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Assessment of Sr/TRU Removal Mechanisms Using AN-102 and AN-107 Tank Waste Samples

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Test Specification: 24590-WTP-TSP-RT-02-013
Test Plan: TP-RPP-WTP-191
Test Exceptions: None
R&T Focus Area: Pretreatment
Testing Scoping Statement: B-38

Battelle—Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification 24590-WTP-TSP-RT-02-013 and Test Plan TP-RPP-WTP-191. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

_____ _____

Gordon Beeman, Manager Date
WTP R&T Support Project

Summary

The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline for pretreating Envelope C low-activity waste (LAW) at Hanford includes a precipitation step for removing radioactive strontium (Sr-90) and transuranic (TRU) isotopes before the waste is vitrified. The current design basis for the Sr/TRU removal process is the addition of strontium nitrate (0.075M), for isotopic dilution and Sr-90 precipitation as $SrCO_3$, and sodium permanganate (0.05M), for precipitation of the TRU elements, at 50°C and 1M additional sodium hydroxide. Section 5 of the *Research and Technology Plan*, prepared by Bechtel National, Inc., identifies further research needs. One need shown is to determine the mechanism of the Sr/TRU precipitation process (SOW Ref.: Sec. C.6 Std.2(a)(3)(ii)(B) and WBS No.: 1.2.10.01 and .02). Reaction mechanism assessment for the Sr/TRU precipitation process is addressed in Scoping Statement B-38, which is included in Appendix C of the *Research and Technology Plan*. In accordance with Scoping Statement B-38, Test Specification 24590-WTP-TSP-RT-02-013, and Test Plan TP-RPP-WTP-191, studies were conducted with actual tank waste samples to develop a better understanding of the TRU decontamination mechanisms.

Objectives

This report discusses investigations into the mechanism of the Sr/permanganate treatment process for removing Sr-90 and TRU from tank supernatant destined for immobilization as LAW. Experiments were conducted with actual waste samples from Envelope C tanks, AN-102 and AN-107. The purpose of these mechanistic studies was to determine the role of permanganate reactions in TRU decontamination. Of specific interest is the importance of various mechanisms, such as oxidation, absorption, precipitation, and ligand displacement, on TRU decontamination. Past studies, supported by additional results from these investigations, have demonstrated the effectiveness of Sr-90 removal by isotopic dilution and precipitation as $SrCO_3$ by added nonradioactive $Sr(NO_3)_2$.

Previous mechanistic investigations were conducted with waste simulant solutions. The results of the simulant tests were used to define experiments for actual waste testing. The results from the actual waste tests are provided in this report and support observations reported from the simulant studies. This is important because the earlier conclusions were based on the removal of surrogate elements, not actual TRU components. Although the chemistry is expected to be similar for the waste simulant solutions, the exact composition of organics and complexants in the actual waste is not fully known or understood. Consequently, tests with actual wastes provide further support for conclusions and recommendations based on simulant results.

Conduct of Testing

Small-scale radioactive tests (~20 mL) were conducted with tank waste samples from both AN-102 and AN-107. A series of tests with each tank waste were conducted approximately 1 month apart. Not all conditions tested were identical for each set of experiments. In all but one experiment (permanganate-only addition), $Sr(NO_3)_2$ was added first for Sr-90 precipitation. The strontium addition was followed by

the addition of a reagent targeted at TRU removal. In general, the experiments examined TRU removal by the impact of the Mn oxidation state [Mn(VII), Mn(VI), Mn(IV) as solid, and Mn(II)]; oxidation by a nonprecipitating, non-Mn-containing, reagent (periodate); and a complexant-competing, highly-charged cation, Zr(IV).

The decontamination of Sr-90 was examined as a function of treatment chemistry. The decontamination factors (DFs) were high (>10) for all experiments [except when no $Sr(NO_3)_2$ was added, Mn(VII) only], and for the Sr/periodate experiment with the AN-102 sample. In general, both wastes gave quite similar results for Sr-90 removal. The most notable differences between the two wastes were for the no-mix experiment and the Sr/periodate treatment. Examination of the [Sr] data shows that this is a direct result of the differences in Sr solubility for these treated samples. All other treatment schemes resulted in an overall decreased [Sr]. The results are consistent with the Sr-90 DF resulting from isotopic dilution and dependent on the final Sr concentration (amount of Sr that precipitates from solution).

The TRU removal is represented by the DF for the sum of alpha emitters. All of the reagents tested gave some level of TRU removal. For AN-102 waste, permanganate generally gave the highest DF, the exception being the high TRU DF for periodate addition. The results for Zr(IV) addition suggest that ligand exchange and precipitation alone do not result in as high TRU removal as when oxidation also occurs (when treated with permanganate). This result, combined with the results from different Mn oxidation states, suggests oxidation is important for TRU decontamination in AN-102. The results suggest that the TRU removal process is most effective for removing the tri-valent actinide ions, Am and Cm. These TRU elements make up approximately 95% of the TRU in the Envelope C wastes.

Ligand exchange, precipitation, and sorption appear to be most important for TRU removal from AN-107. Addition of reduced oxidation states of Mn [Mn(VI) and Mn(II)] was just as effective as permanganate. For AN-107, oxidation-only (periodate) resulted in significant TRU removal (75%), but experiments with no oxidation, Mn(II) and Zr(IV), gave higher TRU removal (>80%). Oxidation was not as important for TRU removal in AN-107 as with AN-102 treatment. When all of the data are examined, including the Sr data discussed above, it is concluded that AN-102 has significantly more EDTA/HEDTA-type complexants than AN-107.

From the simulant results, it was concluded that experiments involving an oxidant generally had higher levels of surrogate TRU element (lanthanides) removal than those in which only precipitation or absorption occurred. Some differences were noted between the simulant and actual waste data. In simulant studies, ligand displacement appeared to be important for Zr(IV), which resulted in similar removal as the permanganate treatment for most of the surrogate TRU elements. The concentration of Zr in the simulant samples (4h and 24h) was also very high. Presumably, Zr(IV) competed well for ligands while remaining soluble, and displaced other metal ions that then precipitated in the basic solution.

The results of the simulant no-mixing tests (1h after reagent addition) were confirmed with actual waste samples. Good mixing during the Mn reduction reactions was not as important as earlier expected, as long as the resulting precipitate was well mixed with the supernatant before the sample was filtered. This was expected for Sr-90 removal (isotopic exchange still occurs with the $SrCO_3$ precipitate), but surprising for permanganate treatment, since a large fraction of the waste was not directly contacted/oxidized with Mn(VII). However, the addition of preformed Mn(IV) solids to AN-102 waste was not as successful;

thus, it is important for precipitation to occur in the waste. The better removal by Mn solids formed in the waste is likely a result of a more active form of Mn precipitate. Results suggest that the precipitate contains substantial quantities of more reduced Mn (III and possibly II).

The time dependence of Sr-90 and TRU removal was examined for selected experiments. The TRU was found to be independent of time, similar to earlier reported bench-scale tests. The greatest time dependence (between the 4h and 24h samples) is noted for the Sr-90 DF, and is directly related to the corresponding [Sr]. This was the same result as observed in the simulant tests. The soluble Sr was nearly twice as high at 4h as 24h, which translates to a near twofold increase in the Sr-90 DF between the 4h and 24h samples. The overall Sr-90 DF is determined by the amount of isotopic dilution and final Sr concentration. Increasing the Sr-90 DF by waiting 24h to filter would have the same effect as doubling the initial nonradioactive Sr addition if filtration were to occur at 4h. These results suggest that crossflow filtration to remove solids should not begin until more than 4h after reagent addition.

Results and Performance Against Objectives

The objective of this work was to demonstrate that the Sr/TRU removal process provides adequate decontamination of Envelope C waste to meet the contract requirements for immobilized low-activity waste (ILAW). The initial waste from Tank AN-107 was higher in both Sr-90 and TRU compared to waste from Tank AN-102. The experimental data from both wastes can be used to predict the loading of Sr-90 and TRU (sum of alpha) expected in the ILAW at a fixed waste sodium loading of 15 wt% (contract limit >10 wt%). At 24h of reaction, the levels of Sr-90 and TRU in the supernatant from treatment of both wastes were quite similar. For Sr-90 loading, the levels were three times below the ILAW requirements of 20 Ci/m^3 and for TRU loading, the levels were four times below the requirements of 100 nCi/g when treated by addition of nonradioactive Sr and sodium permanganate at reagent levels of 0.02M. These reagent levels are significantly below the baseline treatment conditions of 0.075M Sr and 0.05M permanganate.

Quality Requirements

Testing began in September 2002 and continued through January 2003 to assess the reaction mechanisms of Sr/TRU removal by added $Sr(NO_3)_2$ and permanganate. Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements by performing work in accordance with the River Protection Program-Waste Treatment Plant Technical Support Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with Implementing Procedure QA-RPP-WTP-604 contained in the Waste Treatment Plant Support Project Quality Assurance and Description (WTPSP) Quality Assurance Manual. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the test plan objectives.

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1.0 Introduction

This report summarizes work performed by Battelle—Pacific Northwest Division (PNWD) in support of the River Protection Project-Waste Treatment Plant (RPP-WTP) at Hanford. Before the liquid (supernatant) fraction of Envelope C[(a)] wastes (Tanks AN-107 and AN-102) can be disposed of as low-activity waste (LAW), pretreatment is required to remove radioactive strontium (Sr-90) and transuranic (TRU) elements in addition to Cs-137 and the entrained solids. The Sr-90 removal process consists of isotopic dilution by nonradioactive $Sr(NO_3)_2$ addition and precipitation of $SrCO_3$. The TRU removal process involves addition of permanganate, stepwise manganese reduction, Mn(VII) to Mn(VI) to Mn(IV); precipitation of MnO_2; and concomitant TRU precipitation. This TRU decontamination method is based on work conducted at Pacific Northwest National Laboratory by Orth et al. (1995). Entrained solids and Sr/TRU precipitate are to be removed via crossflow filtration; Cs-137 is to be removed by ion exchange. In previous work for the RPP-WTP contractor, PNWD and the Savannah River Technology Center demonstrated Sr/TRU removal with actual waste samples from Envelope C tanks (Hallen et al. 2000a,b; Hallen et al. 2002a,b,c; Nash et al. 2000a,b) by added nonradioactive Sr and permanganate.

Optimized treatment conditions were identified in small-scale tests (20 mL) with AN-102 waste samples (Hallen et al. 2002a). Hallen et al. (2002b,c) conducted additional small-scale and bench-scale tests with a waste blend consisting of AN-102 waste and C-104 high-level waste (HLW) pretreatment streams. This additional testing verified that the optimized process conditions, which minimized reagent addition (0.02M) and reduced the process temperature to ambient ($25 \pm 5°C$), provided adequate Sr-90 and TRU removal to meet immobilized low-activity waste (ILAW) requirements. Results from the bench-scale test established the mechanism of Sr-90 removal (Hallen et al. 2002c). Before Sr addition, the waste was undersaturated with Sr. Sampling the reaction mixture 18 min after $Sr(NO_3)_2$ addition showed the total Sr concentration was near 200 μg/g and that isotopic dilution of Sr-90 in the supernatant was complete. With increased reaction time, the Sr-90 decontamination factor (DF) continued to increase as a result of decreased total Sr concentration. A decrease in temperature resulted in increased total Sr concentration, which decreased the Sr-90 DF (Hallen et al. 2002a).

The primary mechanism for Sr-90 removal was isotopic dilution by added nonradioactive $Sr(NO_3)_2$ and $SrCO_3$ precipitation. The addition of permanganate increased the Sr-90 decontamination, likely a result of oxidation of the chelating agents and precipitation of additional $SrCO_3$. The Sr-90 DFs increased significantly with time. However, this increased Sr-90 decontamination was not a result of increased isotopic exchange or ligand oxidation, but, rather, continued precipitation, i.e., reduction of total soluble Sr concentration. Isotopic exchange was found to be complete 18 min after reagent addition was complete (Hallen 2002c). Therefore, the kinetics of the Sr decontamination reaction were shown to be important; more than 4h was required to approach the final concentration. When the sample was cooled to 22-25°C for filtration testing, the Sr concentration increased (Sr-90 DF decreased) as a result of the retrograde solubility of $SrCO_3$ (Felmy and Mason 2003). Therefore, in addition to isotopic dilution, the other important factor in decontamination is the total Sr concentration (distribution of Sr between the solution and solid phases), which is a function of the carbonate concentration, complexant concentration,

(a) Envelope designations are explained in DOE (2000).

temperature, and time. Both AN-102 and AN-107 have such high levels of carbonate (>0.5M) that the carbonate concentration has little impact on $SrCO_3$ solubility (Felmy and Mason 2003).

The TRU removal from AN-102 diluted waste (Hallen et al. 2002a) and the AN-102/C-104 waste blend (Hallen et al. 2002c) was consistent. The TRU decontamination in the AN-102/C-104 waste blend occurred after the permanganate was added (Hallen et al. 2002b,c). The TRU removal exceeded the requirements for ILAW glass by a factor of 5. This result suggested that blending had no impact on TRU removal. The initial concentrations of TRU elements were also significantly decreased with the waste blending, such that the waste without treatment was below the ILAW levels.

Previous work has shown that permanganate addition to waste results in reduction to a variety of Mn species (Gauger and Hallen 2001). Oxidation of formate and organics in the waste reduces Mn(VII) to Mn(VI), then to Mn(IV) and likely lower Mn oxidation states. Depending on the organic compounds present, soluble Mn(IV) complexes can form, but eventually the Mn precipitates from solution. Further investigations of the Mn reaction chemistry relevant to waste processing were performed with an AN-102/C-104 waste blend simulant (Lilga et al. 2003). The purpose of those mechanistic studies was to determine the importance of oxidation, absorption, precipitation, and ligand displacement on decontamination. The results showed that permanganate is the preferred reagent because all potential mechanisms for TRU removal are operative: oxidation, absorption, precipitation, and ligand displacement. Results from the simulant studies were used to define the conditions for the active (actual) waste tests described in this report.

The objective of the work reported here was to repeat the earlier reaction mechanism experiments (Lilga et al. 2003), using actual waste samples, and determine if similar reaction mechanisms are important. The experiments discussed in this report were performed in radioactive hot cells using approximately 20-mL samples of waste with various amounts of added reagents. Samples of both AN-102 and AN-107 were available for the actual waste tests so potential differences between the two Envelope C wastes could be identified. Earlier results (Hallen et al. 2000a,b and Nash et al. 2000a,b) suggested that AN-107 may be more difficult to treat and may require higher levels of reagent addition to meet the ILAW requirements.

The results from reaction mechanism tests with actual waste samples from AN-102 and AN-107 are presented in this report. Test conditions and experimental procedures are described in Section 2.0. Results from the tests are discussed in Section 3.0. The major conclusions and recommendations are given in Section 4.0. The appendices include the quantities of samples and reagents used for each test matrix, and provide all of the analytical data.

2.0 Test Conditions and Experimental Procedures

Experiments probing the mechanism of Sr-90 and TRU removal used actual samples of AN-102 and AN-107 waste that had been shipped to PNWD for integrated process testing. The waste samples, test conditions, experimental procedures, and chemical analyses are described below. Additional details are provided in Appendices A and B.

2.1 Description of Waste Samples

PNWD received two bottles of AN-102 tank waste from Hanford's 222-S Laboratory. The material was originally collected by grab sampling of AN-102 from riser 022 over the period August 7 through 11, 2000, and shipped to the 222-S Laboratory in Hanford's 200 West Area. The sample material was transferred to 125-mL bottles that were shipped to the Radiochemical Processing Laboratory (RPL) in the 300 Area, where they were inspected on receipt. The two bottles used for testing in this study contained a settled layer of light brown solids, with a dark brownish/black standing liquid. The samples were assumed to be similar in composition to the earlier AN-102 samples characterized by Urie et al. (2002). Analyses determined the AN-102 samples contained approximately 0.25M free hydroxide, 1M carbonate (total inorganic carbon, TIC), and 2M total organic carbon (TOC). The samples were diluted with an appropriate amount of 0.01M NaOH to give a diluted feed of approximately 5.5M sodium prior to reagent addition. Free OH^- in the diluted samples was determined, by titration, to be 0.14M.

