tr>
<th>Role</th>
<th>Signature</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Author</td>
<td>Samuel A. Bryan</td>
<td>4/20/99</td>
</tr>
<tr>
<td>SAL-RPL Representative</td>
<td>Rick T. Steele</td>
<td>4/26/99</td>
</tr>
</tbody>
</table>

**Controlled Document**
Applicability

This test instruction describes the procedure to be used for scoping studies to determining the efficiency of the Sr/TRU removal (decontamination factor) by permanganate addition. The work described herein will be performed in the Shielded Analytical Laboratory (SAL) hot cells located in the Radiochemical Processing Laboratory (RPL). This test instruction supports the Test Plan No. 29953-013.

Work is to be performed by hot cell technicians under the supervision of a cognizant scientist. The cognizant scientist shall be responsible for implementation and adherence to this test instruction. This instruction is specific to:

- Sr/TRU removal by permanganate addition to envelope C waste during part 1B of the privatization; AN-107 will be used as a representative envelope C sample,
- permanganate addition and precipitation of actual wastes in RPL hot cell facilities, and
- tests performed at Battelle in the RPL, by staff in the Environmental Technology Division.

DRD Reference: none

Schedule Reference: Additional work scope not in baseline.

Justification

This activity is a scoping study using actual AN-107 waste to evaluate the potential of permanganate addition for Sr/TRU removal (decontamination). The preferred method by BNFL, strontium and iron precipitation, has not provided the necessary performance based upon process design criteria.

Objective

Because of the recent problem with filtration of the iron precipitates from AN-107 simulant, a series of scoping experiments are needed to determine the ability of permanganate to obtain the necessary DF for Sr and TRU (primarily Am, ~92% of TRU on an activity basis). Permanganate has been examined as both an oxidant (decomplexing waste, solubilizing chromium, and oxidation of technetium species to pertechnetate) and a precursor to MnO₂ and/or Mn(OH)₂ coprecipitants via the “Method of Appearing Reagents,” Krot et.al. Permanganate was found to preferentially oxidize chromium, followed by organic carbon, and lastly nitrite. For wastes such as SY-101, the chromium in the sludge consumes as much as half the permanganate.

Success Criteria

The BNFL flowsheet for AN-107 (Envelope C) requires the separation of the HLW Sr/TRU from the LLW supernate prior to incorporation into glass. BNFL in Phase 1A identified precipitation by strontium and iron addition as the preferred method for decontamination. Decontamination factors are needed to reduce Sr and TRU (primarily Am) to the low level limits. Sr and TRU decontamination factors of 5 or more are needed while minimizing the addition of chemical reagents.

These tests will generate results that can be used for comparison with Envelope C Criteria for Sr/TRU decontamination (DF 5 or more).
Background

Orth et al (1995) recommended permanganate doses of 0.1M for decomplexing SY-101 type wastes where high concentrations of chromium (approximately 0.4%) partly consumed permanganate prior to organic destruction. AN-107 does not have the high chromium values in the sludge compared to SY-101. Due to lower chromium values in AN-107, permanganate is expected to be effective at lower doses, with 0.05M a target treatment level. Reducing the amount of permanganate added will reduce the amount of HLW waste glass produced.

Spill Protection/Response

Hot cell technicians shall conduct tests in a manner to minimize the impact of a spill. In the event of a spill, the cognizant scientist shall be notified and a decision will be made to try to recover the sample or repeat the test condition.

Feed Description

The Sr/TRU removal scoping tests will use AN-107 supernatant previously treated for cesium removal (Envelope C). This feed was diluted and chemicals added such that the approximate final concentrations are: 4.9M sodium and 0.2M hydroxide. Approximately 2 L of diluted AN-107 supernatant is available for these scoping tests, of this approximately 200 mL will be needed.

Equipment Description

The permanganate addition tests will be conducted on a small scale, approximately 20 mL each. Appropriate glass vials or test tubes small be used, such that the samples can be heated to a temperature of 40°C. Some mixing/stirring of the samples after chemical addition should be provided, this may be as simple as swirling the samples. Reagents will be added slowly as liquids, and stirred/mixed/swirled after each reagent is added. Some experiments will require two different reagent to be added in the proper sequence as detailed in the test matrix.

Work Instructions

1.0 Applicability

This test instruction is to be used to perform scoping tests for Sr/TRU removal by permanganate addition. Approximately four (4) 1-L bottles of AN-107 supernatant previously treated by Cs ion exchange is available in SAL for these scoping tests. One bottle of the AN-107 waste will be used for these tests.

2.0 Supporting Documents

This test instruction is not a stand-alone document. Sr/TRU Precipitation and analytical requirements for all BNFL related work are contained in PNNL Test Plan No. BNFL-TP-29953-013. TP-29953-013 also contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation.

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Manager – Rich Hallen
- SFO Manager – Randy Thornhill (Rick Steele)
- Test Scientists – Sam Bryan, Rich Hallen
• Hot Cell Technician – Vaughn Hoops
• Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required
1. Twelve 20 mL glass scintillation vials for filtered, analytical samples, pre-labeled as follows: MN-01 through MN-12. And 11 vials or test tubes for conducting experiments.
2. A 1 liter polyethylene bottle or equivalent for waste.
3. 12 - disposable syringes and 0.45 micron syringe filters.
4. 9 - vessels (vials/test tubes) to conduct tests
5. AN-107 waste in SAL-cell. Use archived sample from "Tc Removal Flow Studies" (Project No. 25865). Archive sample # C3E3
6. heating device
7. Volume dispensing or measuring device, such as a graduated tube or cylinder, for determining the density of the AN-107 sample

4.2 Equipment
1. 100 gram balance
2. Hand held camera (if convenient)
3. Stop-watch
4. Calculator
5. Hot plate
6. Thermometer

4.3 Reagents Needed (see prep sheet)
1. 10 mL of 10M NaOH
2. 10 mL of 1M Ca(NO₃)₂, Sr(NO₃)₂ and Eu(NO₃)₃
3. 50 mL of 0.4M KMnO₄
4. Archive AN-107 sample # C3E3.

4.4 Other Supplies
1. BNFL-TI-29953-037 (this test instruction)
2. Laboratory Record Book (use Red Bound, BNFL lab notebook, BNW-13733)
3. BNFL-TP-29953-013 (Hallen 1999)

5.0 Test Instructions

The laboratory record book (LRB) shall be used to record other testing information as required by this test instruction and all test conditions not stated by this test instruction.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as practical. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying.
5.1 Prestart

5.1.1 Prepare solutions according to the attached preparation sheet.

5.1.2 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

Record Unique ID # of reagents:

- 0.4M KMnO₄
- 10N NaOH Manufacture
- 1M Ca(NO₃)₂
- 1M Sr(NO₃)₂
- 1M Eu(NO₃)₃

5.1.3 Initial and date when each item is completed.

Review the test matrix (Table 1) in the test instructions in BNFL-TI-29953-037. Note the calculation worksheet, which gives quantities of reagents to add. Reagents can be added as volume but always record the mass added.

5.1.4 Obtain the following information:

M&TE List:

- Balance 1: 360 060 010 1C / Metler AE 160
  - Calib ID
  - Calib Exp Date: 8/99
  - Location: cell 2 / SAL

- temperature reading device (thermometer or thermcouple/reader):
  - Calib ID: 065719-SC
  - Calib Exp Date: 2/21/00
  - Location: cell 1 / SAL, cell 2 / SAL (thermometer)

5.2 Operation

5.2.1 Determine the density of the AN-107 sample and record here data and results here:

mass ; volume ; density

Review the test matrix shown below, Table 1. Record data in Table 2. Note to check each activity when complete. This should be done and verified by the cognizant scientist.
Table 1. Test Matrix.

<table>
<thead>
<tr>
<th>Check when complete</th>
<th>Test #</th>
<th>Target [KMnO₄]</th>
<th>Added NaOH</th>
<th>Metal Addition</th>
<th>Comment</th>
<th>Scientist Verification</th>
</tr>
</thead>
<tbody>
<tr>
<td>✓</td>
<td>1</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>control-unfiltered</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>2</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>control-filtered</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>3</td>
<td>0.05 M</td>
<td>none</td>
<td>none</td>
<td>base case test condition</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>4</td>
<td>0.05 M</td>
<td>none</td>
<td>none</td>
<td>duplicate (of # 3)</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>5</td>
<td>0.03 M</td>
<td>none</td>
<td>none</td>
<td>low [KMnO₄]</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>6</td>
<td>0.08 M</td>
<td>none</td>
<td>none</td>
<td>high [KMnO₄]</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>7</td>
<td>0.05 M</td>
<td>to 1M</td>
<td>none</td>
<td>high [OH⁻]</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>8</td>
<td>0.08 M</td>
<td>to 1M</td>
<td>none</td>
<td>high [KMnO₄], high [OH⁻]</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>9</td>
<td>0.05 M</td>
<td>none</td>
<td>to 0.05M Ca</td>
<td>metal addition</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>10</td>
<td>0.05 M</td>
<td>none</td>
<td>to 0.05M Sr</td>
<td>metal addition</td>
<td>SAB</td>
</tr>
<tr>
<td>✓</td>
<td>11</td>
<td>0.05 M</td>
<td>none</td>
<td>to 0.05M Eu</td>
<td>metal addition</td>
<td>SAB</td>
</tr>
</tbody>
</table>

It is preferable that the permanganate treated solution be heated to 40°C and held at this temperature for 1 hour. The permanganate oxidation reaction generates some heat but at these low volume additions, the sample is expected to reach about 30°C without external heating.

Table 2. Data Sheet.

<table>
<thead>
<tr>
<th>Vial ID</th>
<th>Weight, g</th>
<th>Tare Vial</th>
<th>Test Complete (initial)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MN-01</td>
<td>2.249081</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-02</td>
<td>2.48818</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-03</td>
<td>2.47818</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-04</td>
<td>2.50721</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-05</td>
<td>2.49138</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-06</td>
<td>2.49795</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-07</td>
<td>2.48674</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-08</td>
<td>2.45786</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-09</td>
<td>2.49763</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-10</td>
<td>2.48933</td>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>MN-11</td>
<td>2.48780</td>
<td>20</td>
<td>✓</td>
</tr>
</tbody>
</table>

5.2.2 Record the weights of all vials, samples, additions, etc. After the reaction time of 1 hour at 40°C, the samples can be centrifuged to allow easier filtration prior to analytical analysis. If possible, record the volume of centrifuged solids. For the base case, (test 1, 0.05M MnO₄⁻), the centrifuged solids may be submitted for solids analyses. Decant the supernate from this sample,
record the weight of solids/solution remaining; the solids will be submitted as is for acid
digestion/analysis (see Table 3 below). Note that the AN-107 original “as is - without filtering”
volume needs to be submitted for analyses and acid digest because it contains some solids; the “as
received - filtrate” will be the same “as received” waste but treated as the other samples, and is a
control sample (centrifuged then filtered the same manner as the other samples, syringe filter).

6.0 Sample Analysis

The point of contact for the sample analysis from these tests is Mike Urie and Rick Steele.

6.1 Chemical and Radiochemical Analysis

Table 3 below shows the sample analysis list. The table lists the analyses to be performed on
samples generated from this test instruction.

Table 3. Samples and Their Required Analyses

<table>
<thead>
<tr>
<th>Process Variable</th>
<th>Vial ID</th>
<th>ACL No.(^{(a)})</th>
<th>Sample Type</th>
<th>Sample Preparation</th>
<th>Analysis Description(^{(b)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>AN-107 as is</td>
<td>MN-01</td>
<td>99-01595</td>
<td>some solids</td>
<td>acid digest</td>
<td>Sr/Am/Total alpha, ICP</td>
</tr>
<tr>
<td>AN-107 as is</td>
<td>MN-02</td>
<td>99-01596</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha, ICP, [OH⁻]</td>
</tr>
<tr>
<td>0.05M Permanganate</td>
<td>MN-03</td>
<td>99-01597</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha, ICP, [OH⁻]</td>
</tr>
<tr>
<td>0.05M Permanganate(dup)</td>
<td>MN-04</td>
<td>99-01598</td>
<td>Filtrate</td>
<td>acid digest</td>
<td>Sr/Am/Total alpha, ICP</td>
</tr>
<tr>
<td>0.03 Permanganate</td>
<td>MN-05</td>
<td>99-01599</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>0.08 Permanganate</td>
<td>MN-06</td>
<td>99-01600</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>1M Hydroxide, 0.05M Permanganate</td>
<td>MN-07</td>
<td>99-01601</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>1M Hydroxide, 0.08M Permanganate</td>
<td>MN-08</td>
<td>99-01602</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>Ca Cation Addition</td>
<td>MN-09</td>
<td>99-01603</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>Sr Cation Addition</td>
<td>MN-10</td>
<td>99-01604</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>Eu Cation Addition</td>
<td>MN-11</td>
<td>99-01605</td>
<td>Filtrate</td>
<td>0.45 µm dead end</td>
<td>Sr/Am/Total alpha</td>
</tr>
<tr>
<td>0.05M Permanganate</td>
<td>MN-12</td>
<td>99-01606</td>
<td>Centrifuge Solutions</td>
<td>acid digest</td>
<td>Sr/Am/Total alpha, ICP</td>
</tr>
</tbody>
</table>

(a) Analytical Chemistry Laboratory (ACL) tracking number is a unique identification for these samples.
The samples are submitted for analysis under the Analytical Service Request (ASR) number 5345.
(b) Descriptions of analyses are contained in Table 4.
### Table 4. Description of Analyses

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Analysis Method</th>
<th>PNNL Procedure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Americium-241, Eu isotopes</td>
<td>GEA</td>
<td>PNL-ALO-450</td>
</tr>
<tr>
<td>Strontium-90 (Yttrium-90)</td>
<td>Separations and Beta Counting</td>
<td>PNL-ALO=476/431</td>
</tr>
<tr>
<td>Total Alpha</td>
<td>Gross Alpha</td>
<td>PNL-ALO-420/421</td>
</tr>
<tr>
<td>Hydroxide</td>
<td>EPA SW-846 Modified Method, 310(3)</td>
<td>PNL-ALO-228</td>
</tr>
<tr>
<td>Metal Ions (see list Table 5)</td>
<td>ICP-AES</td>
<td>PNL-ALO-211/280</td>
</tr>
</tbody>
</table>