A 500-mL bottle of AN-107 diluted feed (designated AN-107 UFC, Urie et al. 1999a) had been retained for future testing during the bench-scale testing with AN-107 (Hallen et al. 2000b). Urie et al. (1999a) had prepared the diluted feed to a target of 7.7M sodium and 1.1M added hydroxide. AN-107 was hydroxide-deficient when originally received (Urie et al. 1999b), but the diluted feed preparation, addition of 1.1M hydroxide, resulted in a free hydroxide content of 0.7M. The caustic adjustment resulted in 86.4% of the total sodium as waste sodium. The carbonate (TIC) and TOC concentrations were determined to be 1.4M and 2.5M, respectively. Since the waste had been stored in the hot cell and aged for approximately 4 years, samples were taken and reanalyzed before the experiments were begun. The sodium concentration was determined to be 8.6M by inductively coupled plasma-atomic emission spectrometry (ICP-AES). Samples were analyzed to confirm free hydroxide was still present before testing was initiated. Titration determined the AN-107 UFC sample contained 0.55M free hydroxide. Since excess free hydroxide was still present in the AN-107 diluted feed, no caustic adjustment was required. The [Na] data were used to determine the quantity of 0.01M NaOH to add to the AN-107 UFC sample to yield approximately 5.5M sodium in the waste prior to treatment.

2.2 Development of Test Conditions

Experimental conditions were defined using the results from earlier reaction mechanism studies with AN-102/C-104 waste blend simulant (Lilga et al. 2003). Based on these studies, minimum levels (0.02M) of reagents were added to determine the differences in the effectiveness of treatment conditions. The addition of 0.02M $Sr(NO_3)_2$ and permanganate showed adequate Sr-90 and TRU decontamination (Hallen et al. 2002a). The test matrix for each tank sample was slightly different. The total number of tests was

held to a minimum to limit the volume of waste used and to keep associated analytical costs within the budget. This information was used to construct the test matrices shown in Tables 2.1 and 2.2 for AN-102 and AN-107, respectively. The target concentrations listed in the test matrices are based on the final composition after addition of all reagents. Both test matrices included a repeat of the second experimental conditions as the final experiment in the matrix to examine the variability in conducting duplicate experiments. The quantity of each reagent to add to the waste to achieve these values, as well as the actual quantities that were used, are listed in Appendix A.

Table 2.1. Test Matrix for Experiments Using Tank AN-102 Waste Samples

Experiment Number	Sr^{+2}	Mn(VII)	Mn(VI)	Mn(IV)	Mn(II)[a]	Other[b]	Stir	Added OH^-
RX-01	--	--	--	--	--	--	No	AR[c]
RX-02	0.02M	0.02M	--	--	--	--	Yes	AR
RX-03	--	0.02M	--	--	--	--	Yes	AR
RX-04	0.02M	0.02M	--	--	--	--	Yes	0.3M
RX-05	0.02M	0.02M	--	--	--	--	No[d]	AR
RX-06	0.02M	--	0.02M	--	--	--	Yes	AR
RX-07	0.02M	--	--	Solid[e]	--	--	Yes	AR
RX-08	0.02M	--	--	--	0.02M	--	Yes	AR
RX-09	0.02M	--	--	--	--	IO_4^-[f]	Yes	AR
RX-10	0.02M	--	--	--	--	Zr(IV)[g]	Yes	AR
RX-11	0.02M	0.02M	--	--	--	--	Yes	AR

(a) Mn^{+2} precipitates as $Mn(OH)_2$, which air oxidizes to Mn(IV).
(b) Periodate (IO_4^-) is a non-precipitating oxidant, and zirconium, Zr(IV), is a non-oxidizing precipitant.
(c) AR = as received, 0.14M OH^-; no added hydroxide.
(d) Mix sample after 1h of reaction.
(e) Solid reagent, freshly precipitated $MnO(OH)(ONa) \cdot xH_2O$, same number of Mn equivalents added.
(f) Solid reagent, KIO_4^-.
(g) 0.02M $Zr(NO_3)_4$.

Table 2.2. Test Matrix for Experiments Using Tank AN-107 Waste Samples

Experiment Number	Sr^{+2}	Mn(VII)	Mn(VI)	Mn(II)[a]	Other[b]	Stir
SS-01	--	--	--	--	--	No
SS-02	0.02M	0.02M	--	--	--	Yes
SS-03	0.02M	--	0.02M	--	--	Yes
SS-04	0.02M	0.02M	--	--	--	No[c]
SS-05	0.02M	--	--	0.02M	--	Yes
SS-06	0.02M	--	--	--	IO_4^-[d]	Yes
SS-07	0.02M	--	--	--	Zr(IV)[e]	Yes
SS-08	0.02M	0.02M	--	--	--	Yes

(a) Mn^{+2} precipitates as $Mn(OH)_2$, which air oxidizes to Mn(IV).
(b) Periodate (IO_4^-) is a non-precipitating oxidant, and zirconium, Zr(IV), is a non-oxidizing precipitant.
(c) Mix sample after 1h of reaction.
(d) Solid reagent, KIO_4^-.
(e) 0.02M $Zr(NO_3)_4$.

2.3 Experimental

The waste samples were diluted with 0.01M NaOH just prior to waste testing in the Shielded Analytical Laboratory hot cells (in the RPL). The small-scale experiments were conducted in 60-mL sample jars with approximately 20 mL of the diluted tank waste. The reagents were added rapidly to the wastes with an adjustable pipette, in the order listed in Tables 2.1 and 2.2 (from left to right), at ambient hot cell temperature, and mixed with magnetic stir bars when specified. The ambient hot cell temperatures ranged from 26°C to 28°C on the days of these tests. Each series was conducted over a 2-day period, approximately 1 month apart. Samples were collected at the specified times of 4h and/or 24h and filtered immediately with a 0.2-μm disposable syringe filter. Duplicate samples of initial waste, RX-01 and SS-01, were filtered, along with the other samples, but no chemical reagents were added. The samples for chemical and radiochemical analyses were acidified and diluted to the appropriate levels for the analytical method. Samples for titration were submitted without any chemical addition.

Sample RX-06, Mn(VI) addition, was spilled during weighing after the reagent addition. The sample could not be recovered, and no additional waste remained to re-run this condition. The Mn(VI) test condition, SS-03, was examined with AN-107 waste to provide data for reaction mechanism assessment.

Stock solutions of the reagents were prepared for addition to the waste. The experiments used 0.44M solutions of $Sr(NO_3)_2$, $NaMnO_4$, $MnCl_2$, and $Zr(NO_3)_4$. The Mn(VI) solution was freshly prepared before each series of tests by reducing $NaMnO_4$ (0.44M) with an equal volume of 0.22M $NaHCO_2$ in 2M NaOH. The resulting solution was 0.22M Mn(VI), and 2 mL were added to approximately 20 mL of waste to give the target of 0.02M Mn addition. The 1M free hydroxide stabilized the Mn(VI) solutions for up to 24h after preparation. Without additional hydroxide, the Mn(VI) would disproportionate to Mn(VII) and Mn(V), ultimately leading to loss of Mn(VI) from the solution.

The Mn(IV) solids were prepared by the $NaMnO_4$ reduction with $NaHCO_2$ in 0.1M NaOH. After complete precipitation of the Mn (no remaining solution color), the solids were collected on a filter, washed with deionized water, and then dried in a vacuum oven. A sample of the dried solids was digested and analyzed by ICP-AES. The formula weight of the Mn(IV) solids was determined to be 205 grams per mole of Mn. The target Mn addition of 0.02M is equal to 0.09 grams of solids added for each approximately 20 mL of sample. Potassium periodate, target 0.06M or 0.3 grams, was added as a solid because of its low solubility in water at room temperature. Caustic addition to RX-04 involved adding 1 mL of 6.6M NaOH to approximately 20 mL of waste. The actual quantities of waste and reagents used are given in Appendix A.

The test specification stated the temperature for these tests as 25 ± 5°C. The ambient hot cell temperature on the days of these tests varied from 26°C to 29°C, which was within the temperature requirement; hence, no external heating or cooling was provided for the samples during this testing.

2.4 Chemical Analyses

All of the chemical analyses were conducted at PNWD. The test specification designated the analytes of interest and minimum reportable quantities (Abodishish 2002). Alpha energy analysis was used to

determine the TRU content based on the reported sum of the alpha emitters. Total alpha counting was conducted on the RX samples only. The Sr-90 concentration was determined by chemical separation followed by beta counting. Sodium concentration was determined by ICP-AES, as were the other metals listed in the test instructions. Selected samples were also analyzed by direct titration with 0.2M HCl to determine the free hydroxide concentration (free hydroxide in the sample corresponds to the first equivalence point). All of the analytical results are included in Appendix B.

2.5 Quality Assurance Requirements

PNWD implements the RPP-WTP quality requirements by performing work in accordance with the River Protection Program-Waste Treatment Plant Technical Support Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. This work was performed to the quality requirements of NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Subpart 2.7. These quality requirements are implemented through the Waste Treatment Plant Support Project Quality Assurance Requirements and Description (WTPSP) Quality Assurance Manual. The analytical requirements for this work were implemented through the Laboratory's internal QA Manual, Conducting Analytical Work in Support of Regulatory Programs.

Experiments that are not method-specific were performed in accordance with QA Implementing Procedures QA-RPP-WTP-1101 "Scientific Investigations" and QA-RPP-WTP-1201 "Calibration Control System," assuring that sufficient data were taken with properly calibrated measuring and test equipment to obtain quality results. All QA Implementing Procedures are contained in the WTPSP Quality Assurance Manual.

PNWD addressed internal verification and validation activities by conducting an Independent Technical Review of the final data report in accordance with Implementing Procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the test plan objectives.

BNI's QAPjP, 24590-QA-0001, is not applicable since the work was not performed in support of environmental/regulatory testing, and the data should not be used as such.

3.0 Results and Discussion

The results of experiments with AN-102 and AN-107 waste to assess the reaction mechanisms for the Sr/TRU removal process are discussed in this section.

3.1 Decontamination of Sr-90

Each series of experiments using AN-102 and AN-107 waste involved multiple samples, analyzed as multiple analytical batches, and provided analytical results to determine the change in waste composition upon treatment. Duplicate samples of each starting waste were analyzed after filtration to determine the initial composition of the supernatant. The radionuclide composition of the treated samples was compared with the initial composition to determine the extent of decontamination. The 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 during sample treatment:

$$DF = [A]_i / ([A] * MD)$$

where $[A]_i$ is the concentration of component A per mass in the initial sample; [A] 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 the mass of initial waste and all dilutions, adjustments, and/or reagent additions.

The DFs for Sr-90 from treated AN-102 samples are shown in Figure 3.1. Sr-90 removal was observed for all experiments where nonradioactive $Sr(NO_3)_2$ was added, consistent with the mechanism for Sr-90 removal involving isotopic dilution and $SrCO_3$ precipitation. The addition of permanganate alone resulted in no Sr-90 removal; nonradioactive Sr must be added to remove Sr-90 from Envelope C wastes. The Sr-90 DFs were greatly increased by the solids digest time of 24h versus 4h. The added free hydroxide (0.3M) had little impact on the Sr-90 DFs compared with treatment of the waste without any additional hydroxide (0.1M). The no-mix experiment for the first hour of reaction did not have a significant impact on the Sr-90 DF, supporting the earlier conclusions from simulant tests that mixing during reagent addition and initial permanganate reduction are not important as long as the treated samples are well mixed before filtration. The oxidation state of the Mn added had little impact on the Sr-90 DF. Sr-90 removal was less effective when combined with periodate addition, compared with other experiments where $Sr(NO_3)_2$ was added. Nonradioactive Sr and Zr(IV) addition gave a Sr-90 DF that was nearly as high as permanganate. These results suggest that complexant oxidation is not important for Sr-90 removal and that isotopic dilution and $SrCO_3$ precipitation are the primary mechanisms.

Addition of 0.02M nonradioactive Sr would result in 1400 μg/g [Sr] if $SrCO_3$ precipitation did not occur. The [Sr] data for the AN-102 tests are shown in Figure 3.2. Note that the relatively high concentration of Sr for periodate addition corresponds to precipitation of 84% of the added Sr, and the highest DFs (permanganate addition) correspond to precipitation of 94% of the added Sr. The [Sr] data also show the same consistent trend as found in earlier tests: at 4h, the total [Sr] is still relatively high; after 24h, the values are generally half the 4h values, resulting in a doubling of the Sr-90 DF. However, the

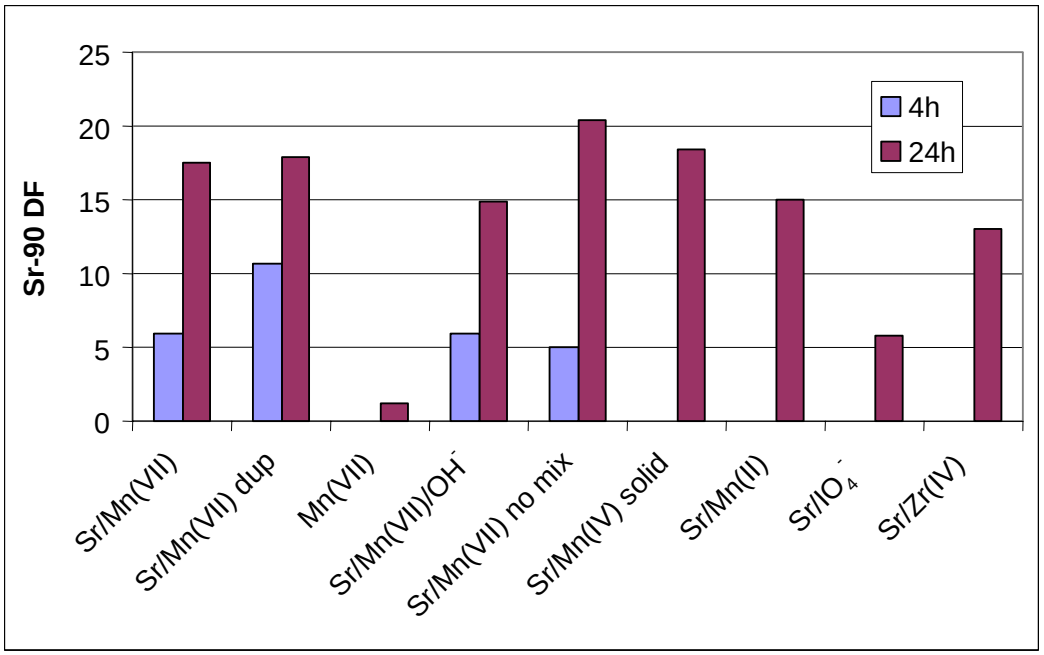

Figure 3.1. Strontium-90 Decontamination Factors for Treated AN-102 Samples as a Function of Added Reagent and Reaction Time. Target Reagent Levels: 0.02M for all except OH^- (target of an additional 0.3M) and IO_4^- (target concentration of 0.06M).