### Table 5. Analytical Requirements for Supernate/Filtrate and Centrifuged Solids

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Centrifuged Solids Minimum Reportable Quantity microCi/gm</th>
<th>Supernate/Filtrate Minimum Reportable Quantity microCi/ml</th>
<th>Analysis Method</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>microgm/gm</td>
<td>microgm/ml</td>
<td></td>
</tr>
<tr>
<td>Strontium-90</td>
<td>7.01E+01</td>
<td>1.5E-01</td>
<td>Chemical Separation &amp; Beta Count</td>
</tr>
<tr>
<td>Americium-241</td>
<td>1.2E-03</td>
<td>7.2E-04</td>
<td>GEA</td>
</tr>
<tr>
<td>Total Alpha</td>
<td>1.0E-03</td>
<td>2.3E-01</td>
<td>Total Alpha</td>
</tr>
<tr>
<td>Al</td>
<td>3.3E+02</td>
<td>7.5E+01</td>
<td></td>
</tr>
<tr>
<td>Ba</td>
<td>6.0E+02</td>
<td>7.8E+01</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>1.8E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Cd</td>
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<td></td>
</tr>
<tr>
<td>Co</td>
<td>3.0E+00</td>
<td>3.0E+01</td>
<td></td>
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<tr>
<td>Cr</td>
<td>1.2E+02</td>
<td>1.5E+01</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>1.8E+01</td>
<td>1.7E+01</td>
<td></td>
</tr>
<tr>
<td>Eu</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>1.4E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>K</td>
<td>1.5E+03</td>
<td>2.0E+02</td>
<td></td>
</tr>
<tr>
<td>La</td>
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<td></td>
</tr>
<tr>
<td>Mg</td>
<td>5.4E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Mn</td>
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</tr>
<tr>
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<td>Ni</td>
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</tr>
<tr>
<td>Pb</td>
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<td>3.0E+02</td>
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</tr>
<tr>
<td>Si</td>
<td>3.0E+03</td>
<td>1.7E+02</td>
<td></td>
</tr>
<tr>
<td>Sr</td>
<td>NA</td>
<td>NA</td>
<td>Acid Digestion followed by ICP-AES</td>
</tr>
<tr>
<td>Ti</td>
<td>1.5E+02</td>
<td>1.7E+01</td>
<td></td>
</tr>
<tr>
<td>U</td>
<td>6.0E+02</td>
<td>6.0E+02</td>
<td></td>
</tr>
<tr>
<td>Zn</td>
<td>6.0E+00</td>
<td>1.6E+01</td>
<td></td>
</tr>
<tr>
<td>OH-</td>
<td>0.05M</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
### 7.0 Calculation and Important Information

Density of AN-107 sample that has cesium removed = estimated 1.22 g/mL

Density of 0.4M KMnO₄ solution = 1.0361 g/mL (measured from bench sheet)

Density of 10M NaOH solution = 1.33 g/mL (CRC)

Density of 1M Ca(NO₃)₂ solution = 1.16 g/mL (measured from bench sheet)

Density of 1M Sr(NO₃)₂ solution = 1.162 g/mL (measured from bench sheet)

Density of 1M Eu(NO₃)₃ solution = 1.277 g/mL (measured from bench sheet)

20 mL = 24.4 grams of AN-107 waste

Mass of Solutions based on above density data. Densities (and masses) need to be verified based on actual solution densities. This will be performed after solutions are prepared in Step 4.3.

<table>
<thead>
<tr>
<th>test #</th>
<th>test Condition</th>
<th>ADD in mL</th>
<th>Ca</th>
<th>Sr</th>
<th>Eu</th>
<th>KMnO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.05 M</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>1.818182</td>
</tr>
<tr>
<td>2</td>
<td>0.05 M</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>1.818182</td>
</tr>
<tr>
<td>3</td>
<td>0.03 M</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>1.052632</td>
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<tr>
<td>4</td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>3.076923</td>
</tr>
<tr>
<td>5</td>
<td>0.05 M</td>
<td>2.222222</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>1.818182</td>
</tr>
<tr>
<td>6</td>
<td>0.08 M</td>
<td>2.222222</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>3.076923</td>
</tr>
<tr>
<td>7</td>
<td>0.05 M</td>
<td>0</td>
<td>1.052632</td>
<td>0</td>
<td>1.818182</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.05 M</td>
<td>0</td>
<td>0</td>
<td>1.052632</td>
<td>0</td>
<td>1.818182</td>
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<tr>
<td>9</td>
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<td>0</td>
<td>0</td>
<td>1.052632</td>
<td>1.818182</td>
<td></td>
</tr>
</tbody>
</table>

Initial waste mass = 24.4 grams
8.0 References


Metal Additions: Prep of Stock Solutions

Ca, Sr, or Eu need to be added to the waste for Sr/TRU decontamination. These solutions will be added before the permanganate.

Record all information and observations on prep sheet or in lab notebook.

Balance number: 360-06-11
Balance calibration: 8.9

Makeup Ca(NO₃)₂ solution

1 M

use Ca(NO₃)₂·4H₂O

236.15 grams/mole (FW) (lot #7603B H )

Tare 25 mL volumetric flask 52.0877 grams

add 5.90375 grams Ca(NO₃)₂·4H₂O flask + Ca(NO₃)₂ 27.993 grams

actual weight of Ca(NO₃)₂ added 5.9058 grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 50.0618 grams

calculate actual [Ca] = actual weight/236.15/volume in Liters = 1.003 M

calculate density of solution, weight of solution/volume = 1.1164 grams/mL

Transfer stock solution to bottle and label unique ID # Ca(NO₃)₂.

Makeup Sr(NO₃)₂ solution

1 M

use Sr(NO₃)₂

211.63 grams/mole (FW) (lot #939B F)

Tare 10 mL volumetric flask 16.8460 grams

add 5.29975 grams Sr(NO₃)₂ flask + Sr(NO₃)₂ 13.969 grams

actual weight of Sr(NO₃)₂ added 2.1149 grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 23.4676 grams

calculate actual [Sr] = actual weight/211.63/volume in Liters = 0.993 M

calculate density of solution, weight of solution/volume = 1.1622 grams/mL

Transfer stock solution to bottle and label unique ID # Sr(NO₃)₂.

Makeup Eu(NO₃)₃ solution

1 M

use Eu(NO₃)₃·6H₂O

446.07 grams/mole (FW) (lot #0331 F Y)

Tare 5 mL volumetric flask 15.0322 grams

add 11.5175 grams Eu(NO₃)₃·6H₂O flask + Eu(NO₃)₃·6H₂O 17.3411 grams

actual weight of Eu(NO₃)₃·6H₂O add 2.2439 grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
fill to volumetric line with Milli-Q water. Record total weight: 21.4943 grams

calculate actual [Eu] = actual weight/446.07/volume in Liters = 1.014 M

calculate density of solution, weight of solution/volume = 1.2724 grams/mL

Transfer stock solution to bottle and label unique ID # Eu(NO₃)₃.

Date prepared: 4-19-99
Prepared by: [Signature]
Work Package Number: W5-130e

Calculations prepared by RT Hallen
Calculations checked by: 4/16/99
Preparation of Stock Permanganate Solution

Permanganate needs to be added to the waste for Sr/TRU decontamination. This solution will be added after any other reagents, if needed.

Record all information and observations on prep sheet or in lab notebook.

Balance number: 360-06-01-040
Balance calibration: 8-99-8-99

Makeup stock KMnO₄ solution 0.4 M
use KMnO₄ 99+, ACS Reagent Grade
158.04 grams/mole (FW) (lot #3616 )

Tare 50 mL volumetric flask 31.9342 grams
add 3.1608 grams KMnO₄ flask + KMnO₄ 34.9856 grams
actual weight of KMnO₄ added 34.9856 grams
add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
add approximately .5 mL of 1N NaOH or 0.05mL of 10N NaOH to stabilize permanganate
all will probably not dissolve because 0.4M is near saturation, add more water, repeat
fill to volumetric line with Milli-Q water. Record total weight: 83.6305 grams
calculate actual [MnO₄] = actual weight/158.04/volume in Liters = 0.4001 M
calculate density of solution, weight of solution/volume = 1.0361 grams/ml

Transfer stock solution to bottle and label unique ID # K MnO₄

Date prepared: 4/19-99
Prepared by:
Work Package Number: W5130C

Make up 30% NaOH (w/w) (w/νN)
in poly bottle add 30gram NaOH pellet 29.9858 grams added
then add 70grams water 70.0622 grams total weight = 160.3336

calculate 29.97% NaOH (w/w)

Calculation Prepared by RT Hallen
Calculation checked by BNFL Permanganate Scoping Studies 4/16/99
Greenwood, Larry R

To: Hallen, Richard T
Cc: Bryan, Samuel A
Subject: RE: Sr-90 and GEA Results - ASR 5392 Revised

Rich - I have attached the density results - see the tabs labeled density on each worksheet. If you need anything else, just let me know.

99-1595.xls 99-2102.xls

---Original Message---
From: Hallen, Richard T
Sent: Tuesday, August 10, 1999 11:10 AM
To: Greenwood, Larry R
Subject: RE: Sr-90 and GEA Results - ASR 5392 Revised

Larry, You are correct, ASR-5345 (99-1595, 4/30/99) data was all reported on a per gram basis. However, if density data is readily available, I would appreciate receiving it for this series of samples for completeness, Mn-01 to Mn-12 (99-1595 to 99-1606).

Also density data for ASR-5426 (99-2102 to 99-2112) would be nice.

Thanks, Rich Hallen

---Original Message---
From: Greenwood, Larry R
Sent: Friday, August 06, 1999 5:04 PM
To: Hallen, Richard T; Bryan, Samuel A
Cc: Urie, Michael W
Subject: Sr-90 and GEA Results - ASR 5392 Revised

Rich - I revised the reports to give you all data on a weight basis. The densities are shown on one tab; tabs with (g) mean that results are on a per gram basis. If any questions, just let me know. As far as I see, all results for ASR 5345 were already on a weight basis.

<< File: 99-1856.xls >>

---Original Message---
From: Greenwood, Larry R
Sent: Friday, August 06, 1999 5:04 PM
To: Hallen, Richard T; Bryan, Samuel A
Cc: Urie, Michael W
Subject: Sr-90 and GEA Results - ASR 5392 Revised

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APPENDIX B
Appendix B: Test Instruction TI-040 and Data Sheets
| **Title:** Sr/TRU Removal from AN-107, Permanganate Optimization Studies  |
| **Work Location:** RPL SFO SAL  | **Page:** 1 of 13 |
| **Author:** SA Bryan  | **Effective Date:** New |
| **Effective Date:** New  | **Supersedes Date:** New |

| **Use Category Identification:** Reference  |
| **Identified Hazards:**  |
| _ Radiological  |
| x Hazardous Materials  |
| _ Physical Hazards  |
| _ Hazardous Environment  |
| _ Other:  |

| **Required Reviewers:**  |
| x Author  |
| x Technical Reviewer  |
| RPL Manager  |
| Project Manager  |
| RPG Quality Engineer  |
| BNFL (not required)  |

| **Are One-Time Modifications Allowed to this Procedure?**  |
| x Yes  |
| No  |

**NOTE:** If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

| **On-The Job Training Required?**  |
| Yes  |
| No  |

**FOR REVISIONS:**
| **Is retraining to this procedure required?**  |
| Yes  |
| No  |

| **Does the OJT package associated with this procedure require revision to reflect procedure changes?**  |
| Yes  |
| No  |
| N/A  |

<table>
<thead>
<tr>
<th><strong>Approval</strong></th>
<th><strong>Signature</strong></th>
<th><strong>Date</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Author</td>
<td></td>
<td>5/24/99</td>
</tr>
<tr>
<td>Technical Reviewer</td>
<td></td>
<td>5/24/99</td>
</tr>
<tr>
<td>SAL-RPL Representative</td>
<td></td>
<td>5/24/99</td>
</tr>
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</table>

*RT Hallen comments - checks in blue ink. BR Hallen 8/13/99*

**Controlled Document**
Applicability

This test instruction describes the procedure to be used for studies to determine the efficiency of the Sr/TRU removal (decontamination factor) by permanganate addition. The work described herein will be performed in the Shielded Analytical Laboratory (SAL) hot cells located in the Radiochemical Processing Laboratory (RPL). This test instruction supports the Test Plan No. 29953-013 and is a follow on of work conducted in Test Instruction No. 29953-037.

Work is to be performed by hot cell technicians under the supervision of a cognizant scientist. The cognizant scientist shall be responsible for implementation and adherence to this test instruction. This instruction is specific to:

- Sr/TRU removal by permanganate addition to envelope C; archived AN-107 (previously treated to remove Cs) will be used as a representative envelope C sample,
- permanganate addition and precipitation of actual wastes in RPL hot cell facilities, and
- tests performed at Battelle in the RPL, by staff in the Environmental Technology Division.

DRD Reference: none

Schedule Reference: Additional work scope not in baseline.

Justification

This activity is a study using archived AN-107 waste to evaluate the potential of permanganate addition for Sr/TRU removal (decontamination). The preferred method by BNFL, strontium and iron precipitation, has not provided the necessary performance based upon process design criteria.

Objective

The objective of this work is to optimize process conditions to obtain Sr and TRU decontamination with minimal reagent addition, and produce a precipitate that is easy to filter by cross-flow filtration.

Success Criteria

The BNFL flowsheet for AN-107 (Envelope C) requires the separation of the HLW Sr/TRU from the LLW supernate prior to incorporation into glass. BNFL in Phase 1A identified precipitation by strontium and iron addition as the preferred method for decontamination. Decontamination factors are needed to reduce Sr and TRU (primarily Am) to the low level limits. TRU/Am decontamination factors of 5 or more are needed while minimizing the addition of chemical reagents. Sr decontamination factors of greater than 10 are needed. Minimizing reagent addition will also reduce the cost of treatment.

Background

Because of the recent problem with filtration of the iron precipitates from AN-107 simulant, a series of scoping experiments were conducted to determine the ability of permanganate to obtain the necessary DF for Sr and TRU (primarily Am, ~92% of TRU on an activity basis). Permanganate has been examined as both an oxidant (decomplexing waste, solubilizing chromium, and oxidation of technetium species to pertechnetate) and a precursor to MnO2 and/or Mn(OH)2 coprecipitants via the “Method of Appearing Reagents,” Krot et.al. Permanganate was found to preferentially oxidize chromium, followed by organic carbon, and lastly nitrite. In previous tests with archived AN-107, permanganate was successful in obtaining the necessary DFs for Am and TRU, but did not give adequate Sr DF (need a DF for Sr of greater than 10).
Orth et al (1995) recommended permanganate doses of 0.1M for decomplexing SY-101 type wastes where high concentrations of chromium (approximately 0.4%) partly consumed permanganate prior to organic destruction. AN-107 does not have the high chromium values in the sludge compared to SY-101. Due to lower chromium values in AN-107, permanganate was effective for TRU/Am removal at lower doses, 0.05M. Reducing the amount of permanganate added will reduce the amount of HLW waste glass produced. To increase the Sr DF, higher levels of \( \text{Sr(NO}_3\text{)}_2 \) will be added and the samples will be treated at a higher temperature, 50°C, for a longer period of time, 4 hours.

**Spill Protection/Response**

Hot cell technicians shall conduct tests in a manner to minimize the impact of a spill. In the event of a spill, the cognizant scientist shall be notified and a decision will be made to try to recover the sample or repeat the test condition.

**Feed Description**

The Sr/TRU removal tests will use archived AN-107 supernatant previously treated for cesium removal (Envelope C). This feed was diluted and chemicals added such that the approximate final concentrations are: 5M sodium and no hydroxide. Approximately 2.1 L of archived AN-107 supernatant is available for scoping/optimization tests, of this approximately 284 mL will be needed for these studies.

**Equipment Description**

The permanganate addition tests will be conducted on a small scale, approximately 20 mL each. Appropriate glass vials will be used, such that the samples can be heated in a sample block heater to a temperature of 50°C. Some mixing/stirring of the samples after chemical addition should be provided, this may be as simple as periodic swirling the samples. Reagents will be added slowly as liquids, and stirred/mixed/swirled after each reagent is added. Some experiments will require two or more different reagents to be added in the proper sequence as detailed in the test matrix.