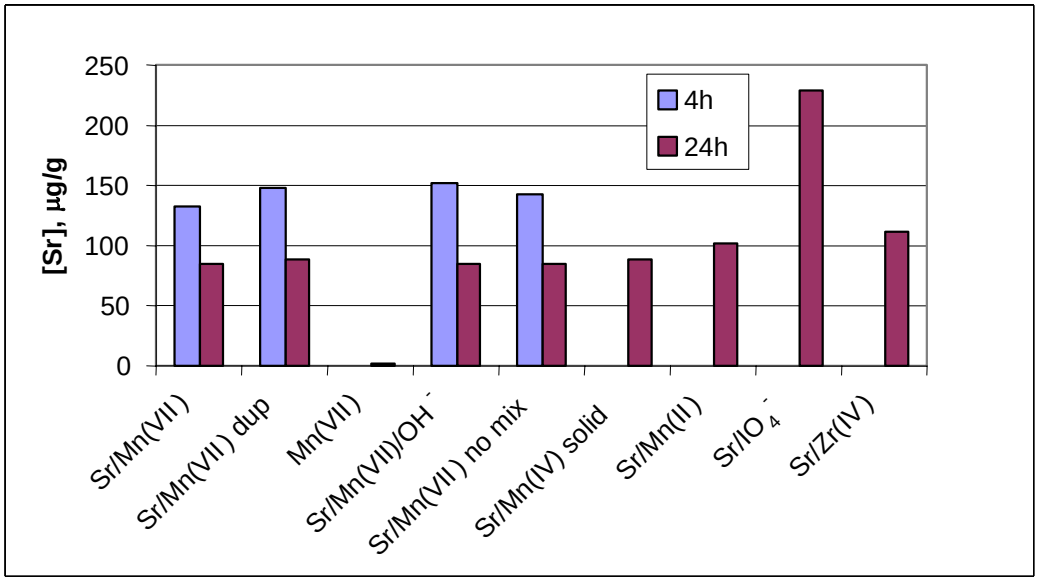

Figure 3.2. Total Strontium Solubility in the Treated AN-102 Samples as a Function of Reagent Addition and Reaction Time

[Sr] is not the only difference in DF for the 4h samples. When the isotopic dilution ratio, Sr-90/[Sr], is examined (Table 3.1), three of the four samples taken at 4h of reaction time do not appear to have reached the final equilibrium ratio (~0.018 μCi/μg). The experimental average of the 24h samples and the calculated ratio based on the quantity of added $Sr(NO_3)_2$ agree quite well, showing that isotopic exchange is complete. The slow isotopic exchange for the 4h samples is likely a result of the procedure used for the small-sample tests with reagent addition via an autopipetter, where the $Sr(NO_3)_2$ solution is injected rapidly into the waste solution. In the bench-scale tests where complete isotopic exchange was noted at 18 min, the Sr solution was added slowly over 6 min with continuous stirring by an overhead-driven impeller. The undersaturation of Sr in the initial waste can be seen by comparing the [Sr] in the initial waste (~1 μg/g) to the [Sr] in any of the samples with added nonradioactive Sr (85-228 μg/g).

The results for experiments with AN-107 waste can be compared to those for AN-102 waste. The comparison of Sr-90 DFs at 24h of reaction is shown in Figure 3.3. Sr-90 removal was high (DF >10) for the AN-107 waste and in most cases quite similar to AN-102. The corresponding TRU removal process did not significantly impact Sr-90 removal from AN-107 as was noted for AN-102. Only minor differences appear for Sr-90 removal from the two Envelope C wastes for the no-mix and periodate experiments. The AN-102 sample results appear to be more sensitive to changes in process conditions than for corresponding treatment conditions with AN-107 samples. The carbonate (TIC) concentrations are high (>0.5M) in both these wastes and likely have little impact on the differences observed, as only 0.02M is consumed on $SrCO_3$ precipitation.

Table 3.1. Isotopic Exchange Ratio, [Sr-90]/[Sr] in μCi/μg, for AN-102 Tests

Conditions	[Sr-90], μCi/g		[Sr], μg/g		[Sr-90]/[Sr], μg/g	
	4h	24h	4h	24h	4h	24h
Initial	28	28	[1.2]	[1.1]	24.1	25.8
Sr/Mn(VII)	4.4	1.5	132	85	0.0330	0.0177
Sr/Mn(VII) dup	2.5	1.5	149	88	0.0165	0.0166
Mn(VII)	--	22	--	[1.0]	--	22.2
Sr/Mn(VII)/OH^-	4.3	1.7	151	85	0.0281	0.0198
Sr/Mn(VII) no mix	5.3	1.3	141	85	0.0373	0.0150
Sr/Mn(IV) solid	--	1.5	--	89	--	0.0164
Sr/Mn(II)	--	1.7	--	101	--	0.0172
Sr/IO_4^-	--	4.7	--	228	--	0.0204
Sr/Zr(IV)	--	2.0	--	111	--	0.0180
Values in brackets [ ] are greater than the method detection limit (MDL) but less than the estimated quantitation limit (EQL), with errors likely to exceed 15%.					Experimental Avg.	0.0176
					Stddev	0.0018
					Calculated ratio	0.0185

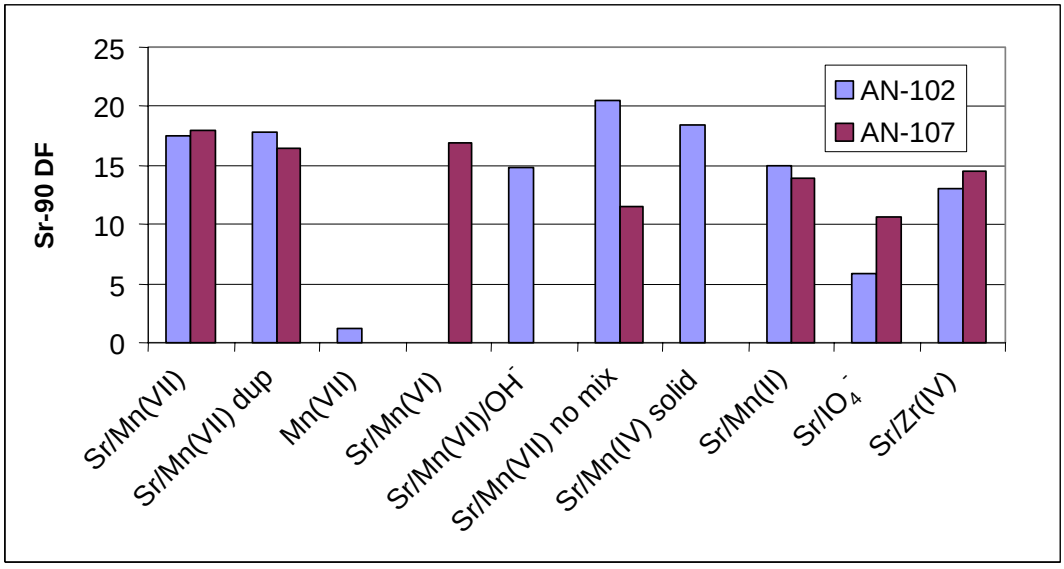

Figure 3.3. Comparison of Sr-90 DF for AN-102 and AN-107 Experiments

3.2 Decontamination of TRU

The effectiveness of the various treatment conditions for TRU removal from AN-102 can be seen by examining the DFs for the sum of the alpha shown in Figure 3.4. The DFs were significantly higher when

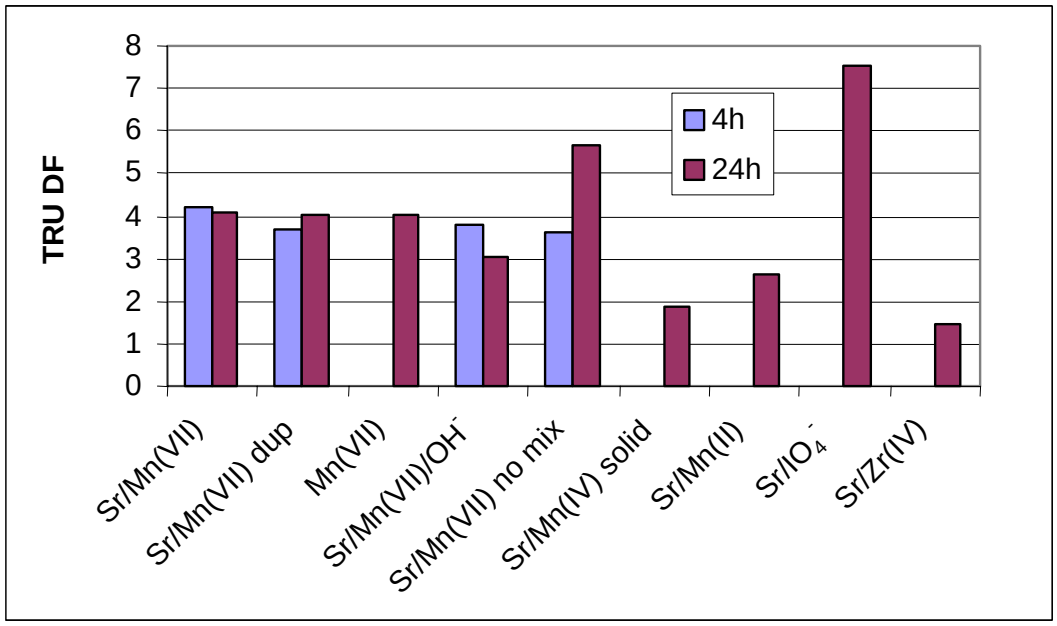

Figure 3.4. TRU (Sum of Alpha) Decontamination Factors for Treated AN-102 Samples as a Function of Reagent Addition and Reaction Time

oxidation occurred, i.e., experiments where permanganate, Mn(VII), or periodate was added. The periodate treatment (oxidation) showed even greater TRU removal than permanganate, possibly because the target concentration of periodate was 0.06M, which corresponds to about twice the oxidation equivalence as permanganate at 0.02M. Process changes coupled with permanganate addition (no Sr addition; hydroxide added; or no mixing for the first hour of reaction) did not significantly impact TRU decontamination. Precipitation, sorption, and ligand exchange by reduced states of Mn (IV and II) and Zr(IV) resulted in only low levels of TRU removal, DF ~2 (50% removal). The precipitation of Mn solids in the waste solution (no mix) resulted in higher TRU DFs than addition of preformed Mn(IV) solids. The high organic content in the waste solution likely resulted in the formation of more reduced Mn solids, which were more effective at TRU removal than preformed Mn(IV) solids. There was very little difference in time dependence in the TRU DFs, with little difference between DFs at 4h and 24h sample times.

Two different radiochemical methods were used to determine the alpha content of the waste samples. Individual isotope pairs were determined by the alpha counting technique and added together to give the sum of alpha. Total alpha was determined in a separate technique by counting the entire sample. The DFs for the two techniques and individual isotopes can be plotted individually for comparison (Figure 3.5). DFs for the two techniques and Am and Cm isotopes are very similar. The Am and Cm

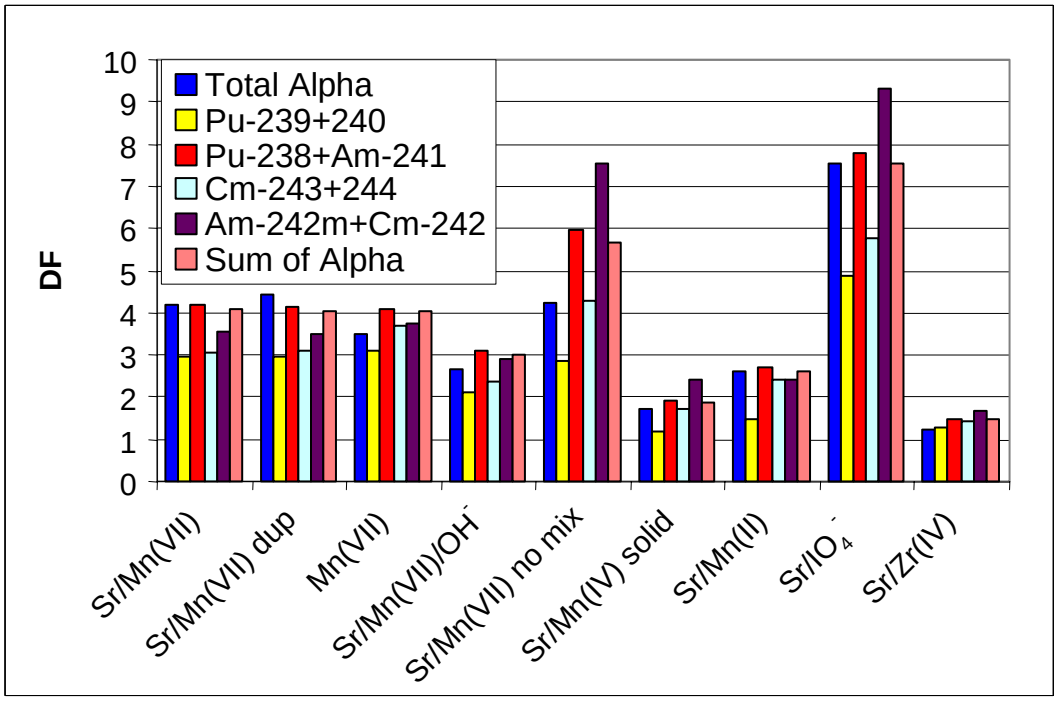

Figure 3.5. Comparison of Decontamination Factors for Total Alpha, the Various Isotope Pairs, and the Sum of Alpha (Treated AN-102 at 24h)

isotopes are most likely in the most stable +3 oxidation state. Since Am-241 accounts for approximately 90% of the TRU in AN-102 waste, the Pu-238+Am-241 data can be considered all Am-241. The Cm isotopes, primarily 243 and 244, account for approximately 5% of the total TRU. The Cm isotope data have greater variability due to increased analytical error associated with the very low concentrations of these isotopes. The DFs for the Pu isotopes (Pu-239+240) are consistently lower than found for the Am and Cm isotopes. Both Pu and U+Np isotopes were reported to have lower DFs than Am and Cm in earlier bench-scale tests (Hallen et al. 2002c). Permanganate treatment is most effective for removing the +3 valence TRU elements.

Treatment of AN-107 waste under similar conditions gave significantly different results than AN-102; see the comparison shown in Figure 3.6. TRU removal from AN-107 was significantly higher for most treatment schemes compared to the corresponding treated AN-102 sample. The results for Mn(II), IO_4^-, and Zr(IV) show that oxidation is not as important for TRU decontamination in AN-107 waste. This was somewhat surprising because AN-107 is initially higher in TOC and concentrations of soluble Fe and Mn. Co-precipitation, sorption, and ligand exchange result in high TRU removal from AN-107 waste. AN-102 waste may have higher concentrations of active complexants that require oxidation for effective TRU removal.

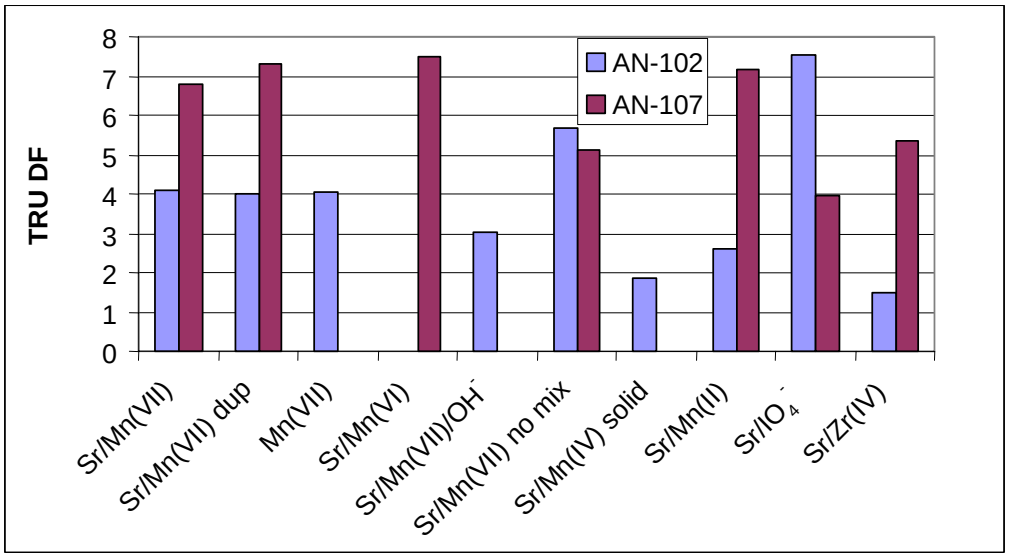

Figure 3.6. Comparison of TRU (Sum of Alpha) DF for AN-102 and AN-107 Waste Samples (24h data)

3.3 Change in Chemical Composition

ICP-AES data can be used to determine the impact of the various process conditions on the chemical composition of the supernatant. The impact of the process condition on the chemical composition of the treated supernatant is calculated as a percent removal relative to the starting waste. Table 3.2 shows the composition of the AN-102 starting waste in μg/g, and the percent change that occurred for the various treatment conditions. Similar data are shown for AN-107 waste and treated samples in Table 3.3.