**Work Instructions**

1.0 **Applicability**

This test instruction is to be used to perform tests for Sr/TRU removal by permanganate addition. Three (3) 1-L bottles of archived AN-107 supernatant previously treated by Cs ion exchange is available in SAL for these scoping tests. One bottle of the AN-107, C3E3, waste will be used for these tests.

2.0 **Supporting Documents**

This test instruction is not a stand-alone document. Sr/TRU Precipitation and analytical requirements for all BNFL related work are contained in PNNL Test Plan No. BNFL-TP-29953-013. TP-29953-013 also contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation. These are follow on studies to TI-29953-037.

3.0 **Responsible Staff**

The staff responsible for executing this test plan are as follows.
- Task Manager – Rich Hallen
- SFO Manager – Randy Thornhill (Rick Steele)
- Test Scientists – Sam Bryan, Rich Hallen
- Hot Cell Technician – Vaughn Hoops
- Radiological Control Technician
4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Fifteen each 40 mL glass and plastic scintillation vials for filtered, analytical samples, pre-labeled as follows: PR-01 through PR-15. And 14, 40-mL glass scintillation vials for conducting experiments, labeled T-14, with a 20 mL volume mark on each vial.

2. A 0.5 liter polyethylene bottle or equivalent to use for caustic adjustment to the waste. Mark a line at 300 mL on bottle and label, Archived AN-107 caustic adjusted to 1M.

3. 14 - disposable syringes and 0.45 micron syringe filters.

4. AN-107 waste in SAL-cell. Use archived sample from "Tc Removal Flow Studies" (Project No. 25865). Archive sample # C3E3

5. heating device

6. volume dispensing or measuring device, such as graduated tube or cylinder, for determining the density of the AN-107 sample

4.2 Equipment

1. 100 gram balance

2. Hand held camera (if convenient)

3. Stop-watch

4. Calculator

5. Hot plate

6. Thermometer

4.3 Reagents Needed In Hot Cell (see prep sheet)

1. 20 mL of 19M NaOH

2. 10 mL of 1M Ca(NO₃)₂

3. 25 mL of 1M Sr(NO₃)₂

4. 25 mL of 1M NaMnO₄

5. NaN₃ weighed in vial #13

6. HNO₃ weighed in vial #14

4.4 Other Supplies

1. BNFL-TI-29953-040 (this test instruction)

2. Laboratory Record Book (use Red Bound, BNFL lab notebook, record number of book, BNW-13733)

3. BNFL-TP-29953-013 (Hallen 1999)

5.0 Test Instructions

The laboratory record book (LRB) shall be used to record other testing information as required by this test instruction and all test conditions not stated by this test instruction.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as practical. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Prestart

5.1.1 Prepare solutions according to the attached preparation sheet. Calculate solution densities and record these values. All vials should be labeled and marked with the 20 mL line before they are taken into the hot cell. NOTE: Tare weigh bottle/vials with caps/lids. Keep lids on containers to minimize potential for spill, and to prevent evaporation.
For caustic adjustment to the archived AN-107: Outside of the hot cell a 0.5 Liter poly bottle should be marked with a line on the bottle at 300 mL and labeled “Archive AN-107, caustic adjusted to 1M OH.” Tare the empty bottle, record tare weight. Add 12 grams of solid NaOH pellets, or 24 grams (16 mL) of 19M (50%) NaOH solution to poly bottle. Cap tightly for transfer into the hot cell. Transfer a second poly bottle marked with a line at 100mL to be used for 3 x 100mL additions to the “Archive AN-107, caustic adjusted to 1M OH.” bottle. Weigh and record mass of the poly bottle before and after each 100mL addition.

Tare bottle 7.3775g, bottle + NaOH 14.3976g

Tare 100mL poly bottle bottle + addition 1.45371 g, bottle + addition 2.484167 g bottle + addition 3.1456306 g

Type of NaOH used 19M 50% Amount of NaOH added to bottle 24.1201 g

For test #13: Take vial #13, tare empty vial, add 2.55 grams of NaN03 to the vial. Record the weight added to the vial, and set with other vials for transfer into the hot cell. Then treat as usual in hot cell with other samples.

Tare vial #13 24.918 g, vial + NaNO3 27.4994 g

amount of NaNO3 added to vial #13 2.5839 g

For test #14: Before going into the hot cell, take vial #14, tare the empty vial, add 1.8 grams or 1.27 mL of concentrated Nitric Acid to vial, reweigh vial and record weight. Tightly cap vial and place with other for transfer into the hot cells.

Tare vial #14 21.4692 g, vial + HNO3 26.3744 g, amount of HNO3 added to vial #14 1.8701 g

5.1.2 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.4 Obtain the following information:

M&TE List: □ Balance 1: (and Balance 2 if used)

Calib ID 360-06-01-040 Calib Exp Date 8/99 360-06-01-016 8/99

Cell 2
5.2 Operation

5.2.1 First, hydroxide adjustment must be done to the waste. It is preferred that this be completed a day in advance of the test matrix. In side the hot cell, transfer waste from bottle labeled “AN-107 - C3E3” to the 300 mL line of the poly bottle (now containing NaOH). Add 300 mL or 381 g of waste if NaOH pellets were used or add 284 mL or 360.7 g of waste if 19M NaOH was used (density = 1.21 g/mL). Record tare of bottle and weight after waste added.

Tare of bottle __________ g, bottle + waste __________ g, waste added __________ g

Replace cap tightly to keep from picking up CO₂ from the air. Invert bottle several times to well mix. Waste solution will warm with dissolution/dilution of the NaOH. This is the material to use for all of these experiments, with the exception of test #14 which has special instruction starting with the original AN-107 - C3E3 waste. Set this waste aside and allow to cool to cell temperature. After cool, use a volumetric flask (ball flask) to determine the new density of this solution. Record the density of the waste, and use this density to determine the weight of 20 mL of waste.

Tare flask __________ g, flask + waste __________ g, waste mass __________ g, flask volume __________ mL

density of AN-107 w/caustic __________ g/mL

Record Hot Cell temperature __________ °C

For vial #14: This vial/sample should be prepared ahead of time with the hydroxide adjustment described above. After the hydroxide adjustment is complete and the bottle of AN-107-C3E3 is still available, take vial #14 (which now contains concentrated HNO₃) and record the tare weight. Then very slowly, drop wise, add AN-107 waste to vial #14 containing the nitric acid. The waste will react with the acid, foam and bubble, liberating carbon dioxide. Periodically swirl the vial to insure good mixing. The sample will also heat up because of the acid-base reaction. The waste is to be added slowly until approximately 10 mL is added and the bubbling/foaming has stopped. Then the additional 10 mL, up to the total volume of 20 mL of waste can be added. Record the weight of vial #14 with waste. Allow this sample to cool to cell temperature. Then add 1.11 mL or 1.47 grams of 19M (50%) NaOH. Record weight. Place vial #14 back with the other vials and treat with other samples as specified in test instructions.

Tare of vial __________ g, vial + waste __________ g, vial + waste + NaOH __________ g

weight of waste added __________ g and weight of NaOH added __________ g

Review the test matrix shown below, Table 1. Record data in Table 2. Note to check each activity when complete. This should be done and verified by the cognizant scientist.
Table 1. Test Matrix.

<table>
<thead>
<tr>
<th>Check complete</th>
<th>Test #</th>
<th>Target [MnO4]</th>
<th>Target [Sr]</th>
<th>Other Addition</th>
<th>Comment</th>
<th>Scientist Verification</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>control-unfiltered</td>
<td>SAB</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>none</td>
<td>none</td>
<td>none</td>
<td>control-filtered</td>
<td>SAB</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.05 M</td>
<td>none</td>
<td>none</td>
<td>repeat of MN-07(^{(a)})</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>0.08 M</td>
<td>none</td>
<td>none</td>
<td>repeat of MN-08(^{(a)})</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.16 M</td>
<td>none</td>
<td>none</td>
<td>2X [MnO4]</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.05 M</td>
<td>0.075 M</td>
<td>none</td>
<td>base case</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>0.05 M</td>
<td>0.075 M</td>
<td>none</td>
<td>duplicate of base case</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>0.03 M</td>
<td>0.075 M</td>
<td>none</td>
<td>low [MnO4]</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>0.05 M</td>
<td>0.05 M</td>
<td>none</td>
<td>low Sr</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.03 M</td>
<td>0.05 M</td>
<td>none</td>
<td>low MnO4 and Sr</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>0.03 M</td>
<td>0.05 M</td>
<td>0.05M Ca</td>
<td>Ca effect</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>0.05 M</td>
<td>0.075 M</td>
<td>0.05M Ca</td>
<td>Ca effect</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>0.05 M</td>
<td>0.075 M</td>
<td>1.5 M Na(^{(a)})</td>
<td>Na effect</td>
<td>SAB(^{(a)})</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>0.05 M</td>
<td>none</td>
<td>1 M H(^{+(a)})</td>
<td>CO3 effect</td>
<td>SAB(^{(a)})</td>
</tr>
</tbody>
</table>

** See special instruction for samples 13 and 14.

\(^{(a)}\) Described in BNFL-TI-29953-037

The permanganate oxidation reaction generates some heat but at these low volume additions, the sample is expected to reach only about 30°C without external heating. After all of the chemical additions are complete to all of the vials, the vials (except for PR-01, control) should be heated to 50°C and held at this temperature for 4 hours. The vials should be periodically removed from the heat block and swirled to insure samples are mixed. Sample will not generate significant gas, or built up pressure at 50°C but vial caps do not need to be overly tight during heating.
5.2.2 Record the weights (and volumes where appropriate) of all vials, samples, additions, and dilutions. After the reaction time of 4 hours at 50°C, the samples can be centrifuged to allow easier filtration prior to analytical preparation. If possible, record the volume of centrifuged solids. For the base case, (test PR-06 or PR-07, 0.05M MnO₄⁻ and 0.075 Sr) the centrifuged solids should be digested and submitted for analyses. Decant the supernate from this sample, record the weight of solids/solution remaining; the solids will be submitted as is for acid digestion/analysis (see Table 3 below). Note that the AN-107/OH "control - unfiltered" (PR-01) should not be heated, should not be filtered with the syringe filter, and needs to be acid digested "as is" (because it contains some solids) for analyses. The "AN-107/OH as is -
filtrate” (PR-02) will be the same “AN-107/OH” waste but treated as the other samples, i.e. heated and filtered with others samples (centrifuged then filtered the same manner as the other samples, syringe filter).

6.0 Sample Analysis

All sample dilution/digestions are to be recorded noting both volume and mass. The data for from preparation of the samples for analyses shall be recorded in a table format, or on a data sheet. The point of contact for the sample analyses from these tests is Rick Steele.

6.1 Chemical and Radiochemical Analysis

Table 3 below shows the sample analysis list. The table lists the analyses to be performed on samples generated from this test instruction.

Table 3. Samples and Their Required Analyses

<table>
<thead>
<tr>
<th>Process Variable</th>
<th>Vial ID</th>
<th>Sample Type</th>
<th>Sample Preparation</th>
<th>Analysis Description(a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>caustic adjusted AN-107/OH as is</td>
<td>PR-01</td>
<td>some solids</td>
<td>acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>AN-107/OH as is</td>
<td>PR-02</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH]^−</td>
</tr>
<tr>
<td>0.05 M MnO4 only</td>
<td>PR-03</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.08 M MnO4 only</td>
<td>PR-04</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.16 M MnO4 only</td>
<td>PR-05</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.05 M MnO4 + Sr</td>
<td>PR-06</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH]^−</td>
</tr>
<tr>
<td>0.05 M MnO4+ Sr</td>
<td>PR-07</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.03 M MnO4+ Sr</td>
<td>PR-08</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.05 M MnO4+ Sr</td>
<td>PR-09</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.03 M MnO4+ Sr</td>
<td>PR-10</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.03 M MnO4+ Sr + Ca</td>
<td>PR-11</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.05 M MnO4+ Sr + Ca</td>
<td>PR-12</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH]^−</td>
</tr>
<tr>
<td>0.05 M MnO4+ Sr</td>
<td>PR-13</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>0.05 M MnO4</td>
<td>PR-14</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH]^−</td>
</tr>
<tr>
<td>solids from PR-06</td>
<td>PR-15</td>
<td>Centrifuge Solids</td>
<td>acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
</tbody>
</table>

(a) Descriptions of analyses are contained in Table 4.
(b) Separate vial for [OH] analysis, filtered, but no acid digest treatment.

Table 4. Description of Analyses

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Analysis Method</th>
<th>PNNL Procedure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Americium-241, Eu isotopes</td>
<td>GEA</td>
<td>PNL-ALO-450</td>
</tr>
<tr>
<td>Strontium-90 (Yttrium-90)</td>
<td>Separations and Beta Counting</td>
<td>PNL-ALO-476/431</td>
</tr>
<tr>
<td>Hydroxide</td>
<td>EPA SW-846 Modified Method, 310(3)</td>
<td>PNL-ALO-228</td>
</tr>
<tr>
<td>Metal Ions (see Table 5 list)</td>
<td>ICP-AES</td>
<td>PNL-ALO-211/280</td>
</tr>
</tbody>
</table>
Table 5. Analytical Requirements for Supernate/Filtrate and Centrifuged Solids

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Centrifuged Solids Minimum Reportable Quantity microCi/gm</th>
<th>Supernate/Filtrate Minimum Reportable Quantity microCi/ml</th>
<th>Analysis Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strontium-90</td>
<td>7.0E+01</td>
<td>1.5E-01</td>
<td>Chemical Separation &amp; Beta Count</td>
</tr>
<tr>
<td>Americium-241</td>
<td>1.2E-03</td>
<td>7.2E-04</td>
<td>GEA</td>
</tr>
<tr>
<td>Al</td>
<td>3.3E+02</td>
<td>7.5E+01</td>
<td></td>
</tr>
<tr>
<td>Ba</td>
<td>6.0E+02</td>
<td>7.8E+01</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>1.8E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Cd</td>
<td>1.1E+01</td>
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<tr>
<td>Co</td>
<td>3.0E+00</td>
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<td></td>
</tr>
<tr>
<td>Cr</td>
<td>1.2E+02</td>
<td>1.5E+01</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>1.8E+01</td>
<td>1.7E+01</td>
<td></td>
</tr>
<tr>
<td>Eu</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>1.4E+02</td>
<td>1.5E+02</td>
<td>Acid Digestion followed by ICP-AES</td>
</tr>
<tr>
<td>K</td>
<td>1.5E+03</td>
<td>2.0E+02</td>
<td></td>
</tr>
<tr>
<td>La</td>
<td>6.0E+01</td>
<td>3.5E+01</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>5.4E+02</td>
<td>1.5E+02</td>
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<td>Mn</td>
<td>3.0E+02</td>
<td>1.5E+02</td>
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<td>Mo</td>
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<td>Na</td>
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<td>Ni</td>
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<td>Pb</td>
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<td>Si</td>
<td>3.0E+03</td>
<td>1.7E+02</td>
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<td>Sr</td>
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<tr>
<td>Zn</td>
<td>6.0E+00</td>
<td>1.65E+01</td>
<td></td>
</tr>
<tr>
<td>OH-</td>
<td></td>
<td>0.05M</td>
<td></td>
</tr>
</tbody>
</table>
7.0 Calculation and Important Information

Density of starting AN-107 sample that has cesium removed = 1.26 g/mL

Estimated density of AN-107/OH caustic adjusted to 1M = estimated 1.31 g/mL

Density of 1M NaMnO₄ solution = 1.086 g/mL

Density of 19M NaOH solution = 1.51 g/mL

Density of 1M Ca(NO₃)₂ solution = 1.117 g/mL (extrapolated from CRC data)

Density of 1M Sr(NO₃)₂ solution = 1.157 g/mL (extrapolated from CRC data)

20 mL = 26.2 grams of AN-107 caustic adjusted waste

Mass of Solutions based on above density data. Densities (and masses) need to be verified based on actual solution densities. This will be performed after solutions are prepared in Step 4.3.