Table 3.2. AN-102 Chemical Composition and Change on Treatment as Percent Removal

Analyte	Initial Waste, µg/g	Sr/Mn(VII), % Removed		Sr/Mn(VII) Dup, % Removed		Mn(VII), % Removed	Sr/Mn(VII)/OH, % Removed		Sr/Mn(VII) No Mix, % Removed		Sr/Mn(IV), % Removed	Sr/Mn(II) , % Removed	Sr/IO_4^-, % Removed	Sr/Zr(IV), % Removed
		4h	24h	4h	24h	24h	4h	24h	4h	24h	24h	24h	24h	24h
Al	4,890	6	4	-4	-3	8	5	6	6	5	-2	-5	-1	-2
Ca	201	17	19	18	20	7	19	21	19	22	19	20	45	21
Cd	27	5	3	-1	-2	7	5	5	6	6	-1	-3	17	-2
Co	[2]	-1	0	10	10	-1	1	-4	15	15	8	8	9	6
Cr	84	26	22	24	23	27	25	18	34	39	24	39	6	20
Cu	7	6	5	7	7	8	11	13	12	11	6	3	10	5
Fe	8	72	73	60	74	74	71	72	75	83	49	61	70	37
K[(a)]	935	4	3	-7	-7	6	5	5	7	8	-6	-8	-210	-7
La	[7]	61	71	70	78	44	73	70	68	79	59	66	>79	49
Mn[(a)]	[2]	-96	>39	-53	37	2	-51	>35	-72	-126	-23	-233	47	>35
Mo	22	3	2	1	0	5	4	3	5	6	1	-3	0	0
Na[(a)]	98,700	5	4	-4	-6	8	0	0	0	3	-4	-5	-4	-4
Nd	14	63	63	73	72	59	64	48	70	78	43	60	81	27
Ni	181	4	2	1	1	5	4	3	5	4	2	-1	1	0
P	825	6	4	1	1	7	6	5	8	7	1	-1	-1	9
Pb	82	11	10	11	11	12	18	18	21	18	22	13	36	23
Sr[(a)]	[1]	-12,600	-8,100	-14,300	-8,400	20	-15,100	-8,500	-13,500	-8,100	-8,200	-9,700	-21,300	-10,600
W	[57]	6	7	1	2	8	6	5	9	8	8	-1	-2	-1
Zr[(a)]	[3]	57	61	61	64	62	57	62	49	62	30	45	53	-3,705

(a) Reagent containing this element was added to some samples during testing, which resulted in increased concentration (a negative percent removal).
># = analyte was below the method detection limit (MDL) and % removal given as greater than the MDL.
Values in brackets [ ] are greater than the MDL but less than the estimated quantitation limit (EQL), with errors likely to exceed 15%.

Table 3.3. AN-107 Chemical Composition and Change on Treatment as Percent Removal

Analyte	Initial Waste, µg/g	Sr/Mn(VII), % Removed		Sr/Mn(VII) Dup, % Removed		Sr/Mn(VI), % Removed	Sr/Mn(II), % Removed	Sr/Mn(VII) No Mix, % Removed	Sr/IO_4^-, % Removed	Sr/Zr(IV), % Removed
		4h	24h	4h	24h	24h	24h	24h	24h	24h
Al	1740	-2	0	2	1	1	3	1	2	5
Ca	336	20	19	25	18	18	20	16	24	25
Cd	27	0	0	2	0	1	2	2	2	3
Co	2	4	0	-2	-2	-2	-4	-5	4	-7
Cr	43	26	21	24	20	24	52	29	-9	39
Cu	9	0	0	2	1	2	2	2	3	3
Fe	242	97	97	97	97	97	93	85	91	83
K[(a)]	732	-5	-4	0	0	-3	-1	-3	-232	3
La	[6]	79	79	>75	80	77	70	77	76	75
Mn[(a)]	51	90	97	90	96	96	78	82	96	88
Mo	15	2	2	1	1	2	3	-1	1	2
Na[(a)]	100,000	-2	2	2	0	-1	6	5	2	3
Nd	19	78	77	76	76	75	65	75	72	72
Ni	222	1	1	-1	-1	2	3	2	0	1
P	302	0	0	1	1	1	1	1	1	4
Pb	102	29	30	24	25	30	32	33	20	48
Sr[(a)]	[3]	-6400	-2800	-5800	-3200	-3000	-3500	-3100	-4700	-3400
W	75	2	0	4	3	1	2	3	3	5
Zr[(a)]	15	79	82	80	82	82	73	79	67	-137

(a) Reagent containing this element was added to some samples during testing, which resulted in increased concentration (a negative percent removal).
># = analyte was below the method detection limit (MDL) and % removal given as greater than the MDL.
Values in brackets [ ] are greater than the MDL but less than the estimated quantitation limit (EQL), with errors likely to exceed 15%.

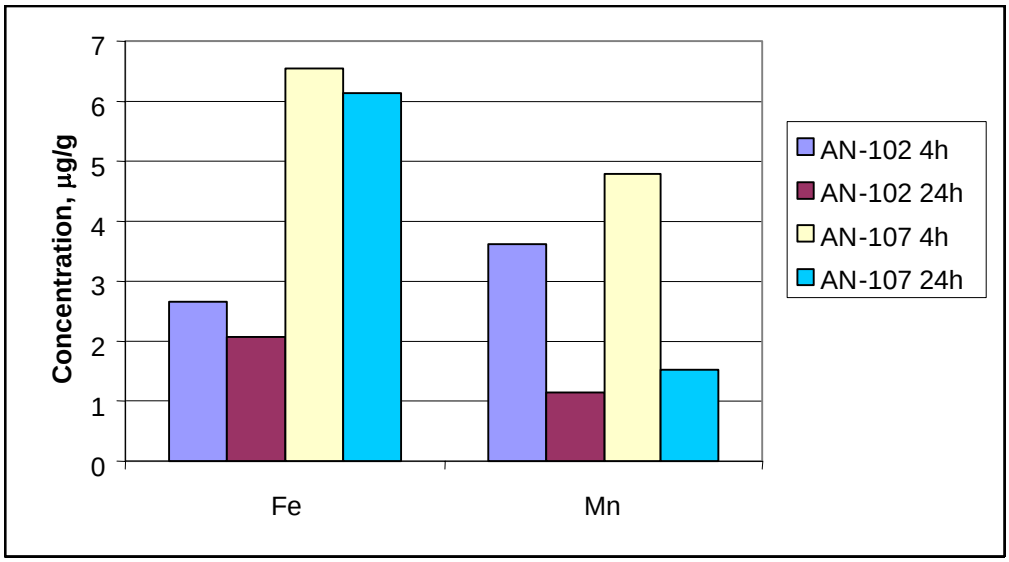

Figure 3.7. Fe and Mn Levels in AN-102 and AN-107 Samples After Addition of Sr and Permanganate

Both treated wastes showed similar trends with the analytes that had little or no significant change for the various treatments: Al, Cd, Co, Cu, K, Mo, Ni, and P. It is important that both Al and P stay in the supernatant that goes to the LAW glass melter, because these components can limit waste loading in the glass. Chromium is also an important element that is preferred in the supernatant, because it can limit waste loading in the glass. Some Cr is removed when permanganate is added. Some differences were noted for Cr, with very little Cr removal with IO_4^-. However, with the low reagent addition much less Cr is removed. Earlier tests with AN-107 waste and 0.05M added permanganate showed Cr removal ranging from 50% to as high as 90% removal. Consequently, the reduced level of permanganate has resulted in less Cr in the Sr/TRU precipitate (HLW).

As discussed in Section 3.1, Sr addition caused a large increase in [Sr] in the treated supernatant. The Sr addition removed Ca from solution. This is likely a result of the competition of Sr and Ca for the complexing agents (EDTA/HEDTA) and precipitation of calcium carbonate. However, the Ca removal, like the Sr-90 decontamination, was not significantly impacted by the addition of permanganate, hydroxide, or other reagents. The Zr concentration also decreased with permanganate treatment and increased when $Zr(NO_3)_4$ was added. This supports the conclusion that Zr(IV) effectively competes for the organic complexants with TRU elements by ligand exchange, which results in TRU precipitation.

Large differences can be seen in the initial concentrations and removal of Fe and Mn from the two wastes and various treatment conditions. The initial levels of Fe and Mn are approximately 25 times greater in AN-107 than in AN-102. However, after treatment, the levels of Fe and Mn are quite similar (Figure 3.7). The Fe levels show little change with time, a similar trend as the TRU DFs. However, the Mn concentration is significantly decreased after 24h of reaction. Reduced levels of soluble Mn will result in less potential for post-filtration precipitation.

3.4 Comparison of AN-102 Active and Simulant Test Results

Similar chemical analyses of actual waste (active) and simulant (inactive) test samples were performed by ICP-AES. The ICP-AES results can be used to compare the [Sr] results from the active AN-102 tests with those of Lilga et al. (2003) using simulant (Figure 3.8). The data shown are only for the 24h samples. The data are quite close, and the slightly higher Sr levels in the simulant suggest the EDTA/HEDTA concentration in the simulant experiments was higher than in the diluted AN-102 waste. With respect to Sr chemistry, the waste simulant results appear to represent actual waste treatment; hence, the corresponding [Sr] in the treated simulant can be used to assess the expected Sr-90 DF.

Of the target analytes evaluated in the simulant tests (Lilga et al. 2003), the actual waste samples had concentrations of Fe, La, and Nd above the minimum detection limit. Results for these elements for selected treatment schemes of AN-102 simulant and active waste are shown in Figure 3.9. The results of the actual tank waste, 60-80% removal, are very similar to the simulant results for La and Nd. The Fe removal in the simulant was typically higher, ranging from 80-90%. The La and Nd removal was also similar to the TRU elements (DF 3-5 is 67-80% removal). This is likely because the primary TRU elements, Am and Cm (>95% of the TRU), are +3 actinide series ions that are similar in charge to the lanthanide ions. These results support the conclusions from simulant tests and the use of La and Nd removal as an indication of TRU decontamination of Envelope C wastes (AN-102 and AN-107).

Oxidation appears to be important for TRU removal from AN-102 waste, similar to the results reported by Lilga et al. (2003) for waste simulant using surrogate lanthanide ions as an indication of TRU removal. Some differences are noted between the actual and simulant tests; Lilga et al. (2003) found Zr(IV) addition was nearly equivalent to permanganate addition, not found for actual AN-102 waste tests.

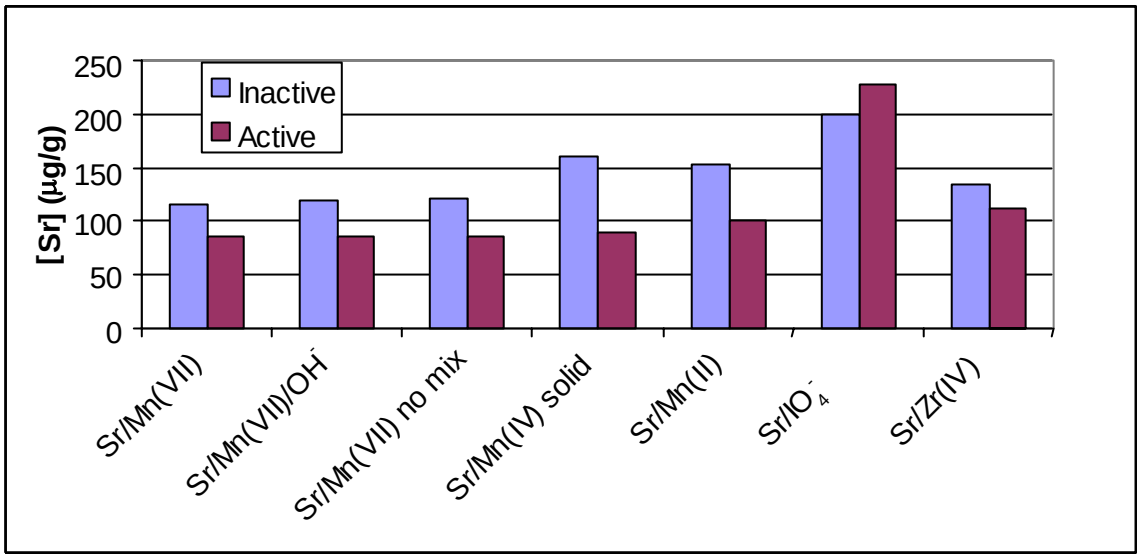

Figure 3.8. Comparison of Strontium Solubility for Active AN-102 and Inactive AN-102/C-104 Waste Blend Treated with Similar Reagents and Sampled (24h Data)

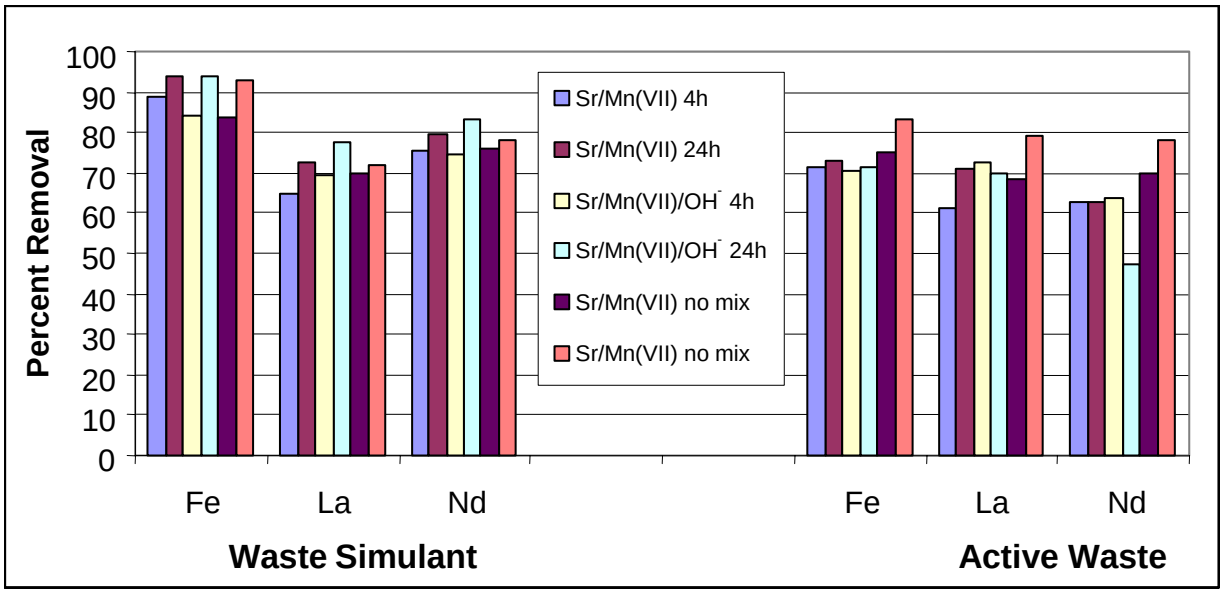

Figure 3.9. Percent Removal of Target ICP Metals for Selected Samples

3.5 Estimated Sr-90 and TRU Levels in ILAW Glass

The data from the experiments discussed here can be used to estimate the Sr-90 and TRU loadings that would be expected in ILAW glass made from the treated supernatant. The Sr-90 data are used directly for the calculation with an assumed LAW glass density of 2.76 g/mL. The TRU activity is calculated by summing the individual TRU isotopes (the sum of alpha). For AN-107 waste samples, the waste Na was 86.4% of the total Na because of the caustic adjustment (Hallen et al. 2000b). The calculated glass loadings are listed in Table 3.4 for the current baseline design waste glass concentration of 15 wt% waste Na_2O. The results show that all treated samples were below the contract limits for ILAW glass (DOE 2000), except for Sr-90 when no $Sr(NO_3)_2$ was added (permanganate-only). The target level of 50% below the limit was not met for Sr-90 at 4h of reaction. Thus, 0.02M added $Sr(NO_3)_2$ and ambient temperature are adequate to meet the contract requirement if the reaction time is 24h.

TRU loadings well below 50% of the contract limit were met when permanganate was added. Addition of other forms of Mn, periodate, and Zr(IV) generally led to higher levels of TRU in the LAW but still low enough to meet the contract requirements.