<table>
<thead>
<tr>
<th>Sodium Manganese (IV) Oxide Levels</th>
<th>mL/20mL waste</th>
<th>grams/20mL waste</th>
<th>density</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.03 M</td>
<td>0.62 mL</td>
<td>0.67 g</td>
<td>1.086</td>
</tr>
<tr>
<td>0.05 M</td>
<td>1.05 mL</td>
<td>1.14 g</td>
<td>1.086</td>
</tr>
<tr>
<td>0.08 M</td>
<td>1.74 mL</td>
<td>1.89 g</td>
<td>1.086</td>
</tr>
<tr>
<td>0.16 M</td>
<td>3.81 mL</td>
<td>4.14 g</td>
<td>1.086</td>
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</table>

<table>
<thead>
<tr>
<th>OH Level</th>
<th>mL/20mL waste</th>
<th>grams/20mL waste</th>
<th>density</th>
</tr>
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<tbody>
<tr>
<td>1.0 M</td>
<td>1.11 mL</td>
<td>1.68 g</td>
<td>1.51</td>
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<table>
<thead>
<tr>
<th>Calcium (II) Nitrate Levels</th>
<th>mL/20mL waste</th>
<th>grams/20mL waste</th>
<th>density</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01 M</td>
<td>0.20 mL</td>
<td>0.23 g</td>
<td>1.12</td>
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<tr>
<td>0.05 M</td>
<td>1.05 mL</td>
<td>1.22 g</td>
<td>1.16</td>
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<tr>
<td>0.075 M</td>
<td>1.62 mL</td>
<td>1.88 g</td>
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<table>
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<tr>
<th>Strontium (II) Nitrate Levels</th>
<th>mL/20mL waste</th>
<th>grams/20mL waste</th>
<th>density</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 M</td>
<td>1.27 mL conc  HNO₃</td>
<td>2.55 g of solids NaNO₃</td>
<td>1.42</td>
</tr>
<tr>
<td>HNO₃</td>
<td>1.0 M</td>
<td>1.80 g of conc  HNO₃</td>
<td></td>
</tr>
<tr>
<td>Test</td>
<td>Condition</td>
<td>Sr</td>
<td>Chemical</td>
</tr>
<tr>
<td>------</td>
<td>-----------</td>
<td>----</td>
<td>----------</td>
</tr>
<tr>
<td>1</td>
<td>unfiltered control</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>filtered control</td>
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<td>0.00</td>
</tr>
<tr>
<td>3</td>
<td>0.05 M</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>4</td>
<td>0.08 M</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>5</td>
<td>0.16 M</td>
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</tr>
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</tr>
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<td>0.05 M</td>
<td>0.00</td>
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</tr>
<tr>
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</tr>
<tr>
<td>14</td>
<td>0.05 M</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

**Initial Volume: 20 mL**

**Initial Mass: 26.2 g**
8.0 References


Preparation of 19M NaOH

19M NaOH is needed to adjust the waste to 1M OH-. This solution will be added to the archived AN-107 before the optimization tests. Record all information and observations on prep sheet or in lab notebook.

Balance number: 380-06-01-012
Balance calibration: 3-1-99

Makeup NaOH solution
use NaOH pellets/beads 19 M
40 grams/mole (FW) (lot # 19 Lof # 6)

Tare 25 mL volumetric flask ________ grams

add 19 g NaOH flask + NaOH ________ grams

actual weight of NaOH add 18.94815 grams

add approximately 1/2 the volume of Milli-Q water and swirl to dissolve solution/flask will get very hot, be careful. Allow to cool down.

add the other 1/2 the volume of Milli-Q water and swirl until all dissolves
Allow to cool to near room temperature, 20-25 C.

fill to volumetric line with Milli-Q water. Record total weight: 37.81147 grams

calculate actual [OH] = actual weight/40.00/volume in Liters = ______ M

calculate density of solution, weight of solution/volume = ______ grams/mL

Transfer stock solution to bottle and label unique ID #

Date prepared: 5/20/09
Prepared by: 2Halin
Work Package Number: W51702
Metal Additions: Prep of Stock Solutions

Ca, Sr, or Eu need to be added to the waste for Sr/TRU decontamination. These solutions will be added before the permanganate.

Record all information and observations on prep sheet or in lab notebook.

Balance number: 330-06-01-01 2
Balance calibration: 3-1-99.

Makeup Ca(NO3)2 solution

use Ca(NO3)2*4H2O Fisher-Scientific Cert. ACS

236.15 grams/mole (FW) (lot #90308)

Tare: 25 mL volumetric flask grams
add: 5.90375 grams Ca(NO3)2 flask + Ca(NO3)2 grams
actual weight of Ca(NO3)2 added grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved

fill to volumetric line with Milli-Q water. Record total weight: grams

calculate actual [Ca] = actual weight/236.15/volume in Liters = M

calculate density of solution, weight of solution/volume = grams/mL

Transfer stock solution to bottle and label unique ID #

Makeup Sr(NO3)2 solution

use Sr(NO3)2 Fisher-Scientific Cert. ACS

211.63 grams/mole (FW) (lot #981987)

Tare: 25 mL volumetric flask grams
add: 5.29075 grams Sr(NO3)2 flask + Sr(NO3)2 grams
actual weight of Sr(NO3)2 added grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved

fill to volumetric line with Milli-Q water. Record total weight: grams

calculate actual [Sr] = actual weight/211.63/volume in Liters = M

calculate density of solution, weight of solution/volume = grams/mL

Transfer stock solution to bottle and label unique ID #

Makeup Eu(NO3)3 solution

use Eu(NO3)3*6H2O

446.07 grams/mole (FW) (lot #)

Tare: 25 mL volumetric flask grams
add: 11.15175 g Eu(NO3)3*6H2O flask + Eu(NO3)3*6H2O grams
actual weight of Eu(NO3)3*6H2O add grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved

fill to volumetric line with Milli-Q water. Record total weight: grams

calculate actual [Eu] = actual weight/446.07/volume in Liters = M

calculate density of solution, weight of solution/volume = grams/mL

Transfer stock solution to bottle and label unique ID #

Date prepared:
Prepared by:
Work Package Number:

Calculations prepared by RT Hallen
Calculations checked by: 5/19/99
Preparation of Stock Permanganate Solution

Permanganate needs to be added to the waste for Sr/TRU decontamination. This solution will be added after any other reagents, if needed. Record all information and observations on prep sheet or in lab notebook. Note this is Na now!!

Balance number: 380-01-01
Balance calibration: 3-1-99.

Makeup stock NaMnO4 solution 1 M
use NaMnO4.1 H2O, 97+, ACS Reagent Grade
159.94 grams/mole (FW) (lot # A010675901)

Tare 50 mL volumetric flask grams
add 7.997 grams NaMnO4 flask + NaMnO4 grams
actual weight of NaMnO4 added 7.997 grams

add approximately 1/2 the volume of Milli-Q water and swirl until dissolved
add approximately 5 mL of 1N NaOH or 0.05 mL of 10N NaOH to stabilize permanganate, 2 drops
fill to volumetric line with Milli-Q water. Record total weight: 54.254 grams

calculate actual [MnO4] = actual weight/159.94/volume in Liters = 1.00 M

calculate density of solution, weight of solution/volume = 1.036 grams/mL

Transfer stock solution to bottle and label unique ID #

Date prepared: 5/20/99
Prepared by: D. Adler
Work Package Number: W51302

pH = 10.4
Appendix C: Test Instruction TI-043
Title: Sr/TRU Removal from AN-107 Archived Waste and Diluted Feed, Minimum Reagent Addition Studies

Work Location: RPL SFO SAL

Author: SA Bryan

Effective Date: New

Supersedes Date: New

Use Category Identification: Reference

Identification of Hazards:
- Radiological
- Hazardous Materials
- Physical Hazards
- Hazardous Environment
- Other:

Required Reviewers:
- Author
- RPL Manager
- Project Manager
- RPG Quality Engineer
- BNFL (not required)

Are One-Time Modifications Allowed to this Procedure?  Yes  No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required?  Yes  No

FOR REVISIONS:
Is retraining to this procedure required?  Yes  No

Does the OJT package associated with this procedure require revision to reflect procedure changes?  Yes  No  N/A

Approval  Signature  Date
Author  Samuel A. Bryan  6/23/99
SAL-RPL Representative  Rick T. Steele  6/23/99

Controlled Document
Applicability

This test instruction describes the procedure to be used for studies to determine the efficiency of the Sr/TRU removal (decontamination factor) by permanganate addition. The work described herein will be performed in the Shielded Analytical Laboratory (SAL) hot cells located in the Radiochemical Processing Laboratory (RPL). This test instruction supports the Test Plan No. 29953-013 and is a follow on of work conducted in Test Instruction No. 29953-037 and -040.

Work is to be performed by hot cell technicians under the supervision of a cognizant scientist. The cognizant scientist shall be responsible for implementation and adherence to this test instruction. This instruction is specific to:

- Sr/TRU removal by permanganate addition to envelope C; archived AN-107 (previously treated to remove Cs) and AN-107 diluted feed (remaining from leaching studies) will be used as a representative envelope C sample,
- permanganate addition and precipitation of actual wastes in RPL hot cell facilities, and
- tests performed at Battelle in the RPL, by staff in the Environmental Technology Division.

DRD Reference: none

Schedule Reference: Additional work scope not in baseline.

Justification

This activity is a study using archived AN-107 waste and actual AN-107 diluted feed to evaluate the potential of minimizing the amount of strontium and permanganate addition to achieve the necessary Sr/TRU removal (decontamination). The preferred method by BNFL, strontium and iron precipitation, has not provided the necessary performance based upon process design criteria.

Objective

The objective of this work is to optimize process conditions to obtain Sr and TRU decontamination with minimal reagent addition, and produce a precipitate that is easy to filter by cross-flow filtration.

Success Criteria

The BNFL flowsheet for AN-107 (Envelope C) requires the separation of the HLW Sr/TRU from the LLW supernate prior to incorporation into glass. BNFL in Phase 1A identified precipitation by strontium and iron addition as the preferred method for decontamination. Decontamination factors are needed to reduce Sr and TRU (primarily Am) to the low level limits. TRU/Am decontamination factors of 5 or more are needed while minimizing the addition of chemical reagents. Sr decontamination factors of greater than 10 are needed. Minimizing reagent addition will also reduce the cost of treatment.

Background

Because of the recent problem with filtration of the iron precipitates from AN-107 simulant, a series of scoping experiments were conducted to determine the ability of permanganate to obtain the necessary DF for Sr and TRU (primarily Am, ~92% of TRU on an activity basis). Permanganate has been examined as both an oxidant (decomplexing waste, solubilizing chromium, and oxidation of technetium species to pertechnetate) and a precursor to MnO₂ and/or Mn(OH)₂ coprecipitants via the “Method of Appearing Reagents,” Krot et.al. Permanganate was found to preferentially oxidize chromium, followed by organic carbon, and lastly nitrite. In previous tests with archived AN-107, permanganate was successful in obtaining the necessary DFs for Am and TRU, but did not give adequate Sr DF (need a DF for Sr of greater than 10).
Orth et al (1995) recommended permanganate doses of 0.1M for decomplexing SY-101 type wastes where high concentrations of chromium (approximately 0.4%) partly consumed permanganate prior to organic destruction. AN-107 does not have the high chromium values in the sludge compared to SY-101. Due to lower chromium values in AN-107, permanganate was effective for TRU/Am removal at lower doses, 0.05M. Reducing the amount of permanganate added will reduce the amount of HLW waste glass produced. To increase the Sr DF, higher levels of Sr(NO₃)₂ be added and the samples will be treated at a higher temperature, 50°C, for a longer period of time, 4 hours.

Spill Protection/Response

Hot cell technicians shall conduct tests in a manner to minimize the impact of a spill. In the event of a spill, the cognizant scientist shall be notified and a decision will be made to try to recover the sample or repeat the test condition.

Feed Description

The Sr/TRU removal tests will use archived AN-107 supernatant previously treated for cesium removal (Envelope C) with the free hydroxide adjusted to 1 and 0.5 M. AN-107 diluted feed left over from leaching studies will be used for the real waste tests. Approximately 100 mL of caustic adjusted, archived AN-107 (AN-107/OH) supernatant is available for these tests. At least 40 mL of archived AN-107 (C3E3) is left to prepare the samples at 0.5 M free hydroxide. Samples CL-1 and AQ-10 (40 mL each) will be combined and used as the AN-107 diluted feed.

Equipment Description

The permanganate addition tests will be conducted on a small scale, approximately 20 mL each. Appropriate glass vials will be used, such that the samples can be heated in a sample block heater to a temperature of 50°C. Some mixing/stirring of the samples after chemical addition should be provided, this may be as simple as periodic swirling the samples. Reagents will be added slowly as liquids, and stirred/mixed/swirled after each reagent is added. Experiments will require addition of two different reagents to be added in the proper sequence as detailed in the test matrix. The samples are to be heated for two hours in the sample block heater for 2 hours after the addition of each reagent.

Work Instructions

1.0 Applicability

This test instruction is to be used to perform tests for Sr/TRU removal by strontium and permanganate addition. The studies shall use approximately 100 mL of caustic adjusted, archived AN-107 (AN-107/OH), approximately 40 mL of archived AN-107 (bottle ID# C3E3), and approximately 80 mL of AN-107 diluted feed (combined CL-1 and AQ-10 samples from Lumetta’s leaching studies).

2.0 Supporting Documents

This test instruction is not a stand-alone document. Sr/TRU Precipitation and analytical requirements for all BNFL related work are contained in PNNL Test Plan No. BNFL-TP-29953-013. TP-29953-013 also contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation. These are follow on studies to TI-29953-037 and TI-29953-040.

3.0 Responsible Staff

The staff responsible for executing this test plan are as follows.

- Task Manager – Rich Hallen
- SFO Manager – Randy Thornhill (Rick Steele)
- Test Scientists – Sam Bryan, Rich Hallen
4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

1. Eleven each 20 mL glass and plastic scintillation vials for filtered, analytical samples, pre-labeled as follows: OP-01 through OP-05, LH-06 and LH-07, and RW-08 through RW-11. And 11, 40-mL glass scintillation vials for conducting experiments, labeled 1-11, with a 20 mL volume mark on each vial.
2. A 100 mL polyethylene bottle or equivalent to use for adding NaN\(_3\) to the caustic adjusted waste, labeled “archived AN-107 caustic adjusted to 1 M plus 1.5 M sodium.”
3. A 50 mL vial/bottle to adjust C3E3 waste to 0.5M free hydroxide and additional 1.5 M sodium, labeled “archived AN-107 caustic adjusted to 0.5M plus 1.5 M sodium.”
4. A 100 mL bottle to combine AN-107 diluted feed from samples CL-1 and AQ-10, labeled “AN-107 diluted feed combined sample.”
5. 11 - disposable syringes and 0.45 micron syringe filters.
6. AN-107 wastes in SAL-cell. Archived AN-107 caustic adjusted to 1 M, archived sample from "Tc Removal Flow Studies" (Project No. 25865) bottle number C3E3, and AN-107 diluted feed from leaching studies, sample numbers CL-1 and A_Q-10.