AN-107 waste initially had higher levels of Sr-90 and TRU than AN-102. After treatment, the levels of Sr-90 and TRU in the supernatant from both wastes were quite similar. These results show good decontamination of both wastes at low levels of reagent addition, 0.02M $Sr(NO_3)_2$ and 0.02M $NaMnO_4$, and ambient temperature. However, to minimize the concentration of Sr-90 in the ILAW waste, filtration should not begin until approximately 24h after reagents are added.

Table 3.4. Sr-90 and TRU ILAW Glass Loadings for 15 wt% Waste Na_2O

AN-102	Reaction Time, h	Sr-90, Ci/m^3	Sum of Alpha, nCi/g
ILAW Limits		**20**	**100**
Initial Waste	NA	88	89
Sr/Mn(VII)	4	16	--
	24	5	17
Sr/Mn(VII) dup	4	8	--
	24	5	19
Mn(VII)	24	78	24
Sr/Mn(VII)/OH^-	4	15	--
	24	6	26
Sr/Mn(VII) no mix	4	18	--
	24	4	21
Sr/Mn(IV) solid	24	5	35
Sr/Mn(II)	24	6	29
Sr/IO_4^-	24	15	11
Sr/Zr(IV)	24	6	52
AN-107	**Reaction Time, h**	**Sr-90, Ci/m^3**	**Sum of Alpha, nCi/g**
ILAW Limits		**20**	**100**
Initial Waste	NA	107	160
Sr/Mn(VII)	4	13	19
	24	6	24
Sr/Mn(VII) dup	4	13	21
	24	7	22
Sr/Mn(VI)	24	6	21
Sr/Mn(VII) no mix	24	8	24
Sr/Mn(II)	24	10	33
Sr/IO_4^-	24	10	41
Sr/Zr(IV)	24	8	31

4.0 Conclusions and Recommendations

Experiments were conducted with samples of diluted AN-102 and AN-107 waste at various modified Sr/TRU removal process conditions. These experiments provided a better understanding of the Sr/TRU removal process. Conclusions from this work and recommendations to consider for plant operation are presented in this section.

Experiments were conducted to assess the precipitation chemistry for Sr/TRU removal. A primary focus was to assess the reaction chemistry of Mn species relevant to the mechanism of TRU removal by permanganate treatment and to determine the importance of various mechanisms for decontamination, such as precipitation, absorption, ligand exchange, and oxidation of organic complexants. These studies were conducted with AN-102 and AN-107 waste samples. The optimized treatment conditions—no added hydroxide, addition of Sr (0.02M target concentration) followed by sodium permanganate (0.02M target concentration) with mixing at ambient temperature—were used as a reference for comparison. Reagent and treatment conditions were varied to give information about mechanisms of TRU decontamination and Sr-90 removal in the treatment process. Sr-90 is removed from solution by isotopic dilution with added nonradioactive Sr and precipitation as $SrCO_3$.

The addition of a chemical oxidant did not increase Sr-90 decontamination. None of the various treatment schemes or reagents added showed an improved Sr-90 DF. Strontium concentrations, and therefore decontamination levels, are time dependent. In all experiments in which Sr was added, the Sr concentrations decreased significantly between 4h and 24h. In some cases, the concentration was halved over this time, i.e., a doubling of the Sr-90 DF. This behavior is the same as that observed by Lilga et al. (2003) for simulated waste samples. The Sr concentrations in the actual waste and the simulated waste were also about the same, supporting the conclusions from the simulant studies.

The important factors for determining Sr-90 decontamination are the isotopic dilution ratio and the [Sr]. Approximately 95% of the added Sr is precipitated from solution. The isotopic dilution is quite rapid relative to $SrCO_3$ precipitation. The precipitation of $SrCO_3$ is slow, and adequate time must be allowed for equilibrium to be approached. Therefore, these results suggest that the treated waste should not be filtered at 4h after reagent addition; instead, additional time should be allowed for Sr to precipitate.

Several treatments employing various species of Mn (VII, VI, IV, and II); an alternative oxidant; and competing metal ion, Zr(IV), were examined. The DF of TRU (sum of alpha) was used as an indication of the extent/effectiveness of treatment. The performance of the reference treatment conditions was very similar to earlier tests with actual waste (Hallen et al. 2002c). In general, the reference process conditions gave the best TRU removal of the treatments tested. For AN-102, the treatments involving oxidation performed the best, although addition of Mn(IV), Mn(II) and Zr(IV) resulted in some TRU removal. For AN-107, oxidation appears to be less important, and similar TRU removal was found for permanganate and reduced Mn(II) and Zr(IV). AN-107 appears easier to treat for TRU removal. Ligand displacement, precipitation, and sorption of TRU ions appeared to be important for treatments of AN-107.

Time has very little impact on the TRU removal when comparing the 4h and 24h data. However, increased levels of Mn were noted for the treated samples after only 4h of reaction, whereas all Mn was removed by 24h of reaction. These results again suggest that the treated waste should not be filtered after only 4h of reaction; in this case, the increased Mn levels will likely lead to post-filtration precipitation. Instead, if filtration begins after 24h of reaction, all Mn will already have precipitated from solution.

The results for AN-102 simulant and active tests were very similar, supporting the earlier conclusions of Lilga et al. (2003). The lanthanide elements used in simulant studies as surrogates for the TRU elements have very similar removals as the actual waste samples and correlate well to the TRU decontamination. This correlation is likely because 95% of the TRU in the Envelope C wastes are Am and Cm isotopes that are in a similar oxidation state as the lanthanide elements.

The initial waste from Tank AN-107 was higher in both Sr-90 and TRU compared to waste from Tank AN-102. The experimental data from both wastes can be used to predict the loading of Sr-90 and TRU (sum of alpha) expected in the ILAW at a fixed waste sodium loading of 15 wt% (contract limit >10 wt%). At 24h of reaction, the levels of Sr-90 and TRU in the supernatant from treatment of both wastes were quite similar. For Sr-90 loading, the levels were three times below the ILAW requirements of 20 Ci/m^3 and for TRU loading, the levels were four times below the requirements of 100 nCi/g when treated by addition of nonradioactive Sr and sodium permanganate at reagent levels of 0.02M. These reagent levels are significantly below the baseline treatment conditions of 0.075M Sr and 0.05M permanganate.

5.0 References

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Appendix A

Sample Data

Table A.1. Mass Dilution Factors (MDF) Used in DF and Percent Removed Calculations for AN-102 Tests

Experiment Number	AN-102 Added		Sr Added (0.44 M)		Reagents Added			MDF
	Target, mL	Actual wt, g	Target, mL	Actual wt, g	Reagent	Target, mL	Actual wt, g	
RX-01	10	16.9160	--	--	None	--	--	1
RX-02	20	25.3779	1	1.0164	Mn(VII)	1	1.0213	1.0803
RX-03	20	25.2622	0	0.0000	Mn(VII)	1	1.0218	1.0404
RX-04	20	25.0623	1	1.0413	Mn(VII)/OH^-	1 Each	2.1939	1.1291
RX-05	20	25.3820	1	(a)	Mn(VII)	1	2.0222	1.0797
RX-07	20	24.8547	1	1.0378	Mn(IV)	Solid	0.0767	1.0448
RX-08	20	25.2263	1	1.0411	Mn(II)	1	1.0298	1.0821
RX-09	20	25.2498	1	1.0452	KIO_4^-	Solid	0.276	1.0523
RX-10	20	24.9617	1	1.0385	Zr(IV)	1	1.0605	1.0841
RX-11	20	24.8819	1	1.0369	Mn(VII)	1	1.0289	1.0830
(a) No-mix experiment; no individual weights.								

Table A.2. Mass Dilution Factors (MDF) Used in DF and Percent Removed Calculations for AN-107 Tests

Experiment Number	AN-107 Added		Sr Added (0.44M)		Reagents Added			MDF
	Target, mL	Actual wt, g	Target, mL	Actual wt, g	Reagent	Target, mL	Actual wt, g	
SS-01	~40	47.3920	--	--	None	--	--	1
SS-02	20	25.3219	1	1.0622	Mn(VII)	1	1.0206	1.0823
SS-03	20	25.3296	1	1.0713	Mn(VI)	2	2.1183	1.1259
SS-04	20	25.6255	1	1.0553	Mn(II)	1	1.0339	1.0815
SS-05	20	24.9558	1	(a)	Mn(VII)	1 Each	2.1042	1.0843
SS-06	20	24.8524	1	1.0141	KIO_4^-	Solid	0.2461	1.0507
SS-07	20	24.7991	1	1.0699	Zr(IV)	1	1.0759	1.0865
SS-08	20	24.6714	1	1.0669	Mn(VII)	1	1.0253	1.0848
(a) No-mix experiment; no individual weights.								

Appendix B

Analytical Data

Battelle PNNL/RS&E/Inorganic Analysis ... ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	42365 / W63934
ASR#:	6583
Client:	R. Hallen
Total Samples:	18 (liquid)

	First	Last
RPL#:	02-03410	02-03428
Client ID:	RX-01-04	AN-107-UFC-Dup

Sample Preparation: PNL-ALO-128 (SAL/vh)

Revision 1 (Fe and Mg added to AOI section of data report)

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

Analyst: D.R. Sanders

Analysis Date (File): 10-01-2002 (A0849)
10-02-2002 (A0850)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

B.M. Dem 11/1/02
Preparer

11/1/02
Review and Concur

Eighteen supernatant samples submitted under Analytical Service Request (ASR) 6583 were analyzed by ICPAES. The samples were prepared by acid extraction per PNL-ALO-128 in the Shielded Analytical Laboratory (SAL) using a nominal 1.0 mL of sample and diluting to a final volume of approximately 25 mL. Sample preparation and analysis were conducted in two separate batches.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (9 pages). Analytes of interest (AOIs) were specified in the ASR, and are listed in the upper section of the report. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified as AOIs are reported in the bottom section of the data report, but have not been fully evaluated for QC performance.

The results are given as μg/mL for each detected analyte, and have been adjusted for all laboratory processing factors. Minimum Reportable Quantity (MRQ) values were specified in the ASR for selected AOIs. To meet this requirement, method detection limits (MDL) for the ICPAES analyses should be ≤ (MRQ ÷ 3). The required MRQ levels were met for all of the selected AOIs except for potassium. However, potassium was detected in all the samples at levels from about 1000 to 1500 μg/mL, which is above the MDL and also well above the MRQ of 75 μg/mL.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For each extraction processing, a process blank, blank spike, matrix spike, and duplicate were prepared along with the samples. The blank spikes and matrix spikes were prepared using 1.5 and 0.5 mL respectively of multi-element spike solutions BPNL-QC-1A and -2A. One of the AOIs, europium, was not present in the combined spike solution.

Process Blank:

A process blank (reagents only) was prepared with each group of samples. The concentrations of all AOIs in the blank were within the acceptance criteria of <EQL (estimated quantitation level ≡ 10 x MDL) or <5% of the concentration in the samples.

Blank Spike:

A blank spike (reagents and spike solution) was prepared with each group of samples. Except for sodium in the second sample batch, the recovery values were within the acceptance criterion of 80% to 120% for all AOIs. Analytes recovered at levels less than the EQL are shown as bold.

The second component of the blank spike (BPNL-QC-2A) contains 0.7% HF. Thus, the slight over-recovery for sodium in the second batch is almost certainly due to leaching of Na (as well as B and Si) from the glass container used to prepare the sample. Note, that the recovery for Na was also at the high end of the acceptance criterion for the first blank spike, and that B and Si were over-recovered for both blank spikes. It should be noted that sodium results for sample analyses not containing the BPNL-QC spike are not affected by this leaching process.

Duplicate RPD (Relative Percent Difference):

A duplicate was prepared with each group of samples. RPDs are listed for all analytes with concentrations ≥ EQL. The RPDs were within the acceptance criteria of ±15% (±10% for Na) for all AOIs meeting the above requirement.

Laboratory Co ıtrol Standard (LCS):

No LCS samples were prepared for analysis.

Matrix Spiked Sample:

Matrix spikes were prepared with Samples 02-03413 and 02-03421. Recovery values are listed for all analytes in the spike measured above the EQL, and with spike concentrations ≥ 20% of that in the sample. The recovery values were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirements. Analytes not meeting these requirements have either no recovery value listed (< EQL), or are listed as not recovered ("nr").

Post-Spiked Samples (Spike A Elements):

A post-spike A was conducted on Samples 02-03410 and 02-03421. Recovery values are listed for all analytes in the spike with concentrations > 20% of that in the sample. The recovery values were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirement. Analytes not meeting the 20% requirement are listed as not recovered ("nr"). Analytes recovered at levels below the EQL are shown as bold.

Post-Spiked Samples (Spike B Elements):

A post-spike B was conducted on Samples 02-03410 and 02-03421. Recovery values are listed for all analytes in the spike with concentrations > 20% of that in the sample. The recovery values were within the acceptance criterion of 75% to 125% for all AOIs. Analytes recovered at levels below the EQL are shown as bold.

Serial dilution (Percent Difference):

Five-fold serial dilution was conducted on Samples 02-03410 and 02-03421. Percent differences (%Ds) are listed for all analytes with concentrations ≥ EQL in the diluted sample. The %Ds were within the acceptance criterion of ±10% for all AOIs meeting the above requirement. Note, that the %Ds for sodium were obtained from the 5x/25x dilutions.

Other QC:

All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' by the IDL. Estimated quantitation limit (EQL) is equal to 10 x MLD.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 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). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
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 two.