4.2 Equipment

1. 100 gram balance
2. Hand held camera (if convenient)
3. Stop-watch
4. Calculator
5. Hot plate
6. Thermometer

4.3 Reagents Needed In Hot Cell (see prep sheet)

1. 2 mL of 19M NaOH (or 1 grams of NaOH pellets)
2. 10 mL of 1M Sr(NO\(_3\))\(_2\)
3. 6 mL of 1M NaMnO\(_4\)
4. 16 grams reagent grade NaN\(_3\)

4.4 Other Supplies

1. BNFL-TI-29953-043 (this test instruction)
2. Laboratory Record Book (use Red Bound, BNFL lab notebook, record number of book, BNW-13733)
3. BNFL-TP-29953-013 (Hallen 1999)

5.0 Test Instructions

The laboratory record book (LRB) shall be used to record other testing information as required by this test instruction and all test conditions not stated by this test instruction.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as practical. Those tools that are reused should be washed and rinsed prior to reuse.
Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Prestart

5.1.1 Prepare solutions according to the attached preparation sheet. Calculate solution densities and record these values. All vials should be labeled and marked with the 20 mL line before they are taken into the hot cell. 

NOTE: Tare weigh bottle/vials with caps/lids. Keep lids on containers to minimize potential for spill, and to prevent evaporation.

For archived AN-107 caustic adjusted to 1 M plus 1.5 M sodium: Outside of the hot cell a 100 mL bottle should be marked with a line on the bottle at 80 mL and labeled “Archived AN-107 caustic adjusted to 1M OH plus 1.5M sodium.” Tare the empty bottle, record tare weight. Add 10.2 grams of solid NaNO₃ bottle. Cap tightly for transfer into the hot cell.

\[
\text{Tare bottle: } 24.1818 \, \text{g, bottle + NaNO}_3 \quad 34.3843 \quad + \quad \text{NaOH} \quad 40.7863 \quad = \quad 50.2537 \, \text{g}
\]

For archived AN-107 caustic adjusted to 0.5 M plus 1.5 M sodium: Outside of the hot cell a 50 mL vial/bottle should be marked with a line at 40 mL and labeled “Archived AN-107 caustic adjusted to 0.5M OH plus 1.5M sodium.” Tare the empty bottle, record tare weight. Add 5.1 grams of solid NaNO₃ bottle. Add 1.68 grams of 19 M NaOH (50% by weight) (or 0.84 grams of NaOH pellets). Cap tightly for transfer into the hot cell.

\[
\text{Tare bottle: } 24.0993 \, \text{g, bottle + NaNO}_3 \quad 29.1929 \quad + \quad \text{NaOH} \quad 30.9028 \quad = \quad 84.1950 \, \text{g}
\]

For AN-107 diluted feed: Take a 100 mL vial/bottle into the hot cell for combining CL-1 and AQ-10 that is labeled AN-107 diluted feed combined sample.

5.1.2 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

Record Unique ID # of reagents:

1M NaMnO₄
19N NaOH 501, Na₂O₂
1M Sr(NO₃)₂

5.1.3 Initial and date when each item is completed.

Review the test matrix (Table 1) in the test instructions in BNFL-TI-29953-043. Note the calculation worksheet, which gives quantities of reagents to add. Reagents can be added as volume but always record the mass added.

5.1.4 Obtain the following information:

M&TE List:

____ Balance 1: (and Balance 2 if used)

Calib ID ____________ Calib Exp Date ________

Location __________

____ temperature reading device (thermometer or thermcouple/reader):
5.2 Operation

(Note: The sodium adjustment, caustic adjustment, and sample combining could all be completed a day in advance of the test matrix.)

5.2.1 First, 20 mL or 25.2 grams of waste (density = 1.26 g/mL) from the archived AN-107 caustic adjusted to 1 M must be transferred to vial # OP-02, record weight transferred in Table 2 below. After this is complete, transfer 80 mL or 100.8 grams of the caustic adjusted waste to the 100 mL bottle containing the NaNO₃, Archived AN-107 caustic adjusted to 1 M plus 1.5 M sodium. Record data below: (If 80 mL of the caustic adjusted AN-107/OH is not available add all plus any additional “C3E4” waste as required to give 80 mL).

Tare of bottle 40.7863 g, bottle + waste 42.7153 g, waste added 92.9810 g

Replace cap tightly to keep from picking up CO₂ from the air. Invert bottle several times to well mix. Waste solution will warm with dissolution/dilution of the NaOH.

All solutions will have new density. If needed and after cool, a volumetric flask (ball flask) can be used to determine the new density of these solution. Record the density of the waste, and use this density to determine the weight of 20 mL of waste.

tare flask 9.3326 g, flask + waste 22.6402 g, waste mass 13.3076 g, flask volume 10.00 mL

new density of archived AN-107 1.3308 g/mL

Record Hot Cell temperature 25°C

Hydroxide and sodium adjustments must be done to the archived waste for tests LH-06 and LH-07. In the hot cell, transfer 40 mL of waste from bottle labeled “AN-107 - C3E4” to the vial/bottle labeled “archived AN-107 caustic adjusted to 0.5 M plus 1.5 M sodium (bottle containing NaOH and NaNO₃). Add 40 mL or 50.4 g of waste (density = 1.26 g/mL). Record tare of bottle and weight after waste added.

Tare of bottle 30.9028 g, bottle + waste 35.713 g, waste added 48.6685 g

Replace cap tightly to keep from picking up CO₂ from the air. Invert bottle several times to well mix. Waste solution will warm with dissolution/dilution of the NaOH.

All solutions will have new density. If needed and after cool, a volumetric flask (ball flask) can be used to determine the new density of these solution. Record the density of the waste, and use this density to determine the weight of 20 mL of waste.

tare flask 9.4071 g, flask + waste 22.5401 g, waste mass 13.1330 g, flask volume 10.00 mL

new density of archived AN-107 $1.33$ g/mL.

Record Hot Cell temperature $25^\circ C$.

For RW-08 to RW-11: AN-107 diluted feed from samples CL-1 and AQ-10 need to be combined. Record the tare weight vial/bottle used to combine these samples. Then add each sample and record the weights. (density should be 1.36 g/mL based on analytical report BNFL-RPT-003 Rev. 0)

Replace cap tightly to keep from picking up CO\(_2\) from the air. Invert bottle several times to well mix.

The density should be 1.36 g/mL based on analytical report. If needed and after cool, a volumetric flask (ball flask) can be used to determine the new density of these solution. Record the density of the waste, and use this density to determine the weight of 20 mL of waste.

Replace cap tightly to keep from picking up CO\(_2\) from the air. Invert bottle several times to well mix.

Review the test matrix shown below, Table 1. Record data in Table 2. Note to check each activity when complete. This should be done and verified by the cognizant scientist.
Table 1. Test Matrix.

<table>
<thead>
<tr>
<th>Check complete</th>
<th>Test #</th>
<th>Test ID #</th>
<th>Target [MnO4]</th>
<th>Target [Sr]</th>
<th>AN-107 Waste ID</th>
<th>Comment</th>
<th>Scientist Verification</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>OP-01</td>
<td>none</td>
<td>none</td>
<td>Archived 1M OH + 1.5M Na</td>
<td>control-filtered</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>OP-02</td>
<td>0.03</td>
<td>0.05</td>
<td>Archived, 1M OH</td>
<td>Repeat of PR-10</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>OP-03</td>
<td>0.03</td>
<td>0.05</td>
<td>Archived 1M OH + 1.5M Na</td>
<td>low Sr and MnO4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>OP-04</td>
<td>0.03</td>
<td>0.05</td>
<td>Archived 1M OH + 1.5M Na</td>
<td>Duplicate of 3</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>OP-05</td>
<td>0.03</td>
<td>0.05</td>
<td>Archived 1M OH + 1.5M Na</td>
<td>Reverse addition</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>LH-06</td>
<td>none</td>
<td>none</td>
<td>Archived 0.5M OH + 1.5M Na</td>
<td>control-filtered</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>LH-07</td>
<td>0.03</td>
<td>0.05</td>
<td>Archived 0.5M OH + 1.5M Na</td>
<td>Low hydroxide</td>
<td></td>
</tr>
<tr>
<td></td>
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<td>RW-08</td>
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<td>Diluted feed</td>
<td>control-filtered</td>
<td></td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>RW-09</td>
<td>0.03</td>
<td>0.05</td>
<td>Diluted feed</td>
<td>low Sr and MnO4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>RW-10</td>
<td>0.03</td>
<td>0.05</td>
<td>Diluted feed</td>
<td>Duplicate of 9</td>
<td></td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>RW-11</td>
<td>0.05</td>
<td>0.075</td>
<td>Diluted feed</td>
<td>High Sr and MnO4</td>
<td></td>
</tr>
</tbody>
</table>

The reaction conditions are much different for these tests. The samples are to be heated to 50°C after each chemical addition is complete and held at this temperature for 2 hours. After the first 2 hours of heating the samples should be removed from the heater block and allowed to cool so weighing will be easier (less balance drift). Then add the second reagent and return vials to the heating block for an additional 2 hours. The vials should be periodically removed from the heat block and swirled to insure samples are mixed. Sample will not generate significant gas, or built up pressure at 50°C but vial caps do not need to be overly tight during heating.
### Table 2. Data Sheet.

<table>
<thead>
<tr>
<th>Test #</th>
<th>Vial ID</th>
<th>Vial Tare Weight, g</th>
<th>Number Indicates Order of Reagent Addition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>AN-107 waste added</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>target mL</td>
</tr>
<tr>
<td>1</td>
<td>OP-01</td>
<td>25.0406</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>OP-02</td>
<td>24.3722</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>OP-03</td>
<td>25.0317</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>OP-04</td>
<td>24.9225</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>OP-05</td>
<td>25.0383</td>
<td>20</td>
</tr>
<tr>
<td>6</td>
<td>LH-06</td>
<td>25.1049</td>
<td>20</td>
</tr>
<tr>
<td>7</td>
<td>LH-07</td>
<td>25.0140</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>RW-08</td>
<td>25.2879</td>
<td>20</td>
</tr>
<tr>
<td>9</td>
<td>RW-09</td>
<td>25.0788</td>
<td>20</td>
</tr>
<tr>
<td>10</td>
<td>RW-10</td>
<td>25.0473</td>
<td>20</td>
</tr>
<tr>
<td>11</td>
<td>RW-11</td>
<td>24.9541</td>
<td>20</td>
</tr>
</tbody>
</table>

5.2.2 Record the weights (and volumes where appropriate) of all vials, samples, additions, and dilutions. After the reagent additions and heating are complete, the samples can be centrifuged to allow easier filtration prior to analytical preparation. If possible, record the volume of centrifuged solids. Note that there is no "control - unfiltered" for these studies. The control-filtered (OP-01, LH-06, and RW-08) will be treated as the other samples, i.e. heated and filtered with other samples (centrifuged then filtered the same manner as the other samples, syringe filter).

6.0 Sample Analysis

All sample dilution/digestions are to be recorded noting both volume and mass. The data from preparation of the samples for analyses shall be recorded in a table format, or on a data sheet. The point of contact for the sample analyses from these tests is Rick Steele.
6.1 Chemical and Radiochemical Analysis

Table 3 below shows the sample analysis list. The table lists the analyses to be performed on samples generated from this test instruction.

Table 3. Samples and Their Required Analyses

<table>
<thead>
<tr>
<th>Process Variable</th>
<th>Vial ID</th>
<th>Sample Type</th>
<th>Sample Preparation</th>
<th>Analysis Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1M caustic adjusted AN-107 plus 1.5M Na</td>
<td>OP-01</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>Repeat of PR-10 (no Na)</td>
<td>OP-02</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am</td>
</tr>
<tr>
<td>Low Sr and MnO4</td>
<td>OP-03</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP</td>
</tr>
<tr>
<td>Duplicate of OP-03</td>
<td>OP-04</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am</td>
</tr>
<tr>
<td>Reverse addition</td>
<td>OP-05</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am</td>
</tr>
<tr>
<td>0.05M caustic &amp; 1.5M Na</td>
<td>LH-06</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am</td>
</tr>
<tr>
<td>Low Sr and MnO4</td>
<td>LH-07</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH] (b)</td>
</tr>
<tr>
<td>Diluted Feed Combined</td>
<td>RW-08</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH] (b)</td>
</tr>
<tr>
<td>Low Sr and MnO4</td>
<td>RW-09</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH] (b)</td>
</tr>
<tr>
<td>Duplicate of RW-09</td>
<td>RW-10</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am</td>
</tr>
<tr>
<td>High Sr and MnO4</td>
<td>RW-11</td>
<td>Filtrate</td>
<td>0.45 um disk/acid digest</td>
<td>Sr/Am, ICP, [OH] (b)</td>
</tr>
</tbody>
</table>

(a) Descriptions of analyses are contained in Table 4.
(b) Separate vial for [OH] analysis, filtered, but no acid digest treatment.