	Run Date=	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002
	Multiplier=	26.4	26.6	133.2	27.1	135.3	27.3	136.4
	RPL/LAB #=	02-03410-PB	02-03410	02-03410 @5	02-03412	02-03412 @5	02-03411	02-03411 @5
Instr. Det. Limit (IDL)	Client ID=	*process blank*	*RX-01-04*		*RX-01-24*		*RX-01-24-Dup*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	[5.7]	6,380		6,160		6,190	
0.250	Ca	--	259		253		256	
0.015	Cd	--	34.3		33.4		33.8	
0.200	Ce	--	--		--		[5.5]	
0.020	Cr	--	108		106		106	
0.100	Eu	--	--		--		--	
0.025	Fe	--	10.7		10.7		10.8	
2.000	K	--	1,210		1,180		1,190	
0.050	La	--	[8.6]		[8.4]		[8.6]	
0.100	Mg	--	--		--		--	
0.050	Mn	--	[2.9]		[2.8]		[2.9]	
0.150	Na	50.9	over range	128,000	over range	124,000	over range	126,000
0.100	Nd	--	[18]		[18]		[19]	
0.030	Ni	--	234		229		232	
0.100	P	--	1,070		1,040		1,050	
0.100	Pb	--	105		104		106	
0.015	Sr	--	[1.5]		[1.4]		[1.4]	
0.050	Zr	--	[4.4]		[4.3]		[4.4]	
Other Analytes								
0.025	Ag	--	--		--		--	
0.250	As	--	--		--		--	
0.050	B	32.4	49.7		48.4		49.2	
0.010	Ba	--	[0.52]		[0.52]		[0.53]	
0.010	Be	--	--		--		--	
0.100	Bi	[14]	[6.9]		[5.6]		[6.8]	
0.050	Co	--	[2.3]		[2.4]		[2.4]	
0.025	Cu	--	9.45		9.17		9.33	
0.050	Dy	--	--		--		--	
0.030	Li	--	--		--		--	
0.050	Mo	--	28.7		28.2		28.4	
0.750	Pd	--	--		--		--	
0.300	Rh	--	--		--		--	
1.100	Ru	--	--		--		--	
0.500	Sb	--	--		--		--	
0.250	Se	--	--		--		--	
0.500	Si	[79]	[82]		[88]		[85]	
0.500	Sn	--	--		--		[17]	
0.500	Te	--	--		--		--	
1.000	Th	--	--		--		--	
0.025	Ti	--	--		--		--	
0.500	Tl	--	--		--		--	
2.000	U	--	--		--		--	
0.050	V	--	--		--		--	
0.500	W	--	[74]		[72]		[73]	
0.050	Y	--	--		--		--	
0.050	Zn	--	[2.6]		[2.6]		[2.7]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002
	Multiplier=	26.0	130.2	26.6	133.1	25.3	126.6
	RPL/LAB #=	02-03413	02-03413 @5	02-03413-DUP	02-03413-DUP @5	02-03414	02-03414 @5
Instr. Det. Limit (IDL)	Client ID=	*RX-02-4*		*RX-02-4-Dup*		*RX-02-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	5,250		5,340		5,300	
0.250	Ca	189		192		185	
0.015	Cd	28.8		29.3		29.1	
0.200	Ce	--		--		--	
0.020	Cr	70.8		71.7		74.0	
0.100	Eu	--		--		--	
0.025	Fe	[2.7]		[2.8]		[2.6]	
2.000	K	1,030		1,040		1,030	
0.050	La	[3.0]		[3.0]		[2.2]	
0.100	Mg	--		--		--	
0.050	Mn	[5.0]		[5.1]		--	
0.150	Na	over range	107,000	over range	108,000	over range	108,000
0.100	Nd	[6.3]		[6.0]		[6.1]	
0.030	Ni	199		202		201	
0.100	P	889		899		897	
0.100	Pb	84.1		84.4		84.1	
0.015	Sr	162		167		104	
0.050	Zr	[1.7]		[1.7]		[1.5]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	43.9		46.5		45.7	
0.010	Ba	--		--		--	
0.010	Be	--		--		--	
0.100	Bi	[5.8]		[5.0]		[4.7]	
0.050	Co	[2.2]		[2.1]		[2.1]	
0.025	Cu	7.88		7.92		7.83	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	24.6		24.9		24.8	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[77]		[79]		[73]	
0.500	Sn	[15]		[14]		[13]	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Ti	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[61]		[62]		[60]	
0.050	Y	--		--		--	
0.050	Zn	--		--		--	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002	10/1/2002
	Multiplier=	26.1	130.5	27.1	135.3	25.9	129.4
	RPL/LAB #=	02-03415	02-03415 @5	02-03416	02-03416 @5	02-03417	02-03417 @5
Instr. Det. Limit (IDL)	Client ID=	*RX-03-24*		*RX-04-4*		*RX-04-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	5,560		5,060		5,030	
0.250	Ca	230		177		172	
0.015	Cd	30.5		27.6		27.4	
0.200	Ce	--		--		--	
0.020	Cr	75.6		68.4		74.7	
0.100	Eu	--		--		--	
0.025	Fe	[2.7]		[2.7]		[2.6]	
2.000	K	1,080		976		965	
0.050	La	[4.6]		[2.0]		[2.2]	
0.100	Mg	--		--		--	
0.050	Mn	[2.7]		[3.7]		--	
0.150	Na	over range	112,000	over range	108,000	over range	107,000
0.100	Nd	[7.3]		[5.7]		[8.2]	
0.030	Ni	212		191		192	
0.100	P	947		849		852	
0.100	Pb	89.0		74.2		73.4	
0.015	Sr	[1.1]		187		105	
0.050	Zr	[1.6]		[1.6]		[1.4]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	44.6		44.6		42.6	
0.010	Ba	--		--		--	
0.010	Be	--		--		--	
0.100	Bi	[4.4]		[3.8]		[4.8]	
0.050	Co	[2.3]		[2.0]		[2.1]	
0.025	Cu	8.29		7.11		6.90	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	26.1		23.5		23.6	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[68]		[67]		[75]	
0.500	Sn	[15]		[14]		[15]	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Ti	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[65]		[59]		[59]	
0.050	Y	--		--		--	
0.050	Zn	--		--		--	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/1/2002	10/1/2002	10/1/2002	10/1/2002
	Multiplier=	27.2	135.9	25.4	126.8
	RPL/LAB #=	02-03418	02-03418 @5	02-03419	02-03419 @5
Instr. Det. Limit (IDL)	Client ID=	*RX-05-04*		*RX-05-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	5,250		5,290	
0.250	Ca	185		179	
0.015	Cd	28.4		28.6	
0.200	Ce	--		--	
0.020	Cr	62.5		58.0	
0.100	Eu	--		--	
0.025	Fe	[2.4]		[1.6]	
2.000	K	986		978	
0.050	La	[2.4]		[1.6]	
0.100	Mg	--		--	
0.050	Mn	[4.4]		[5.8]	
0.150	Na	over range	112,000	over range	109,000
0.100	Nd	[4.9]		[3.6]	
0.030	Ni	197		198	
0.100	P	869		877	
0.100	Pb	74.2		77.4	
0.015	Sr	174		105	
0.050	Zr	[2.0]		[1.5]	
Other Analytes					
0.025	Ag	--		--	
0.250	As	--		--	
0.050	B	46.1		45.8	
0.010	Ba	--		--	
0.010	Be	--		--	
0.100	Bi	--		--	
0.050	Co	[1.8]		[1.8]	
0.025	Cu	7.30		7.39	
0.050	Dy	--		--	
0.030	Li	--		--	
0.050	Mo	24.1		24.0	
0.750	Pd	--		--	
0.300	Rh	--		--	
1.100	Ru	--		--	
0.500	Sb	--		--	
0.250	Se	--		--	
0.500	Si	[64]		[66]	
0.500	Sn	--		--	
0.500	Te	--		--	
1.000	Th	--		--	
0.025	Ti	--		--	
0.500	Tl	--		--	
2.000	U	--		--	
0.050	V	--		--	
0.500	W	[59]		[60]	
0.050	Y	--		--	
0.050	Zn	[2.0]		--	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "mult near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002
	Multiplier=	26.5	26.7	133.5	26.3	131.3	26.4	131.8
	RPL/LAB #=	02-03421-PB	02-03421	02-03421 @5	02-03421-DUP	02-03421-DUP @5	02-03422	02-03422 @5
Instr. Det. Limit (IDL)	Client ID=	*process blank*	*RX-07-24*		*RX-07-24-Dup*		*RX-08-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	[3.9]	5,610		5,820		6,060	
0.250	Ca	--	185		189		189	
0.015	Cd	--	30.0		31.0		32.2	
0.200	Ce	--	--		--		--	
0.020	Cr	--	71.8		73.9		60.1	
0.100	Eu	--	--		--		--	
0.025	Fe	--	[5.4]		[4.5]		[3.9]	
2.000	K	--	1,120		1,150		1,190	
0.050	La	--	[3.1]		[3.2]		[2.7]	
0.100	Mg	--	--		--		--	
0.050	Mn	--	[3.1]		[3.2]		[8.8]	
0.150	Na	46.3	over range	117,000	over range	118,000	over range	122,000
0.100	Nd	--	[9.3]		[9.3]		[6.7]	
0.030	Ni	--	201		206		215	
0.100	P	--	925		948		984	
0.100	Pb	--	72.8		73.7		84.6	
0.015	Sr	--	104		109		129	
0.050	Zr	--	[2.7]		[2.8]		[2.2]	
Other Analytes								
0.025	Ag	--	--		--		--	
0.250	As	--	--		--		--	
0.050	B	30.5	49.2		53.1		52.9	
0.010	Ba	[0.58]	--		--		--	
0.010	Be	--	--		--		--	
0.100	Bi	[23]	[4.8]		[4.2]		[3.3]	
0.050	Co	--	[1.9]		[2.0]		[2.0]	
0.025	Cu	--	7.71		7.93		8.32	
0.050	Dy	--	--		--		--	
0.030	Li	--	--		--		--	
0.050	Mo	--	24.9		25.5		27.0	
0.750	Pd	--	--		--		--	
0.300	Rh	--	--		--		--	
1.100	Ru	--	--		--		--	
0.500	Sb	--	--		--		--	
0.250	Se	--	--		--		--	
0.500	Si	[57]	[68]		[75]		[77]	
0.500	Sn	--	--		--		--	
0.500	Te	--	--		--		--	
1.000	Th	--	--		--		--	
0.025	Ti	--	--		--		--	
0.500	Tl	--	--		--		--	
2.000	U	--	--		--		--	
0.050	V	--	--		--		--	
0.500	W	--	[59]		[61]		[68]	
0.050	Y	--	--		--		--	
0.050	Zn	--	[1.5]		[1.6]		[1.8]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002
	Multiplier=	27.0	135.0	27.0	134.8	26.6	132.9
	RPL/LAB #=	02-03423	02-03423 @5	02-03424	02-03424 @5	02-03425	02-03425 @5
Instr. Det. Limit (IDL)	Client ID=	*RX-09-24*		*RX-10-24*		*RX-11-4*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	5,850		5,710		5,750	
0.250	Ca	131		182		186	
0.015	Cd	25.9		31.0		30.4	
0.200	Ce	--		--		--	
0.020	Cr	93.2		76.8		72.3	
0.100	Eu	--		--		--	
0.025	Fe	[3.0]		[6.1]		[3.8]	
2.000	K	3,430		1,140		1,130	
0.050	La	--		[3.9]		[2.3]	
0.100	Mg	--		--		--	
0.050	Mn	[1.4]		--		[3.9]	
0.150	Na	over range	121,000	over range	118,000	over range	116,000
0.100	Nd	[3.2]		[12]		[4.4]	
0.030	Ni	212		207		204	
0.100	P	986		861		926	
0.100	Pb	62.6		72.8		82.7	
0.015	Sr	284		138		183	
0.050	Zr	[1.9]		149		[1.5]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	47.9		49.7		50.9	
0.010	Ba	--		--		--	
0.010	Be	--		--		--	
0.100	Bi	[3.1]		--		[3.0]	
0.050	Co	[2.0]		[2.0]		[1.9]	
0.025	Cu	7.78		7.96		7.71	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	26.4		25.6		25.1	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[55]		[61]		[63]	
0.500	Sn	--		--		--	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Ti	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[69]		[66]		[64]	
0.050	Y	--		--		--	
0.050	Zn	[1.5]		[2.3]		--	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002	10/2/2002
	Multiplier=	26.1	130.4	130.8	653.8	26.3	658.3
	RPL/LAB #=	02-03426	02-03426 @5	02-03427 @5	02-03427 @25	02-03428	02-03428 @25
Instr. Det. Limit (IDL)	Client ID=	*RX-11-24*		*AN-107-UFC*		*AN-107-UFC-Dup*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	5,740		3,290		3,220	
0.250	Ca	183		637		614	
0.015	Cd	30.7		50.1		49.8	
0.200	Ce	--		[27]		[23]	
0.020	Cr	73.5		116		113	
0.100	Eu	--		--		--	
0.025	Fe	[2.5]		834		820	
2.000	K	1,140		[1,500]		1,330	
0.050	La	[1.7]		[17]		16.1	
0.100	Mg	--		--		[4.1]	
0.050	Mn	[1.6]		224		223	
0.150	Na	over range	119,000	over range	202,000	over range	195,000
0.100	Nd	[4.6]		[57]		51.9	
0.030	Ni	205		423		404	
0.100	P	928		544		547	
0.100	Pb	83.6		237		228	
0.015	Sr	108		[6.0]		5.69	
0.050	Zr	[1.4]		[39]		39.0	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	46.0		[58]		58.9	
0.010	Ba	--		[3.7]		3.62	
0.010	Be	--		--		--	
0.100	Bi	[3.0]		--		--	
0.050	Co	[1.9]		--		[3.7]	
0.025	Cu	7.68		[16]		16.2	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	25.3		[29]		27.2	
0.750	Pd	--		--		[36]	
0.300	Rh	--		--		[9.9]	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[54]		[89]		[94]	
0.500	Sn	--		--		--	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Tl	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		[69]	
0.050	V	--		--		--	
0.500	W	[64]		[140]		138	
0.050	Y	--		[8.7]		[8.4]	
0.050	Zn	--		[16]		16.3	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

QC Performance 10/1/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-03413 & 02-03413-D	LCS/BS	02-03413 & 02-03413-MS	02-03410 + Post Spike A	02-03410 + Post Spike B	02-03410 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	1.7	99	103	103		3.9
Ca	1.6	101	97	100		
Cd	1.5	101	99	102		4.1
Ce		100	97		**100**	
Cr	1.3	104	100	105		7.1
Eu					**99**	
Fe		107	100	105		
K	1.3	100	90	101		
La		98	91		97	
Mg		105	101	109		
Mn		103	97	**107**		
Na	0.4 (b)	117	nr	nr		9.1(b)
Nd		97	89		96	
Ni	1.2	104	100	105		6.3
P	1.2	104	104	98		2.6
Pb	0.4	113	108	111		
Sr	2.9	104	nr	105		
Zr		119	106	105		
Other Analytes						
Ag				**97**		
As				**105**		
B	5.7	132	100	99		
Ba		102	92	101		
Be		98	96	**98**		
Bi		89	90	98		
Co				106		
Cu	0.5	107	99	104		
Dy					100	
Li		101	93	101		
Mo	1.5	105	97	103		
Pd					**107**	
Rh					**100**	
Ru						
Sb				**102**		
Se				**104**		
Si		167	64	115		
Sn					**100**	
Te					**104**	
Th		98	93		**100**	
Tl		102	93	98		
Tl				**100**		
U		101	93		**103**	
V		97	93	**97**		
W						
Y				101		
Zn		106	107	108		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

(a) = RPD <10% for Na; (b) Value for 5x/25x dilutions

QC Performance 10/2/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-03421 & 02-03421-D	LCS/BS	02-03421 & 02-03421-MS	02-03421 + Post Spike A	02-03421 + Post Spike B	02-03421 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	3.6	104	76	96		6.2
Ca	2.3	101	93	101		
Cd	3.5	102	92	104		4.8
Ce		100	97		**98**	
Cr	2.8	101	79	101		7.3
Eu					101	
Fe		105	96	103		
K	3.0	110	96	107		
La		102	95		102	
Mg		102	98	107		
Mn		104	97	**109**		
Na	1.2(b)	125	nr	nr		6.0(b)
Nd		103	94		102	
Ni	2.3	101	76	99		7.4
P	2.5	103	79	97		2.1
Pb	1.3	110	104	109		
Sr	4.4	105	nr	99		
Zr		112	100	103		
Other Analytes						
Ag				**96**		
As				**100**		
B	7.5	135	98	102		
Ba		103	93	100		
Be		97	95	**97**		
Bi		77	92	98		
Co				103		
Cu	2.8	106	97	103		
Dy					102	
Li		113	102	108		
Mo	2.4	102	94	102		
Pd					**94**	
Rh					**99**	
Ru						
Sb				**100**		
Se				**101**		
Si		169	109	110		
Sn					**93**	
Te					**100**	
Th		101	96		**101**	
Ti		101	92	97		
Tl				**98**		
U		105	96		**102**	
V		97	92	**96**		
W						
Y				**100**		
Zn		105	99	105		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

(a) = RPD <10% for Na; (b) Value for 5x/25x dilutions

ASR 6583 Final (2) - ~A0850 R. Hallen ASR-6583 ICP98 hi.XLS

Battelle
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Project No. 42365

Internal Distribution
File/LB

Date May 21, 2003

To R. T. Hallen

From L. R. Greenwood LRG

Subject Radiochemical Analyses for– ASR 6583

Samples of the AN-102 supernate were analyzed for ^{90}Sr, total alpha, and alpha emitters according to ASR 6583. The samples were acid digested in the hot cells according to procedure PNL-ALO-128. Aliquots of these preps were then delivered to the laboratory for analysis. The samples were prepared in two hot cell batches, but analyzed in the lab in one batch. Batch numbers are indicated on the report so that QC results can be associated with the samples in a given batch. Results are reported in uCi per gram of sample weight. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). No ^{137}Cs activity was observed in the separated strontium fractions and negligible levels of ^{90}Sr were found in the process and laboratory preparation blanks. The LCS and matrix spike recoveries were 94% and 96%, respectively. RPD values were 7% and 12% for two sample duplicates. The ^{90}Sr activities were well above the requested MRQ value of 0.15 uCi/ml in all cases.

Total Alpha

The total alpha activities were determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum ZnS alpha scintillation counters according to RPG-CMC-408. All of the samples showed alpha activities at or above the requested MRQ value of 7.24E-4 uCi/ml. Sample duplicates showed acceptable repeatability, with an RPD value of 4% for sample RX-07-24 and a MD value of 0.73 for sample RX-11-24, indicating that the results agree when counting statistics are taken into account. The LCS and matrix spike recoveries were 109% and 104%, respectively. No significant alpha activities were observed in the hot cell or lab blanks. The total alpha activities show good agreement with the sum of the alpha emitters, within expected uncertainty.