Table 4. Description of Analyses

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Analysis Method</th>
<th>PNNL Procedure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Americium-241, Eu isotopes</td>
<td>GEA</td>
<td>PNL-ALO-450</td>
</tr>
<tr>
<td>Strontium-90 (Yttrium-90)</td>
<td>Separations and Beta Counting</td>
<td>PNL-ALO-476/431</td>
</tr>
<tr>
<td>Hydroxide</td>
<td>EPA SW-846 Modified Method, 310(3)</td>
<td>PNL-ALO-228</td>
</tr>
<tr>
<td>Metal Ions (see Table 5 list)</td>
<td>ICP-AES</td>
<td>PNL-ALO-211/280</td>
</tr>
</tbody>
</table>
Table 5. Analytical Requirements for Supernate/Filtrate and Centrifuged Solids

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Centrifuged Solids Minimum Reportable Quantity microCi/gm</th>
<th>Supernate/Filtrate Minimum Reportable Quantity microCi/ml</th>
<th>Analysis Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strontium-90</td>
<td>7.01E+01</td>
<td>1.5E-01</td>
<td>Chemical Separation &amp; Beta Count</td>
</tr>
<tr>
<td>Americium-241</td>
<td>1.2E-03</td>
<td>7.2E-04</td>
<td>GEA</td>
</tr>
<tr>
<td>Al</td>
<td>3.3E+02</td>
<td>7.5E+01</td>
<td></td>
</tr>
<tr>
<td>Ba</td>
<td>6.0E+02</td>
<td>7.8E+01</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>1.8E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Cd</td>
<td>1.1E+01</td>
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<tr>
<td>Co</td>
<td>3.0E+00</td>
<td>3.0E+01</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>1.2E+02</td>
<td>1.5E+01</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>1.8E+01</td>
<td>1.7E+01</td>
<td></td>
</tr>
<tr>
<td>Eu</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>1.4E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>K</td>
<td>1.5E+03</td>
<td>2.0E+02</td>
<td></td>
</tr>
<tr>
<td>La</td>
<td>6.0E+01</td>
<td>3.5E+01</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>5.4E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Mn</td>
<td>3.0E+02</td>
<td>1.5E+02</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>6.0E+00</td>
<td>9.0E+01</td>
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<tr>
<td>Na</td>
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<tr>
<td>Ni</td>
<td>1.6E+02</td>
<td>3.0E+01</td>
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<td>Pb</td>
<td>6.0E+02</td>
<td>3.0E+02</td>
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</tr>
<tr>
<td>Si</td>
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<td>1.7E+02</td>
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</tr>
<tr>
<td>Sr</td>
<td>3.0E+02</td>
<td>8.7E+01</td>
<td></td>
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<tr>
<td>Ti</td>
<td>1.5E+02</td>
<td>1.7E+01</td>
<td></td>
</tr>
<tr>
<td>U</td>
<td>6.0E+02</td>
<td>6.0E+02</td>
<td></td>
</tr>
<tr>
<td>Zn</td>
<td>6.0E+00</td>
<td>1.65E+01</td>
<td></td>
</tr>
<tr>
<td>OH-</td>
<td></td>
<td>0.05M</td>
<td></td>
</tr>
</tbody>
</table>
7.0 Calculation and Important Information

Density of starting AN-107 sample from bottle “C3E3” that has cesium removed = 1.26 g/mL

Density of AN-107/OH caustic adjusted to 1M = 1.26 g/mL

Density of AN-107 diluted feed combined “CL-1+AQ-10” = estimated 1.36 g/mL

Density of 1M NaMnO₄ solution = 1.086 g/mL

Density of 19M NaOH solution (50% by weight) =1.51 g/mL

Density of 1M Sr(NO₃)₂ solution = 1.157 g/mL (extrapolated from CRC data)

20 mL = 25.2 grams of AN-107 caustic adjusted waste

Mass of Solutions based on above density data. Densities (and masses) need to be verified based on actual solution densities. This will be performed after solutions are prepared in Step 4.3.

<table>
<thead>
<tr>
<th>Two NaMnO₄ levels</th>
<th>mL/20mL waste</th>
<th>grams/20mL waste</th>
<th>density</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.03 M</td>
<td>0.62 mL</td>
<td>1 M NaMnO₄</td>
<td>0.67 g 1 M NaMnO₄</td>
</tr>
<tr>
<td>0.05 M</td>
<td>1.05 mL</td>
<td>1 M NaMnO₄</td>
<td>1.14 g 1 M NaMnO₄</td>
</tr>
<tr>
<td>one new OH level</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.5 M</td>
<td>0.56 mL</td>
<td>19N NaOH</td>
<td>0.84 g 19N NaOH</td>
</tr>
<tr>
<td>two Sr levels</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.05 M</td>
<td>1.05 mL</td>
<td>1 M Sr(NO₃)₂</td>
<td>1.22 g 1 M Sr(NO₃)₂</td>
</tr>
<tr>
<td>0.075 M</td>
<td>1.62 mL</td>
<td>1 M Sr(NO₃)₂</td>
<td>1.88 g 1 M Sr(NO₃)₂</td>
</tr>
<tr>
<td>NaNO₃</td>
<td>1.5 M</td>
<td></td>
<td>2.55 g of solids NaNO₃</td>
</tr>
</tbody>
</table>
### Test Sample ID# and Conditions

<table>
<thead>
<tr>
<th>Test Sample ID#</th>
<th>Condition</th>
<th>[Sr]</th>
<th>[MnO4]</th>
<th>NaOH</th>
<th>Sr</th>
<th>NaMnO4</th>
</tr>
</thead>
<tbody>
<tr>
<td>OP-01</td>
<td>filtered control</td>
<td></td>
<td></td>
<td>1.05</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>OP-02</td>
<td>repeat of PR-10</td>
<td>0.05M</td>
<td>0.03M</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>OP-03</td>
<td>low Sr and MnO4</td>
<td>0.05M</td>
<td>0.03M</td>
<td>1.05</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>OP-04</td>
<td>duplicate</td>
<td>0.05M</td>
<td>0.03M</td>
<td>1.05</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>OP-05</td>
<td>reverse addition</td>
<td>0.05M</td>
<td>0.03M</td>
<td>1.05</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>LH-06</td>
<td>filtered control</td>
<td>0.03 M</td>
<td>0.03M</td>
<td>0.55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LH-07</td>
<td>0.03 M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RW-08</td>
<td>filtered control</td>
<td>0.03M</td>
<td>0.03M</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RW-09</td>
<td>0.03M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RW-10</td>
<td>0.03M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RW-11</td>
<td>0.05 M</td>
<td>0.075M</td>
<td>0.05M</td>
<td>1.11</td>
<td>9.0</td>
<td>5.4</td>
</tr>
</tbody>
</table>

### Calculation

1. **Initial Volume**: 20 mL
2. **Initial Mass**: 20 * density
3. **ADD in mL**

---

**Note**: The table contains data related to the optimization of conditions in a laboratory experiment. The abbreviations used are: OP for optimization, LH for low hydroxide, RW for real waste, NaOH for sodium hydroxide, Sr for strontium, and MnO4 for manganese(VI) ion.
8.0 References


APPENDIX D
Appendix D: Analytical Data
Project: 29953
Client: S. Bryan

ACL Number(s): 99-1595 through 99-1606
Client ID: "MN-01" through "MN-12"
ASR Number: 5345
Total Samples: 12

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: J. J. Wagner
Analysis Date (Filename): 4-27-99 (A0525)

See system file: "ICP-325-405-1" for traceability to Calibration, Quality Control, Verification, and Raw Data.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Reviewed by

Jerry Wagner 5-3-99

Concur

5/3/99
Twelve radioactive liquid samples, MN-01 through MN-12, were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL) using PNNL-ALO-128 Acid Digestion procedure. One ml of aqueous sample (also weighed) was digested and diluted to a final volume of 25 ml. Additional dilution, up to 30 fold, was necessary to quantify Mn and Na. All measurement results reported have been corrected for preparation and analytical dilution. Analytes of interest (ASR 5345) include Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Ti, U, and Zn.

All quality control checks met MCS-033 QC tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis requirements under MCS-033.

**Five fold serial dilution:**
(Solid samples) --
(Aqueous samples) All results are within tolerance limit of ≤ 10% after correcting for dilution.

**Duplicate RPD (Relative Percent Difference):**
(Solid samples) --
(Aqueous samples) No duplicate samples were prepared due to limit sample volume available.

**Post-Spiked Samples (Group A):**
(Solid samples) --
(Aqueous samples) All analytes of interest were recovered within tolerance of 75 to 125%.

**Post-Spiked Samples (Group B):**
(Solid samples) --
(Aqueous samples) All analytes of interest were recovered within tolerance of 75 to 125%.

**Blank Spike:**
(Solid samples) --
(Aqueous samples) A blank spike was not prepared.

5/3/99
Matrix Spiked Sample:
(Solid samples) --
(Aqueous samples) A matrix spike was not prepared due to limit sample volume available.

Quality Control Check Standards:
Concentration of all analytes of interest, with one exception, was recovered within tolerance of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV.

Silicon in QC_SSTMCV check standard measured high (+14%) one time and only +4% a second time. However, all Si concentrations measured in QC_MCVA were within tolerance. The concentration of Si in both check standards is similar in concentration. Therefore, Si measurements throughout the analysis are assumed accurate.

High Calibration Standard Check:
Verification of the high-end calibration concentration for all analytes of interest was within tolerance of ± 5% accuracy, including Na at 1000 µg/ml.

Process Blank:
(Solid samples) --
(Aqueous samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except Silicon. The concentration of Silicon in all samples was about the same as that found in the process blank. Silicon contamination is probably due to labware (glass) used in transporting the original sample and glass digestion vessels used to prepare the samples.

Laboratory Control Standard:
(Solid samples) --
(Aqueous samples) LCS not prepared.
Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO₃ or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 μg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.
The attached report is sent for final distribution to the client.

Status these tests as sent to client -
ACL Numbers: (99-1595) (99-016060)
ASR Number: 5345
Tests: ICP Report - REVISED

File in Project File -
Project Number: 26953 or ED Work Order: ____________________
or __ ACL Waste File, or P.E. File: ____________________

Distribution -

<table>
<thead>
<tr>
<th>Send</th>
<th>S</th>
<th>B</th>
<th>R</th>
<th>U</th>
<th>Y</th>
<th>E</th>
<th>P</th>
<th>F</th>
<th>O</th>
<th>A</th>
<th>O</th>
<th>A</th>
<th>R</th>
<th>T</th>
<th>R</th>
</tr>
</thead>
</table>

Send To

Sam Bryan [✓]
Rich Hallen [✓]

___ Special distribution instructions are attached.

Project Manager -
Signature: __________________________ Date: ____________

Return copy of this coversheet to: __________________________

For LSO Use Only
Sent to client by: __________________________ Date: _______
### Battelle PNNL/RPG/Inorganic Analysis...

#### ICPAES Data Report

<table>
<thead>
<tr>
<th>Multiplier=</th>
<th>ALO#=</th>
<th>Client ID=</th>
<th>Process Blank #1</th>
<th>Det. Limit (ug/mL)</th>
<th>Run Date=</th>
<th>ug/g</th>
<th>ug/g</th>
<th>ug/g</th>
<th>ug/g</th>
<th>ug/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>25.0</td>
<td>99-1595-PB</td>
<td>[39x746]</td>
<td>19.7</td>
<td>4/27/99</td>
<td>0.015</td>
<td>Ag</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>99-1595</td>
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<td>19.7</td>
<td>4/27/99</td>
<td>0.060</td>
<td>Al</td>
<td>(7.2)</td>
<td>147</td>
<td>148</td>
<td>115</td>
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<td>19.7</td>
<td>4/27/99</td>
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<td>As</td>
<td>--</td>
<td>--</td>
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<td>--</td>
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<td>B</td>
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Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Data (1) from "A0525 S.Bryan ASR5345, W.Gray ASR5351, G.Lumetta (Rerun) ASR5319 ICP98 low.xls" 9/7/99 @ 1:55 PM
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## Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

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**Note:**
1. Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2. Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3. "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
# Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

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<th>Sample ID</th>
<th>Client Sample ID</th>
<th>Vial Identifier</th>
<th>Sample Volume (ml)</th>
<th>Final Solution Volume (ml)</th>
<th>Process Factor (1)</th>
<th>Vial Weight (g)</th>
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**Note:** A one ml sample was diluted to 25 ml. The final solution was taken to 25 ml in the spectrometer. These samples were processed on 2 blocks, therefore, there are two process blanks.

**Sample filled (yea/nay):** Y

**Process factor** = Final volume (ml)/Sample volume (ml)

**Other sample preparation worksheets may be substituted at the discretion of the cognizant scientist. Use one worksheet per client.**

**Analyst/Date:** Doe, J. S. 7-28-95

**Reviewer/Date:** Should be 99.

**Spice source:** NA

**PNL spike ID number:** A10-128

**Anal. balance M&TE:** 360-10-01-031
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Battelle Pacific Northwest Laboratory  
Radiochemical Processing Group-325 Building  
Radioanalytical Applications Team  
99-1595  
4/30/99

Client: S. Bryan

Cognizant Scientist: [Signature]  
Date: 4/30/99

Concur: [Signature]  
Date: 4/30/99

Measured Activities (uCi/g)

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<tr>
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</table>
Client: S. Bryan

Cognizant Scientist: [Signature]
Concur: [Signature]

Date: 4/30/99

Total Hydroxide Concentration - PNL-ALO-228

<table>
<thead>
<tr>
<th>ALO ID</th>
<th>Client ID</th>
<th>OH⁻ Molarity</th>
<th>+/- 1 σ</th>
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<tbody>
<tr>
<td>99-1596</td>
<td>Mn-02</td>
<td>0.9954</td>
<td>+/-0.084</td>
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<td>Cs-137 Error %</td>
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Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates

and

Operation of Brinkman 636 Auto-Titrator

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<th>Volume (mL)</th>
<th>Wt. (g)</th>
<th>Density g/mL</th>
<th>Initial pH</th>
<th>Titrant Vol. (mL)</th>
<th>Titrant pH</th>
<th>Found millimoles</th>
<th>Molarity base</th>
<th>RPD</th>
<th>Vol. (mL)</th>
<th>pH</th>
<th>Found millimoles</th>
<th>Molarity base</th>
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<tr>
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<td>0.3741</td>
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<td>1.369</td>
<td>7.803</td>
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<td>na</td>
<td></td>
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Performance checks

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<td>C40043</td>
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Prep record on 0.2034 M HCl is on following page.

ASR5345.xls 1 5/19/99
Preparation of Standardized 0.2 M HCl

Standardized 0.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 -- prep. date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole -- Barcode # 52232 --- (see below verification check).

The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.

Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029 M HCl (Chemrec_10) to 0.5 L with DI H2O.

20 mL aliquots of 0.2 M HCl were were neutralized to the phenolphthalein endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02 mL and the pipetting error is estimated to be < 1% @ 1s. Thus total error is < 3% for the measurements.

### NaOH Molarity verification

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<th>Wt. of KAP</th>
<th>Vol. of 0.1021 M NaOH to neutralize</th>
<th>NaOH Molarity = a *</th>
<th>Molarity Error +/- @ 1 s</th>
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<tbody>
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<td>0.80894</td>
<td>38.95</td>
<td>0.1017</td>
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<tr>
<td>2</td>
<td>0.80582</td>
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<td>3</td>
<td>0.96233</td>
<td>46.12</td>
<td>0.1022</td>
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<tr>
<td>Ave</td>
<td></td>
<td></td>
<td>0.1018</td>
<td>0.0003</td>
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</table>

re-certified value

### Titration Id.

<table>
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<tr>
<th>Titration Id.</th>
<th>aliquot of sample</th>
<th>Vol. of 0.1018 M NaOH to neutralize</th>
<th>Molarity of Acid in Sample</th>
<th>Molarity Error +/- @ 1 s</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>2</td>
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<td>39.92</td>
<td>0.2032</td>
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<td>3</td>
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<td>40.04</td>
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Ave Molarity HCl = 0.2034 ± 0.00042;

**Analyst/Date**

5-19-99
<table>
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<th>ROUTINE #</th>
<th>101</th>
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<table>
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<th>PH(INIT)</th>
<th>V(TE)/ML</th>
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<tbody>
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<td>3.798</td>
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**OH⁻ Alkalinity**

1st Run with 2M HCl

Titration Value

Stabilized Value 0.2034 M HCl

Reagent Blank Vol NA 10ml

Titrate 0.2034 M HCl

5/18/99

BRINKMANN
EA-1121A CAT # 2020015-1

DATE 18.05.99 NAME
Standardization Check

Sample = 5 ml of 0.1018 M NaOH

Titrant = 0.2054 M HCl

ROUTINE # 101
# 8 PH(INIT) 11.552 V(TE)/ML 3.905
1 V/ML 2.518 PH(M) 7.849
2 V/ML 2.556 PH(M) 4.545

DATE 18.05.99 NAME

0.25ML/DIV V(START)/ML 0.000 PH
Sample: 99-1596
Vol.: 300 mL
Titrant: 0.2034 M HCl

Routine # 101
#  PH(INIT)  V(TE)/ML  not real inflection point
 1  0.232  19.456
 2  1.463  7.793
 3  2.897  4.725

DATE 18.05.99  NAME
Sample: 0.9 g, 15.96 mL

Vol = 0.500 mL

\[ \text{Sample} \cdot \text{HCl} \]
Sample - 99-1596 Replicate

Vol - 0.300mL

Titrant = 0.2034 M HCl
Routine # 101
# 11 PH(INIT) 10.886 V(TE)/ML 5.000
1 V/ML 1.491 PH(M) 7.862
2 V/ML 2.977 PH(M) 4.697

Date 18.05.99 Name

0.25ML/DIV V(START)/ML 0.000 PH

Sample 99-15297

Vol = 0.300 ml

Titrant = 0.2034 ml HCl
ROUTINE  
# 12  PH(INIT) 10.049  V(TE)/ML  5.000
1 V/ML 1.374  PH(M)  7.815
2 V/ML 2.739  PH(M)  4.714

DATE 18.05.99 NAME
0.25ML/DIV V(START)/ML  0.000  PH
2 3 4 5 6 7 8 9 10 11 12

Sample = 99-1597 dup
Vol = 0.300 mL
Titrant = 0.2034 m HCl
ROUTINE # 101
# 13 PH(INIT) 10.854 V(TE)/ML 5.000
1 V/ML 1.362 PH(M) 7.803
2 V/ML 2.734 PH(M) 4.714

DATE 18.05.99 NAME

0.25ML/DIV V(START)/ML 0.000 PH

Process BL

Vol = 10mL

Titrat 0.2034M HCl.

PG 18-99
Project: 29953
Client: S. Bryan

**UPDATED REPORT**

ACL Number(s): 99-1856 through 99-1870

Client ID: "PR-01" through "PR-15"

ASR Number: 5392
Total Samples: 15

Procedure: PNL-A0-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: J. J. Wagner

Analysis Date (Filename): 06-03-99 (A0527) & 06-04-99 (A0528)

See system file: "ICP-325-405-1" for traceability to Calibration, Quality Control, Verification, and Raw Data.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Reviewed by

8-11-99

Concur

8/11/99
Thirteen radioactive liquid samples, PR-02 (ACL# 99-1857) through PR-14 (ACL# 99-1869), were analyzed by ICPAES after sample preparation using PNNL-ALO-128 Acid Digestion procedure. Typically five ml of aqueous sample (also weighed) was digested and diluted to a final volume of 25 ml.

Two radioactive slurry samples, PR-01 (ACL# 99-1856) and PR-15 (ACL# 99-1870) were initially weighed into glass vials by SAL in the hot cell and moved to SRPL for processing. Approximately 2.5g of sample PR-01 was digested and diluted to a final volume of 25ml. Two aliquots of PR-15 (centrifuged solids) were prepared in duplicate by removing approximately half of the sample from the original vial received from SAL (hot cell) using a spatula and washed off the spatula with water into a second glass vial. The amount of material transferred was determined by weighing the original sample container before and after the aliquot was removed. Each aliquot of PR-15 weighed about 0.25g and each was diluted to 25ml after digestion.

Measurement results reported are in μg/g for the slurry sample, centrifuged solids sample and the liquid samples. All results have been corrected for preparation and analytical dilution. Analytes of interest requested include: Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Ti, U, and Zn. Additional dilution, up to 30 fold, was sometimes necessary to quantify Na, Mn and/or Sr.

All quality control checks met MCS-033 QC tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis requirements under MCS-033.

**Five fold serial dilution:**
(Solid samples) All results are within tolerance limit of ≤ 10% after correcting for dilution.

(Aqueous samples) All results are within tolerance limit of ≤ 10% after correcting for dilution except for Ba, Ca, and Sr in samples PR-08, PR-11 and PR-13. The dilution corrected values for these analytes are biased low by 11% to 12% in the data report based upon this test. The lower value is likely caused by the very high concentration of sodium in the samples.

**Duplicate RPD (Relative Percent Difference):**
(Solid samples) All results are within tolerance limit of ≤ 20% RPD for analytes of interest except Sr (+30% RPD).

8/11/99
All results are within tolerance limit of ≤ 20% RPD for analytes of interest.

Post-Spiked Samples (Group A):
(Solid samples)  
(Aqueous samples) All analytes of interest tested were recovered within tolerance of 75% to 125% except Cr, K, Mo, and Pb in sample PR-14. Analyte recovery was low by 62%, 74%, 65% and 58% respectively. Sodium concentration in the sample was very high, approximately 118,000μg/g.

Post-Spiked Samples (Group B):
(Solid samples)  
(Aqueous samples) All analytes of interest tested were recovered within tolerance of 75% to 125%.

Blank Spike:
(Solid samples) All analytes of interest tested were recovered within tolerance of 80% to 120%.

(Aqueous samples) All analytes of interest tested were recovered within tolerance of 80% to 120%.

Matrix Spiked Sample:
(Solid samples) Matrix spike was not prepared due to limited sample material.

(Aqueous samples) All analytes of interest tested were recovered within tolerance of 80% to 120% except Ba and Pb in sample PR-08 (ACL# 99-1863). The low recovery may be due to relatively high concentration of sulfate in the sample resulting in precipitation of Ba and Pb.

Quality Control Check Standards:
Concentration of all analytes of interest, with two exceptions, was recovered within tolerance of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV.

Strontium in QC_MCVA check standard measured slightly high (+12%) one out of 5 measurements. Silicon in QC_SSTMCV standard
measured high by 19% in one of two measurements. This may have been caused by sample carry-over from a previous sample.

**High Calibration Standard Check:**
Verification of the high-end calibration concentration for all analytes of interest measured in QC_SST was within tolerance of ± 5% except for Fe and Ni which were slightly low in recovery (~6% each). This should not affect measurement results of Fe and Ni since only the very high end concentration is affected while actual sample concentration was much lower in concentration.

**Process Blank:**
(Solid samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except silicon. The concentration of silicon in the slurry samples was similar or slightly higher than that found in the process blank. Silicon contamination is probably due to labware (glass) used in transporting the sample material and use of glass digestion vessels to prepare and store the processed samples prior to analysis.

(Aqueous samples) All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except silicon. The concentration of silicon in the aqueous samples was below EQL and less than that found in the process blank. Silicon contamination is probably due to differences in leaching of the glass digestion vessels used to prepare the blank and samples.

**Laboratory Control Standard:**
(Solid samples) None prepared
(Aqueous samples) None prepared.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.
"Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO₃ or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

Absolute precision, bias and detection limits may be determined on each sample if required by the client.

The maximum number of significant figures for all ICP measurements is 2.
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<th>Multiplier</th>
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<th>Process Blank (129)</th>
<th>PR-01 (ALO-129)</th>
<th>Process Blank (129)</th>
<th>PR-15 (ALO-129)</th>
<th>PR-15 Dau (ALO-129)</th>
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Note: 1) Overall error greater than 10-times detection limit is estimated to be within ±15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicates measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
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Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
2) Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.  
3) "-" indicates measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
# Battelle PNNL/RPG/inorganic Analysis ... ICPAES Data Report

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Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "-" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
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Data (1) from 'A0528 S.Bryan ASR5392 ICP98 low.xls

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Note: 1) Overall error greater than 10-times detection limit is estimated to be within ± 15%.
2) Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.
3) "—" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
Client: Sam Bryan  
Concur:  

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Corrected Report

Measured Activities (μCi/g)

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RPD: 12%  
Matrix Spike: 98%  
Reagent Spike: 89%
Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

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<th>Weight (g)</th>
<th>Density (g/mL)</th>
<th>pH Reading</th>
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<th>Titrant pH</th>
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ASR-5392.xls
Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Equipment: WB76843

Lab Loc.: 525

### Titrant Molarity

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### CO3

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<th>RPD</th>
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<td>3.03</td>
<td>7.977</td>
<td>0.218</td>
<td>0.727</td>
<td>2.54%</td>
<td>4.762</td>
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<td>PR-12</td>
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<td>2.224</td>
<td>7.911</td>
<td>0.212</td>
<td>0.707</td>
<td>3.319</td>
<td>4.767</td>
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<td>PR-12 Replicate</td>
<td>0.300</td>
<td>2.214</td>
<td>7.899</td>
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<td>0.700</td>
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<td>4.759</td>
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<td>PR-14 Replicate</td>
<td>0.300</td>
<td>1.969</td>
<td>7.641</td>
<td>0.155</td>
<td>0.516</td>
<td>2.93%</td>
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### HCO3

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<th>Vol. (mL)</th>
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<th>Found</th>
<th>Molarity</th>
<th>millimole</th>
<th>RPD</th>
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<td>1.881</td>
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<td>PR-06</td>
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<tr>
<td>PR-12</td>
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<td>1.700</td>
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<td>94.5%</td>
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<td>PR-14</td>
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<td>7.801</td>
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<td>88.3%</td>
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Matrix spike recovery is calculated as follows:

\[
\text{Spike} = 2.00 \text{ mL} \times 0.1018 \text{ N NaOH added to the 0.100-mL of sample for each matrix spike.}
\]

\[
\text{Spike Titrant vol. (sample @ 1 mL + spike) - Sample Titrant vol. (average sample only equated to 1 mL) } \times 0.2034 \text{ N (HCl titrant)} = \text{ meq OH}
\]

\[
\text{meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added } \times 100 = \% \text{ recovered.}
\]

Prep record on 0.2034 M HCl is on following page.
Preparation of Standardized 0.2 M HCl

Standardized 0.1021 M NaOH will be re-checked and then used to standardize the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 – prep. date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KH2C8H404 (KAP) = 204.23 g/mole – Barcode # 52232 — (see below verification check).
The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.

Prepared 1 liter of ~0.2 M HCl by diluting 100 mL of 1.029 M HCl (Chemrec_10) to 0.5 L with DI. H2O.
20 mL aliquots of 0.2 M HCl were neutralized to the phenolphthalein endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02 mL and the pipetting error is estimated to be < 1% @ 1s. Thus total error is < 3% for the measurements.

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<th>Verification Test #</th>
<th>Wt. of KAP</th>
<th>Vol. of 0.1021 M NaOH to neutralize</th>
<th>NaOH Molarity = a * NaOH Molarity Error 1000 / b * 204.23 +/- 1s</th>
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<td>Ave=</td>
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Ave Molarity HCl = 0.2034 +/- 0.0004
Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: S. Bryan

ACL Number(s): 99-2102, 99-2104, 99-2108 through 99-2110, and 99-2112
Client ID: "OP-01", "OP-03", "LH-07" through "RW-09", and "RW-11"

ASR Number: 5426
Total Samples: 6

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).
Analyst: DR Sanders
Analysis Date (Filename): 07-21-99 (A0536)


M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Reviewed by

Concur
Six radioactive aqueous samples, OP-01 through RW-11 (ACL# 99-2102 through 99-2112), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 3ml of sample (weighed) was processed and diluted to a final volume of 15ml. All liquid samples were caustic, salt solutions prior to processing. An additional 0.6 ml of concentrated nitric acid was added to each sample beyond the normal amount of acid required by PNL-ALO-128 procedure because of the caustic in the samples. Samples were filtered after being diluted to 15 ml using 0.45 um filter because all of the samples contained a small amount of white to clear grainy material after processing.

All results reported are in µg/g including liquid samples and corrected for preparation and analytical dilution. Volumes and weights have been recorded on bench sheets (included with raw data, etc.). Analytes of interest include Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Ti, U, and Zn.

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous samples) All results were within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):
(Aqueous samples) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):
(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):
(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120%.

8/12/99
Matrix Spiked Sample:
(Aqueous samples) All analytes of interest in the matrix-spike were recovered within tolerance limit of 75% to 125% except for Ba and Pb. Matrix-spike recovery for barium in sample OP-03 was only about 7%. Lead recovery in the same sample was about 15%. Low recovery for these two analytes may be caused by moderately high concentration of sulfate in the sample. Post-spike recovery for all analytes of interest was recovered within tolerance limit of 75% to 125%.

Quality Control Check Standards:
Concentration of all analytes of interest was within tolerance limit of ±10% accuracy in the standards: QC_MCVA and QC_MCVB. Calibration Blank (ICP98.0) concentration was less than two times IDL.

High Calibration Standard Check:
Verification of the high-end calibration accuracy for all analytes of interest except potassium was within ±6% tolerance. The high-end calibration accuracy for potassium was slightly high, +5.5%. This will cause the potassium results to be slightly high by about the same amount since most of the potassium results in the samples were near the concentration test value.

Process Blank:
(Aqueous samples) All analytes of interest were within tolerance limit of ≤EQL or <5% of sample concentration except Si, which was equivalent to about 13% to 93% of that reported in the samples. The concentration of silicon reported in the samples were all below MRQ=170 μg/ml (or μg/g).

Laboratory Control Standard (LCS):
(Aqueous samples) No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%. See attached ICPAES data results.
 Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO₃ or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.
### Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

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**Note:**
1. Overall error greater than 10-times detection limit is estimated to be within ±15%.
2. Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3. *—* indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
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**Note:**
1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "-" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).
# Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

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<th>Sample</th>
<th>ACL Sample ID</th>
<th>ACL order number or Client sample ID</th>
<th>Mass (g)</th>
<th>Volume (ml)</th>
<th>Spike added</th>
<th>Final solution Volume (ml)</th>
<th>Process Factor (1)</th>
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**Analyst's sample preparation comments:** All samples were weighed prior to the digestion. See sample mass above! TCP spiking solution # 9903055k cp 114 added. All samples were run with 2.3 ml of equal per spiked sample. All of the samples were basic salt solutions that started with the addition of 0.4 ml conc. trace metal grade HCL and reacted further with the addition of 0.1 ml conc. trace metal grade HNO3. An additional 1 ml of concentrated 16% HNO3 was used and several samples were deep purple to verify acidity prior to the digestion.

**Spike source:** Standard solution

**PNL spike ID number:** See Notes

**Anal. balance M&TE:** 360.00 ± 0.05

**Sample filtered (yes/no):** Yes

**Other sample preparation worksheets may be substituted at the direction of the Cognizant Scientist. Use one worksheet per client.**

---

**Analyst/Date:** Lori P. Darrell 7/9/99  
**Reviewer/Date:**

---

**Rev. 2.0 7-28-05 JMR**
Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

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<th>Sample Volume (ml)</th>
<th>Spike added</th>
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Analyst's sample preparation comments: Upon completion of the digestion, the samples were diluted to 15 ml and allowed to sit overnight.  All samples #97-2102, #99-2102, #99-2007, #99-2009, #99-3111, and #99-3112 all contained a small amount of white to clear gummy material.  All samples were filtered to 0.45 mm.

Spiked source: See pg 1.

PNL spike ID number: See pg 1.

Anal. balance M&TE: See pg 1.