Total Alpha Energy Analyses

Aliquots of each sample were precipitation plated according to procedure PNL-ALO-496 and counted by alpha energy analysis according to procedure PNL-ALO-422. Four peaks were observed in all of the samples due to unresolved alphas from $^{239}Pu+^{240}Pu$, $^{238}Pu+^{241}Am$, $^{243}Cm+^{244}Cm$, and ^{242}Cm. Most of the individual alpha activities were above the requested MRQ value of 7.24E-4 uCi/ml and the sum of the alpha emitters is in good agreement with the total alpha results (within expected uncertainty). The sum of the alpha emitters is generally the best estimate of the total alpha activity. No significant alpha activities were observed in the hot cell or laboratory blanks. LCS and matrix spike recoveries were 102% and 108%, respectively. Duplicate results showed acceptable RPD values (< 20%) in all cases.

Review:
C Soderquist
5-21-03

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Laboratory -325 Building

01/09/03

Client : R. Hallen
ASR: 6583

Cognizant Scientist: L R Greenwood Date : 1/9/03

Concur : T Trang-le Date : 1/9/03

Procedure: PNL-ALO-476/408 for Sr-90
Reference Date: Dec. 30, 2002

Measured Activities (uCi/g) with 1-sigma error

RPL ID Client ID	Hot Cell Batch #	Sr-90 Error +/-
02-3410 PB Process Blank	1	5.85E-4 11%
02-3410 RX-01-04	1	2.86E+1 2%
02-3411 RX-01-24 DUP	1	2.91E+1 2%
02-3412 RX-01-24	1	2.71E+1 2%
RPD		7%
02-3413 RX-02-4	1	4.16E+0 3%
02-3413 DUP RX-02-4	1	4.59E+0 3%
RPD		10%
02-3414 RX-02-24	1	1.50E+0 5%
02-3415 RX-03-24	1	2.22E+1 2%
02-3416 RX-04-4	1	4.26E+0 3%
02-3417 RX-04-24	1	1.69E+0 5%
02-3418 RX-05-4	1	5.28E+0 3%
02-3419 RX-05-24	1	1.28E+0 6%

Measured Activities (uCi/g) with 1-sigma error

RPL ID Client ID	Hot Cell Batch #	Sr-90 Error +/-
02-3421PB Process Blank	2	<2.E-4
02-3421 RX-07-24	2	1.34E+0 6%
02-3421 DUP RX-07-24 DUP	2	1.59E+0 5%
RPD		17%
02-3422 RX-08-24	2	1.74E+0 5%
02-3423 RX-9-24	2	4.66E+0 3%
02-3425 RX-11-4	2	2.46E+0 4%
02-3426 RX-11-24	2	1.55E+0 5%
02-3426 DUP RX-11-24	2	1.37E+0 6%
RPD		12%
Reagent Spike		94%
Matrix Spike 3412		96%
Lab Blank		<2.E-4

Note: Sample 02-3424 (RX-10-24) was lost during lab processing and will be reanalyzed.

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Laboratory -325 Building

03/28/03
Rev. 1

Client : R. Hallen
ASR: 6583

Cognizant Scientist: L R Greenwood Date : 3/28/03

Concur : T Trang-Le Date : 3/28/03

Procedure: PNL-ALO-476/408 for Sr-90
Reference Date: Jan. 21, 2003

Measured Activities (uCi/g) with 1-sigma error

RPL ID Client ID	Sr-90 Error +/-
02-3424 RX-10-24	2.00E+0 5%
Reagent Spike	90%
Matrix Spike 00096	88%
Lab Blank	<5.E-6

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Laboratory -325 Building

01/23/03
Rerun

Client : R. Hallen
ASR: 6583

Cognizant Scientist: L R Greenwood Date : 1/23/03

Concur : T Trang-le Date : 1/24/03

Procedure: PNL-ALO-4001/422 for Alpha/AEA

Measured Activities (uCi/g) with 1-sigma error

RPL ID Client ID	Hot Cell Batch #	Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238+ Am-241 Error %	Cm-243+ Cm-244 Error %	Am-242m* Cm-242 Error %	Sum of Alpha Emitters Error %
02-3410 PB Process Blank	1	<6.E-5	3.84E-6 8%	1.62E-5 4%	1.20E-5 4%	<6.E-8	3.20E-5 ± 3%
02-3410 RX-01-04	1	7.80E-2 5%	2.39E-3 3%	7.09E-2 2%	2.77E-3 3%	3.01E-4 9%	7.64E-2 ± 2%
02-3411 RX-01-24 DUP	1	7.78E-2 5%	2.72E-3 3%	7.72E-2 2%	3.05E-3 3%	2.94E-4 9%	8.33E-2 ± 2%
02-3412 RX-01-24	1	7.37E-2 5%	2.31E-3 4%	7.20E-2 2%	2.65E-3 4%	2.85E-4 12%	7.72E-2 ± 2%
RPD MD		5% 0.38	16% 1.66	7% 1.23	14% 1.43	3% 0.10	7% 1.42
02-3413 RX-02-4	1	1.92E-2 11%	7.21E-4 6%	1.59E-2 2%	7.46E-4 6%	8.24E-5 17%	1.74E-2 ± 2%
02-3413 DUP RX-02-4	1	1.71E-2 13%	7.73E-4 6%	1.57E-2 2%	6.68E-4 7%	4.96E-5 24%	1.72E-2 ± 2%
RPD MD		12% 0.34	7% 0.41	1% 0.22	11% 0.60	50% 0.89	1% 0.28
02-3414 RX-02-24	1	1.70E-2 13%	7.79E-4 5%	1.62E-2 2%	8.60E-4 5%	7.70E-5 17%	1.79E-2 ± 2%
02-3415 RX-03-24	1	2.09E-2 11%	7.66E-4 5%	1.72E-2 2%	7.37E-4 6%	7.57E-5 17%	1.88E-2 ± 2%
02-3416 RX-04-4	1	8.72E-3 22%	1.31E-3 4%	1.62E-2 2%	7.04E-4 6%	7.56E-5 18%	1.83E-2 ± 2%
02-3417 RX-04-24	1	2.55E-2 10%	1.03E-3 5%	2.10E-2 2%	1.06E-3 5%	8.88E-5 17%	2.32E-2 ± 2%
02-3418 RX-05-4	1	1.94E-2 12%	1.45E-3 4%	1.80E-2 2%	7.85E-4 6%	6.92E-5 19%	2.03E-2 ± 2%
02-3419 RX-05-24	1	1.68E-2 13%	8.06E-4 5%	1.14E-2 2%	6.12E-4 6%	3.61E-5 25%	1.29E-2 ± 2%

		Measured Activities (uCi/g) with 1-sigma error					
RPL ID Client ID	Hot Cell Batch #	Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238+ Am-241 Error %	Cm-243+ Cm-244 Error %	Am-242m* Cm-242 Error %	Sum of Alpha Emitters Error %
02-3421PB Process Blank	2	<5.E-5	5.37E-7 24%	2.25E-6 10%	4.75E-7 23%	<2.E-7	3.26E-6 ± 9%
02-3421 RX-07-24	2	4.37E-2 7%	2.16E-3 4%	3.73E-2 2%	1.58E-3 4%	1.22E-4 16%	4.12E-2 ± 2%
02-3421 DUP RX-07-24 DUP	2	4.19E-2 7%	1.90E-3 6%	3.66E-2 2%	1.54E-3 6%	1.11E-4 23%	4.02E-2 ± 2%
RPD		4%	13%	2%	3%	9%	2%
02-3422 RX-08-24	2	2.70E-2 9%	1.53E-3 4%	2.52E-2 2%	1.09E-3 5%	1.12E-4 14%	2.79E-2 ± 2%
02-3423 RX-9-24	2	9.67E-3 18%	4.84E-4 9%	8.95E-3 2%	4.64E-4 9%	<3E-5	9.93E-3 ± 2%
02-3424 RX-10-24	2	5.81E-2 7%	1.78E-3 5%	4.55E-2 2%	1.85E-3 5%	1.61E-4 16%	4.93E-2 ± 2%
02-3425 RX-11-4	2	1.93E-2 12%	7.65E-4 8%	1.82E-2 2%	7.37E-4 7%	6.85E-5 24%	1.98E-2 ± 2%
02-3426 RX-11-24	2	1.82E-2 12%	7.56E-4 7%	1.61E-2 2%	8.48E-4 7%	8.36E-5 23%	1.78E-2 ± 2%
02-3426 DUP RX-11-24	2	1.38E-2 15%	7.87E-4 7%	1.68E-2 2%	8.35E-4 7%	7.09E-5 24%	1.85E-2 ± 2%
RPD MD		28% 0.73	4% 0.20	4% 0.75	2% 0.08	16% 0.25	4% 0.74
R. Spike		109%	102%				
MS3412		104%	108%				
R. Blank		<4.E-5	<3.E-5	<3.E-5	<3.E-5	<7.E-6	

*Cm-242 (0.45 y) is the daughter of Am-242 (16 h) and Am-242m (141 y).

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Revision 2 6/12/2003

Client: Rich Hallen

Date: 11/7/2002

Subject: Hydroxide Analyses for: AN-102 and AN-107

ASR: 6583- rev 2

VS 6/13/03

Direct sample aliquots of four **AN-102 Sr/TRU samples and two AN-107 DF (diluted feed)** were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228 and using a Brinkman 636 Auto-Titrator. A 0.1018 N NaOH solution was used as a standard and sample spike and the titrant was a 0.2098 M HCl prepared solution for the all the samples (see ChemRec_86 attached).

The attached Report Summary indicates good RPD on the OH molarity (1st inflection point) on the sample and replicate results. The results hydroxide results are also shown converted to ug/ml to be more in line with the analytical service request (ASR). The calculation spreadsheet also shows the equivalent weight to the volume analyzed, therefore results in ug/g can also be easily attained. The MRQ value required equivalent to 0.05M hydroxide concentration was 850 **ug/ml** and in all cases concentrations well above this value were detected and the RPD's were 9% or less except for sample 02-03413 which was at 30%; in this case very low titrant volumes increased the error on this sample. The hydroxide recovery on the standard was 100%, the matrix spike recovery on 02-3410 was 97% and the matrix spike recovery on 02-3427 was 100%.. No hydroxide was detected in the reagent blank. The second and third inflection points showed excellent RPD for all the samples.

Following is the report summary, the sample results calculated from the raw data, and the record file for the standardized acid and base used. Also included in this report are copies of the titration curves.

-- Rev- 2 issued for 2 minor issues, neither of which affected the data as reported.
issue 1 --- strictly editorial -- NIST KAP-- SRM 84k was used instead of 84j as stated on original Standard Prep File ChemRec_86. -- Molecular Wt. did not change in either SRM certificate.
issue 2 -- Although performed, the weight check support data on the 10mL pipet, used to standardized the 0.2M HCl, was not located in original Chem Rec 86 file. Recently, re-certification of this acid titrant was conducted and the comparative results were within 0.29%. Supporting data appears in ChemRec_97.--- see copy attached.

Prepared by: _____ Date: 6/12/03

Reviewed by: _____ Date: 6/13/03

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6583- rev2

WP# W63934

Hydroxide and Alkalinity Determination
Procedure: **PNL-ALO-228** Equip # **WB76843**

Report Summary for ASR # -- 6583- rev2

RPG #	Client ID		OH conc ug/mL	Concentration, moles / Liter: First Point	RPD	Second Point	RPD	Third Point	RPD
02-03410	RX-01-04		2.5E+03	0.15		1.29		0.87	
02-03410	RX-01-04	Rep	2.6E+03	0.15	3%	1.29	0.2%	0.87	0.4%
02-03411	RX-01-04-dup		2.4E+03	0.14		1.29		0.86	
02-03411	RX-01-04-dup	Rep	2.2E+03	0.13	9%	1.30	1%	0.88	3%
02-03413	RX-02-4		2.4E+03	0.14		1.11		0.74	
02-03413	RX-02-4	Rep	1.8E+03	0.11	30%	1.17	5%	0.72	3%
02-03417	RX-04-24		6.9E+03	0.40		1.07		0.73	
02-03417	RX-04-24	Rep	7.1E+03	0.42	3%	1.04	3%	0.72	1%
02-03427	AN-107-UFC		9.6E+03	0.56		1.81		1.49	
02-03427	AN-107-UFC	Rep	8.9E+03	0.52	7%	1.84	2%	1.49	0.1%
02-03428	AN-107-UFC-dup		9.5E+03	0.56		1.78		1.50	
02-03428	AN-107-UFC-dup	Rep	9.7E+03	0.57	2%	1.79	0.4%	1.51	1%
		MRQ Molarity	MRQ ug/mL						
OH conc (ug/mL) = M (g/L) * 17,000		0.05	8.5E+02						
Reag. Blk.1				0					
Standard 1				100%					
MS 02-03410	Matrix spike			97%					
MS 02-03427	Matrix spike			100%					

Note: Results are presented for the first, second, and third inflection points on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second and third points generally represent the carbonate and bicarbonate concentrations.

Analyst: [signature] 6/12/03

Reviewer: [signature] 6/13/03

KS 9/11/03

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6583- rev2
Client: Rich Hallen
WP# W63934

File: R:\radchem\hydroxide\asr 6583- rev2
Analysis Date: 10/11/2002
Revision Date: 11/7/2002

Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Analyst: 6/19/03

Equip # **WB76843** Lab Loc. 525

	Titrant	Molarity	Chem Rec#
Strong	HCl	0.2098	86
Weak	HCl		

Std. & Spike	Molarity
NaOH	0.1018

RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Diluted Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
02-03410	RX-01-04		na	0.200	0.2582	1.291	3	11.379	0.142	10.713	0.030	0.15	
02-03410	RX-01-04	Replicate	na	0.300	0.3867	1.289	4	10.913	0.220	10.244	0.046	0.15	3.23%
02-03411	RX-01-04-dup		na	0.200	0.2566	1.283	6	11.294	0.136	10.672	0.029	0.14	
02-03411	RX-01-04-dup	Replicate	na	0.200	0.2600	1.300	7	11.436	0.124	10.887	0.026	0.13	9.23%
02-03413	RX-02-4		na	0.200	0.2509	1.255	8	11.190	0.137	10.582	0.029	0.14	
02-03413	RX-02-4	Replicate	na	0.200	0.2506	1.253	9	11.296	0.101	10.762	0.021	0.11	30.25%
02-03417	RX-04-24		na	0.200	0.2499	1.250	10	11.578	0.386	10.509	0.081	0.40	
02-03417	RX-04-24	Replicate	na	0.200	0.2486	1.243	11	11.566	0.397	10.486	0.083	0.42	2.81%
02-03427	AN-107-UFC		na	0.200	0.2799	1.400	12	12.072	0.537	11.009	0.113	0.56	
02-03427	AN-107-UFC	Replicate	na	0.200	0.2791	1.396	13	11.785	0.500	10.792	0.105	0.52	7.14%
02-03428	AN-107-UFC-dup		na	0.200	0.2756	1.378	14	11.972	0.532	10.954	0.112	0.56	
02-03428	AN-107-UFC-dup	Replicate	na	0.200	0.2780	1.390	15	11.686	0.544	10.864	0.114	0.57	2.23%
Reag. Blk.1				5.00			1	5.553			OH % Recovery		
Standard 1	0.1018N NaOH			5.000	5.0142	1.003	2	12.291	2.415	7.950	0.5067	99.5%	
MS 02-03410	+ 2mL 0.1N NaOH			0.100	0.1287	1.287	5	12.074	1.009	10.769	0.2117	96.5%	MS
MS 02-03427	+ 2mL 0.1N NaOH			0.100	0.1389	1.389	16	12.050	1.226	10.73	0.2572	99.6%	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	007467-24	161301	Dec-02
4	007374-24	161303	Nov-02
7	007376-24	161302	Nov-02

Initial	pH 7.0 reading =	6.989
Continuing	pH 7.0 reading =	7.015

Performance checks using Balance # 360--01-06-037

Pipet #	Vol.	Wt.	Pipet #	Vol.	Wt.
F04171	5.00	5.0014	92501	0.200	0.1999
F04171	5.00	4.9987	92501	0.200	0.1995
F04171	5.00	5.0116	92501	0.200	0.1988

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6583- rev2 File: R:\radchem\hydroxide\asr

WP# W63934

Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: RPG 6/12/03

Titrant	Molarity
HCl	0.2098

RPG #	0	Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
02-03410	0	0.200	1.371	7.520	0.258	1.289		2.202	4.875	0.174	0.87	
02-03410	Replicat	0.300	2.068	7.410	0.388	1.292	0.24%	3.309	4.881	0.260	0.87	0.4%
02-03411	0	0.200	1.370	7.479	0.259	1.294		2.186	4.881	0.171	0.86	
02-03411	Replicat	0.200	1.366	7.554	0.261	1.303	0.65%	2.204	4.847	0.176	0.88	2.7%
02-03413	0	0.200	1.194	7.582	0.222	1.109		1.896	4.909	0.147	0.74	
02-03413	Replicat	0.200	1.216	7.383	0.234	1.170	5.34%	1.900	4.895	0.144	0.72	2.6%
02-03417	0	0.200	1.406	7.533	0.214	1.070		2.102	4.822	0.146	0.73	
02-03417	Replicat	0.200	1.390	7.557	0.208	1.042	2.68%	2.078	4.880	0.144	0.72	1.2%
02-03427	0	0.200	2.260	7.509	0.361	1.807		3.682	4.838	0.298	1.49	
02-03427	Replicat	0.200	2.257	7.513	0.369	1.843	1.95%	3.681	4.788	0.299	1.49	0.1%
02-03428	0	0.200	2.229	7.516	0.356	1.780		3.659	4.771	0.300	1.50	
02-03428	Replicat	0.200	2.248	7.506	0.357	1.787	0.41%	3.690	4.802	0.303	1.51	0.8%
						2-nd Recovered						
Standard 1		5.000	2.484	4.034	0.01448	2.8%	sample	3.277	3.965			
MS 02-03410		0.100	1.653	7.820			sample	2.120	4.688			
MS 02-03427		0.100	2.089	7.662			sample	2.831	4.791			

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2041 M HCl is on following page.