Sample filter (yes/no): 

(1) Process factor = Final volume (ml) / Sample volume (ml)

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: Lori P. Dardell 7-8-99
Reviewer/Date: ____________________________

Rev. 2: 8-95 JMR
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<td>2.68E-2</td>
<td>2.28E-4</td>
<td>4.80E-5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Matrix Spike | 87% | 67% |
Blank Spike  | 95% | 95% |
Blank       | <4.E-5 | <2.E-6 | <2.E-7 | <2.E-7 |
Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

<table>
<thead>
<tr>
<th>Titrant</th>
<th>Molarity</th>
<th>Std. &amp; Spike</th>
<th>Molarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl</td>
<td>0.2034</td>
<td>NaOH</td>
<td>0.1018</td>
</tr>
</tbody>
</table>

### Titrator Routine

<table>
<thead>
<tr>
<th>RPG #</th>
<th>Sample ID</th>
<th>Sample Vol. (mL)</th>
<th>Sample Wt. (g)</th>
<th>Density g/mL</th>
<th>#</th>
<th>Initial pH reading</th>
<th>1st Equivalence Point Titrant Vol. (mL)</th>
<th>pH</th>
<th>Titrant Molarity</th>
<th>pH Molarity</th>
<th>millimole base</th>
<th>Molarity</th>
<th>millimole RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>99-2108</td>
<td>LH-07</td>
<td>0.050</td>
<td>0.0658</td>
<td>1.316</td>
<td>4</td>
<td>10.982</td>
<td>0.127</td>
<td>10.354</td>
<td>0.026</td>
<td>0.52</td>
<td>4.43%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2108</td>
<td>LH-07</td>
<td>0.200</td>
<td>0.2676</td>
<td>1.338</td>
<td>5</td>
<td>11.427</td>
<td>0.486</td>
<td>10.637</td>
<td>0.099</td>
<td>0.49</td>
<td>4.43%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2109</td>
<td>RW-08</td>
<td>0.100</td>
<td>0.1434</td>
<td>1.434</td>
<td>6</td>
<td>11.344</td>
<td>0.345</td>
<td>10.717</td>
<td>0.070</td>
<td>0.70</td>
<td>0.29%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2109</td>
<td>RW-08</td>
<td>0.100</td>
<td>0.1401</td>
<td>1.401</td>
<td>7</td>
<td>11.419</td>
<td>0.344</td>
<td>10.705</td>
<td>0.070</td>
<td>0.70</td>
<td>0.29%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2110</td>
<td>RW-09</td>
<td>0.100</td>
<td>0.1421</td>
<td>1.421</td>
<td>8</td>
<td>11.505</td>
<td>0.294</td>
<td>10.848</td>
<td>0.060</td>
<td>0.60</td>
<td>0.34%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2110</td>
<td>RW-09</td>
<td>0.100</td>
<td>0.1396</td>
<td>1.396</td>
<td>9</td>
<td>11.459</td>
<td>0.295</td>
<td>10.809</td>
<td>0.060</td>
<td>0.60</td>
<td>0.34%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2112</td>
<td>RW-11</td>
<td>0.100</td>
<td>0.1417</td>
<td>1.417</td>
<td>10</td>
<td>11.456</td>
<td>0.310</td>
<td>10.716</td>
<td>0.063</td>
<td>0.63</td>
<td>0.34%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2112</td>
<td>RW-11</td>
<td>0.100</td>
<td>0.1337</td>
<td>1.337</td>
<td>11</td>
<td>11.500</td>
<td>0.307</td>
<td>10.679</td>
<td>0.063</td>
<td>0.63</td>
<td>0.34%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**QC Data:**

- Reag. Blk.: 5.00
- OH % Recovery, Accur
- 103.9% standard
- 98.6% standard
- 93.7% matrix spk
- 91.7% matrix spk
- 90.8% matrix spk
- 81.8% matrix spk

* -- Volume restrictions existed

### Performance checks

<table>
<thead>
<tr>
<th>Buffer</th>
<th>VWR Lot #</th>
<th>CMS#</th>
<th>Expire Date</th>
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<td>10</td>
<td>981659-24</td>
<td>144109</td>
<td>Jul-00</td>
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<tr>
<td>4</td>
<td>981563-24</td>
<td>144107</td>
<td>Jun-00</td>
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<tr>
<td>7</td>
<td>981894-24</td>
<td>144108</td>
<td>Aug-00</td>
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Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates

and

Operation of Brinkman 636 Auto-Titrator

| Equip # WB76843 |
| Lab Loc. 525 |

<table>
<thead>
<tr>
<th>Titrant</th>
<th>Molarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl</td>
<td>0.2034</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>RPG #</th>
<th>Sample ID</th>
<th>Sample Vol. (mL)</th>
<th>CO3 2nd Equivalence Point Titrant Vol. (mL)</th>
<th>pH</th>
<th>Found millimoles</th>
<th>Molarity base</th>
<th>millimole</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>99-2108</td>
<td>LH-07</td>
<td>0</td>
<td>0.322</td>
<td>7.660</td>
<td>0.040</td>
<td>0.793</td>
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<td></td>
</tr>
<tr>
<td>99-2108</td>
<td>LH-07</td>
<td>Replicate</td>
<td>0.200</td>
<td>7.814</td>
<td>0.164</td>
<td>0.820</td>
<td>3.28%</td>
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</tr>
<tr>
<td>99-2109</td>
<td>RW-08</td>
<td>0</td>
<td>1.290</td>
<td>7.459</td>
<td>0.192</td>
<td>1.922</td>
<td></td>
<td></td>
</tr>
<tr>
<td>99-2109</td>
<td>RW-08</td>
<td>Replicate</td>
<td>0.100</td>
<td>1.253</td>
<td>0.185</td>
<td>1.849</td>
<td>3.88%</td>
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</tr>
<tr>
<td>99-2110</td>
<td>RW-09</td>
<td>0</td>
<td>1.150</td>
<td>7.691</td>
<td>0.174</td>
<td>1.741</td>
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<tr>
<td>99-2110</td>
<td>RW-09</td>
<td>Replicate</td>
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<td>1.153</td>
<td>0.175</td>
<td>1.745</td>
<td>0.23%</td>
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<tr>
<td>99-2112</td>
<td>RW-11</td>
<td>0</td>
<td>1.077</td>
<td>7.601</td>
<td>0.156</td>
<td>1.560</td>
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<tr>
<td>99-2112</td>
<td>RW-11</td>
<td>Replicate</td>
<td>0.100</td>
<td>1.077</td>
<td>0.142</td>
<td>1.424</td>
<td>9.13%</td>
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</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>HCO3 3rd Equivalence Point Titrant Vol. (mL)</th>
<th>pH</th>
<th>Found millimoles</th>
<th>Molarity base</th>
<th>millimole</th>
<th>RPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.556</td>
<td>4.293</td>
<td>0.048</td>
<td>0.95</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.160</td>
<td>4.616</td>
<td>0.177</td>
<td>0.88</td>
<td>7.54%</td>
<td></td>
</tr>
<tr>
<td>2.066</td>
<td>4.704</td>
<td>0.158</td>
<td>1.58</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.028</td>
<td>4.664</td>
<td>0.158</td>
<td>1.58</td>
<td>0.1%</td>
<td></td>
</tr>
<tr>
<td>1.857</td>
<td>4.770</td>
<td>0.144</td>
<td>1.44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.708</td>
<td>4.808</td>
<td>0.128</td>
<td>1.28</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.639</td>
<td>4.681</td>
<td>0.129</td>
<td>1.29</td>
<td>0.16%</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CO3 % Recovered</th>
<th>HCO3 % recovered</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard 1 0.1018 N NaOH</td>
<td>5.000</td>
</tr>
<tr>
<td>Standard 2 0.1018 N NaOH</td>
<td>5.000</td>
</tr>
<tr>
<td>99-2108MS LH-07 + 2mL 0.1N NaOH</td>
<td>0.100</td>
</tr>
<tr>
<td>99-2109MS RW-08 + 2mL 0.1N NaOH</td>
<td>0.100</td>
</tr>
<tr>
<td>99-2110MS RW-09 + 2mL 0.1N NaOH</td>
<td>0.100</td>
</tr>
<tr>
<td>99-2112MS RW-11 + 2mL 0.1N NaOH</td>
<td>0.100</td>
</tr>
</tbody>
</table>

Matrix spike recovery is calculated as follows:

\[
\text{Spike} = 2.00 \text{ mL} \times 0.1018 \text{ N NaOH was added to the } 0.100-\text{mL of sample for each matrix spike.}
\]

\[
\text{Spike Titrant vol. (sample @ .1mL + spike)} - \text{Sample Titrant vol. (average sample only equated to .1mL)} \times 0.2034 \text{ N (HCl titrant)} = \text{meq OH}
\]

\[
\text{meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added} \times 100 = \% \text{ recovered.}
\]

Prep record on 0.2034 M HCl is on following page.
Preparation of Standardized 0.2 M HCl

Standardized 0.1021 M NaOH will be re-checked and then used to standardize the ~0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37—prep.date 2-25-98 for original data) and re-verified against NIST SRM844 Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole — Barcode # 52232 — (see below verification check).

The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.

Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H2O.

20 mL aliquots of 0.2 M HCl were neutralized to the phenolphthalein endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipetting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity verification

<table>
<thead>
<tr>
<th>Verification Test #</th>
<th>Wt. of KAP</th>
<th>Vol. of 0.1021M NaOH to neutralize</th>
<th>NaOH Molarity = a * 1000 / b * 204.23</th>
<th>Molality Error +/- @ 1s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.80894</td>
<td>38.95</td>
<td>0.1017</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.80582</td>
<td>38.84</td>
<td>0.1016</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.96233</td>
<td>46.12</td>
<td>0.1022</td>
<td></td>
</tr>
<tr>
<td>Ave</td>
<td></td>
<td>[re-certified value] 0.1018</td>
<td>0.0003</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Titration Id.</th>
<th>aliquot of sample</th>
<th>Vol. of 0.1018M NaOH to neutralize</th>
<th>Molarity of Acid in Sample</th>
<th>Molality Error +/- @ 1s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.00</td>
<td>39.88</td>
<td>0.2030</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>20.00</td>
<td>39.92</td>
<td>0.2032</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>20.00</td>
<td>40.04</td>
<td>0.2038</td>
<td></td>
</tr>
<tr>
<td>Ave Molarity HCl = 0.2034</td>
<td>0.00042</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Appendix E: Calculations
The MN series were the first run. It was assumed that the archived AN-107 had 5M sodium and 0.2M OH (report said .24 target but only got 0.126).

The waste was titrated and found to have no free hydroxide. So only Mn-7 and 8 had any free hydroxide.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>waste vol</th>
<th>waste wt</th>
<th>OH vol</th>
<th>MnO4 wt</th>
<th>MnO4 v</th>
<th>Ca wt</th>
<th>Ca v</th>
<th>Sr/Eu v</th>
<th>Sr/Eu v</th>
<th>Final Ma</th>
<th>Mass correction</th>
<th>final vol</th>
<th>v</th>
<th>density</th>
<th>predicted ICP, M</th>
<th>ICP(g/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Mn-01</td>
<td>24.85</td>
<td>25.14</td>
<td>19.85</td>
<td>0</td>
<td>1.88</td>
<td>1.86</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>27.028</td>
<td>1.0748</td>
<td>21.7726</td>
<td>1.0907</td>
<td>1.37</td>
<td>5.00</td>
<td>115</td>
</tr>
<tr>
<td>2 Mn-02</td>
<td>25.02</td>
<td>25.16</td>
<td>19.97</td>
<td>0</td>
<td>1.87</td>
<td>1.84</td>
<td>0</td>
<td>0</td>
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<td>27.049</td>
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<td>21.7806</td>
<td>1.0902</td>
<td>1.2884</td>
<td>5.00</td>
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</tr>
<tr>
<td>3 Mn-03</td>
<td>25.07</td>
<td>25.16</td>
<td>19.97</td>
<td>0</td>
<td>1.83</td>
<td>1.81</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>26.959</td>
<td>1.0464</td>
<td>21.6225</td>
<td>1.0543</td>
<td>1.3194</td>
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<tr>
<td>4 Mn-04</td>
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<td>19.97</td>
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<td>2.59</td>
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<td>1.2331</td>
<td>4.33</td>
<td>98.7</td>
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<td>25.16</td>
<td>19.97</td>
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<td>3.52</td>
<td>3.45</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>29.477</td>
<td>1.1955</td>
<td>23.5942</td>
<td>1.2058</td>
<td>1.2552</td>
<td>5.09</td>
<td>118</td>
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<tr>
<td>6 Mn-06</td>
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<td>19.97</td>
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<td>0</td>
<td>0</td>
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<td>25.3841</td>
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<td>1.2377</td>
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<td>109</td>
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<td>25.07</td>
<td>25.16</td>
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<td>0</td>
<td>0</td>
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<td>1.2283</td>
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<td>98.3</td>
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<td>0</td>
<td>0</td>
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<td>1.1454</td>
<td>1.2273</td>
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<td>9 Mn-09</td>
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<td>25.16</td>
<td>19.97</td>
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<td>7.12</td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>26.167</td>
<td>1.1249</td>
<td>21.8581</td>
<td>1.1454</td>
<td>1.2273</td>
<td>4.37</td>
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</tr>
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<td>10 Mn-10</td>
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<td>19.97</td>
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<td>7.95</td>
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<td>0</td>
<td>0</td>
<td>27.916</td>
<td>1.1249</td>
<td>22.8581</td>
<td>1.1454</td>
<td>1.2273</td>
<td>4.37</td>
<td>102</td>
</tr>
<tr>
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<td>19.97</td>
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<td>8.95</td>
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<td>0</td>
<td>29.477</td>
<td>1.1955</td>
<td>23.5942</td>
<td>1.2058</td>
<td>1.2552</td>
<td>5.09</td>
<td>118</td>
</tr>
</tbody>
</table>

Correlation of Predict and ICP Sodium

ICP Na vs Predicted Na
For the PR series of experiments, the target [OH] concentration was 1 M. So all of the waste was caustic adjusted except for PR-14 which first had acid added.

From TI-040, 382,133 g of C3E3, d = 1.26 g/mL and 24.12 g of 50% NaOH (d = 1.5 g/mL) was added. Final density of 1.263 g/mL.

<table>
<thead>
<tr>
<th>test #</th>
<th>Vial ID</th>
<th>Vial tare wt, g</th>
<th>target wt, mL</th>
<th>gross wt, mL</th>
<th>net wt of addition, mL</th>
<th>target volume, mL</th>
<th>gross vol, mL</th>
<th>net vol of addition, mL</th>
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<th>Ca(NO3)2 density, g/mL = 1.1169</th>
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2.5839 grams

In addition to reagents PR-14 had HN03 added

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The third series or Phase 3 experiments were to optimize treatment, and see if conditions from PR-10, minimal reagent addition work. However, the client also asked that we change the treatment scheme. Add Sr first, cook for 2 hours, then add MnO4 and cook for additional 2 hours.

Four different wastes used. OP-01, OP-03 to OP-05 all used C3E4 with 1M OH and 1.5M NaNO3. OP-02 was a repeat of PR-10 and used C3E3 caustic adjusted to 1M, same as PR-01. LH-06 and LH-07 used C3E4 with 0.5M NaOH and 1.5M NaNO3. RW-08 to RW-11 used combined CL-1 and CL-2, AN-107 diluted feed from Lumetta's studies.

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<td>Richard Hallen</td>
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<tr>
<td>Sam Bryan</td>
<td>Scientist/Hot Cell Experiments - lead and direct hot cell experiments</td>
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<td>Vaughn Hoopes</td>
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