Chem Rec_86-rev-1 Prep date: 7/15/2002

Preparation and Standardization of 0.1 M, and 0.01M NaOH
and **Preparation and Standardization of 0.2 M HCl and dilutions**

WP# K88426 Prepared by: rg Swoboda

Request: *I need more NaOH and HCl solutions made up for the OH- analysis procedure --- rgs*
Preparation: Prepared ~ 0.1M NaOH and 0.2M HCl from reagent grade stock . Standardize the ~0.1M NaOH solution against NIST Potassium Acid Phthalate KHC8H4O4 (KAP) . Then prepare 0.2M HCl and standardize against the calibrated 0.1M NaOH. Do a verification check on all the subsequent dilutions of NaOH and HCl.

Standardization : Use NIST SRM 84k, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 186903
Technique used will be via hand-titration to the phenopthalein endpint. Project titration for about 20-25 mL of a 50 mL burrette.
----- KHC8H404 = 204.22 g/mole or mg/meq
Hence, ~20 mL * 0.1M NaOH = 2 meq. and ~2 meq of KAP = 204.22 mg/meq * 2 = ~ 400 mg KAP weighed on 5-place balance --- All preparations will be certified for 2 yrs beyond calibration date --- rgs.

0.1M NaOH and dilutions

Verification Test #	Wt. of **KAP**	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.22	Molarity Error +/- @ 1 s	% error
1	0.43336	20.85	0.10178		
2	0.49981	24.05	0.10176		
3	0.63432	30.50	0.10184		
	Standardized Average NaOH Molarity =		**0.10179**	0.00004	0.04%

10X cut of ~ 0.1M NaOH

Verification Test #	Wt. of **KAP**	Vol. Of ~ **0.01M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.22	Molarity Error +/- @ 1 s	% error
1	0.06842	33.05	0.01014		
2	0.07756	37.42	0.01015		
3	0.07141	34.42	0.01016		
	Standardized Average NaOH Molarity =		**0.01015**	0.00001	0.11%

0.2M HCl and dilutions

Titration Id.	aliquot of acid	Vol. of **0.10179M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	20.00	41.20	0.2097		
2	20.00	41.25	0.2099		
3	20.00	41.20	0.2097		
	Standardized Average HCl Molarity =		**0.2098**	0.00015	0.07%

Titration Id.	aliquot of acid	Vol. of **0.01015M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	20.00	41.05	0.0208		
2	20.00	41.10	0.0209		
3	20.00	41.00	0.0208		
	Standardized Average HCl Molarity =		**0.0208**	0.00003	0.12%

0.0052 M HCl was prepared by making an exact 40X cut of 0.2098 M HCl -- Error ~ 0.5%

Analyst/Date r. g. Swoboda 6/12/03 Expiration Date on Stds. **7/15/2004**

Chem Rec_97

Prep date: 06/11/03

Prep'd by: 6/12/03

Verif. By: 6/12/03

Preparation of Standardized 1.0 M NaOH Verification of 1 M HCl, 0.2 M HCl and 0.1 M NaOH

WP# [ ]

Requester: rg swoboda

Request: Need new NaOH and HCL standardized solutions prepared for Hydroxide procedure.

Preparation: Prepared~ 1.0M NaOH from reagent grade solid NaOH -- then standardize vs KAP.

Standardization : Use NIST SRM 84k, Potassium Acid Phthalate HC8H4O4 (KAP) --CMS# 186903. Dried KAP in oven @ 50 deg C for 30 min., cool and then weigh on 5-place balance. (from Chemrec_94)

Technique used will be via hand-titration to the phenopthalein endpint using Rainin motorized pipets in titration mode.

NaOH Molarity verification performed on 1 M NaOH prep. See Chemrec_94

Verification Test #	(target = 1g) Wt. of KAP	Vol. Of 1M NaOH to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.58126	2.88	0.98823	
2	1.19083	5.91	0.98660	
3	1.24888	6.18	0.98949	
4	1.22452	6.08	0.98615	
		4	**0.98762**	0.0015
			certified value	0.16%

~ 0.2M HCL Standardization Check

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	Molarity of Acid in Sample	Molarity Error +/- @ 1 s	% error
1	8.00	1.745	0.2154		
2	8.00	1.740	0.2148		
3	8.00	1.751	0.2162		
	Standardized Average Acid Molarity =		**0.2155**	0.00068	0.32%

~ 1.0M HCl Standardization

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	Molarity of Acid in Sample	Molarity Error +/- @ 1 s	% error
1	8.50	8.71	1.0120		
4	8.00	8.30	1.0247		
5	9.00	9.27	1.0172		
2	8.50	8.69	1.0097		
3	9.00	9.30	1.0205		
	Standardized Average Acid Molarity =		**1.0168**	0.00611	0.60%

~ 0.1M NaOH Standardization

Titration Id.	aliquot of base	Vol. of 0.2155 M HCl to neutralize	Molarity of Base in Sample	Molarity Error +/- @ 1 s	% error
1	10.00	4.77	0.1028		
2	10.00	4.73	0.1019		
3	10.00	4.77	0.1028		
	Standardized Average Acid Molarity =		**0.1025**	0.00050	0.49%

Recheck of 0.2m HCl

~ 0.2M HCL Standardization Check (from Chemrec 86)

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	Molarity of Acid in Sample	Molarity Error +/- @ 1 s	% error
1	8.00	1.694	0.2091		
2	8.00	1.693	0.2090		
3	8.00	1.697	0.2095		
	Standardized Average Acid Molarity =		**0.2092**	0.00026	0.12%

Date	Balance M&TE#		Calib. Expire Date:		Temp in deg C	cm3/g H2O
06/10/03	**360-06-01-017**		**August 31, 2003**		**24.0**	1.0027
Balance check	200 mg =	0.2000 g				
	2 g =	2.0001 g				
	20 g =	20.0009 g				

Rainin Pipet ID:	Delivery setting in mL	pipeted mass in g	Average mass (g)	1s Std. Dev.	Temp. corrected ave.	% of Nominal	% pipet error @ 1s
H30213	1.000	0.9975					
H30213	1.000	0.9956					
H30213	1.000	0.9968	0.9963	0.0010	0.9993	99.93%	0.0962%
H30973	8.000	8.0084					
H30973	8.000	8.0095					
H30973	8.000	8.0001	8.0060	0.0051	8.0276	100.34%	0.0640%

Date	Balance M&TE#		Calib. Expire Date:		Temp in deg C	cm3/g H2O
06/11/03	**360-06-01-017**		**August 31, 2003**		**22.5**	1.0023
Balance check	2 g =	2.0002 g				
	20 g =	20.0008 g				

Rainin Pipet ID:	Delivery setting in mL	pipeted mass in g	Average mass (g)	1s Std. Dev.	Temp. corrected ave.	% of Nominal	% pipet error @ 1s
H30973	4.75	4.7291					
H30973	4.75	4.7409					
H30973	4.75	4.7278	4.73260	0.0072	4.7453	99.90%	0.1521%

Battelle PNNL/RS&E/Inorganic Analysis ... ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 42365 / W63934
ASR#: 6618
Client: R. Hallen
Total Samples: 13 (liquid)

	First	Last
RPL#:	03-00086	03-00098
Client ID:	SS-01-4	AN-102 Sr/TRU decon-24hr

Sample Preparation: PNL-ALO-128 (SAL/vh)

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

Analyst: D.R. Sanders

Analysis Date (File): 10-29-2002 (A0855)
10-30-2002 (A0856)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

D.N. Olson 11/7/02
Preparer

11/8/02
Review and Concur

Thirteen aqueous samples submitted under Analytical Service Request (ASR) 6618 were analyzed by ICPAES. The samples were prepared by acid extraction per PNL-ALO-128 in the Shielded Analytical Laboratory (SAL) using a nominal 1.0 mL of sample and diluting to a final volume of approximately 25 mL in Teflon vials. Sample preparation and analysis were conducted in two separate batches.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (8 pages). Analytes of interest (AOIs) were specified in the ASR, and are listed in the upper section of the report. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified as AOIs are reported in the bottom section of the data report, but have not been fully evaluated for QC performance.

The results are given as µg/mL for each detected analyte, and have been adjusted for all laboratory processing factors. Minimum Reportable Quantity (MRQ) values were specified in the ASR for selected AOIs. To meet this requirement, method detection limits (MDL) for the ICPAES analyses need to be ≤ (MRQ ÷ 3). The required MRQ levels were met for all of the selected AOIs except for K (for all samples) and for Na (for the two AN-102 samples). However, since both K and Na were detected at levels well above the MRQ level of 75 µg/mL, this is not an issue in the present results.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For each extraction processing, a process blank, blank spike, matrix spike, and duplicate were prepared along with the samples. The blank spikes and matrix spikes were prepared using 1.5 and 0.5 mL respectively of multi-element spike solutions BPNL-QC-1A and -2A. One of the AOIs, europium, was not present in the combined spike solution.

Process Blank:

A process blank (reagents only) was prepared with both groups of samples. Except for Fe and Ni in the first process blank, the concentrations of all AOIs were within the acceptance criteria of <EQL (estimated quantitation level ≡ 10 x MDL) or <5% of the concentration in the samples. In the first process blank, iron was present at a level of ~18 µg/mL, representing from ~6 to 250% of that measured in the samples. Nickel was present at a level of ~13µg/mL, representing ~6% of that measured in the samples. Both analytes are suspected to be from tramp contamination in the SAL, probably from stainless steel. No analytes were detected above the MDL in the second process blank.

Blank Spike:

A blank spike (reagents and spike solution) was prepared with both groups of samples. Recovery values for both blank spikes were within the acceptance criterion of 80% to 120% for all AOIs. Analytes recovered at levels less than the EQL are shown as bold.

Duplicate RPD (Relative Percent Difference):

Duplicates were prepared for Samples 03-00086 and 03-00093. RPDs are listed for all analytes that had concentrations ≥ EQL. The RPDs for both duplicates were within the acceptance criteria of ±15% (±10% for Na) for all AOIs meeting the above requirement.

Laboratory Control Standard (LCS):

No LCS samples were prepared for analysis.

Matrix Spiked Sample:

Matrix spikes were prepared with Samples 03-00086 and 03-00093. Recovery values are listed for all analytes in the spikes that were measured above the EQL, and that had a spike concentration ≥ 20% of that in the sample. Analytes not meeting these requirements have either no recovery value listed (< EQL), or are listed as not recovered ("nr").

The recovery values for the first matrix spike (03-00086-MS) were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirements. The recovery values for 03-00093-MS, however, were outside the acceptance criteria for Cr, Fe, and Ni. As was the case for the earlier process blank, the over-recoveries (from 137 to 289%) are suspected to be from tramp contamination in the SAL.

Post-Spiked Samples (Spike A Elements):

A post-spike A was conducted with Samples 03-00086 and 03-00093. Recovery values are listed for all analytes in the spikes that had a concentration > 20% of that in the sample. The recovery values were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirement. Analytes not meeting the 20% requirement are listed as not recovered ("nr"). Analytes recovered at levels below the EQL are shown as bold.

Post-Spiked Samples (Spike B Elements):

A post-spike B was conducted with Samples 03-00086 and 03-00093. Recovery values are listed for all analytes in the spikes that had a concentration > 20% of that in the sample. The recovery values were within the acceptance criterion of 75% to 125% for all AOIs. Analytes recovered at levels below the EQL are shown as bold.

Serial dilution (Percent Difference):

Five-fold serial dilution was conducted on Samples 03-00086 and 03-00093. Percent differences (%Ds) are listed for all analytes that had a concentration ≥ EQL in the diluted sample. The %Ds were within the acceptance criterion of ±10% for all AOIs meeting the above requirement. Note, that the %Ds for sodium were obtained from the 5x/25x dilutions.

Other QC:

All other instrument-related QC tests for the AOIs passed within the appropriate acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis, unless specifically noted.
2) Instrument detection limits (IDL) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' by the IDL. Estimated quantitation limit (EQL) is equal to 10 x MLD.
3) Routine precision and bias is typically ±15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 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). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
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 two.

	Run Date=	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002
	Multiplier=	26.9	26.9	27.2	136.0	25.9	129.5
	RPL/LAB #=	03-00086-B	03-00086-B CHECK RUN	03-00086	03-00086 @5	03-00086-DUP	03-00086-DUP @5
Instr. Det. Limit (IDL)	Client ID=	*process blank*	*process blank - rerun*	*SS-01-4*		*SS-01-4-Dup*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	53.5	53.3	2,180		2,170	
0.250	Ca	--	--	420		418	
0.015	Cd	--	--	34.1		34.0	
0.200	Ce	--	--	[8.8]		[8.5]	
0.020	Cr	[4.5]	[4.5]	53.8		53.5	
0.100	Eu	--	--	--		--	
0.025	Fe	18.4	17.1	300		299	
2.000	K	--	--	917		914	
0.050	La	--	--	[7.2]		[7.1]	
0.100	Mg	--	--	--		--	
0.050	Mn	[5.5]	[5.6]	64.3		63.9	
0.150	Na	220	229	over range	124,000	over range	124,000
0.100	Nd	--	--	[23]		[23]	
0.030	Ni	13.7	12.8	278		275	
0.100	P	[3.7]	--	378		376	
0.100	Pb	--	--	128		126	
0.015	Sr	--	--	[3.5]		[3.5]	
0.050	Zr	--	--	18.6		18.5	
Other Analytes							
0.025	Ag	--	--	--		--	
0.250	As	--	--	--		--	
0.050	B	--	--	19.3		19.3	
0.010	Ba	[0.59]	[0.58]	[1.2]		[1.2]	
0.010	Be	--	--	--		--	
0.100	Bi	[14]	--	[2.8]		--	
0.050	Co	--	--	[2.5]		[2.5]	
0.025	Cu	--	--	11.0		11.0	
0.050	Dy	--	--	--		--	
0.030	Li	--	--	--		--	
0.050	Mo	--	--	18.5		18.4	
0.750	Pd	--	--	--		--	
0.300	Rh	--	--	--		--	
1.100	Ru	--	--	--		--	
0.500	Sb	--	--	--		--	
0.250	Se	--	--	--		--	
0.500	Si	--	--	[24]		[24]	
0.500	Sn	--	--	--		--	
0.500	Te	--	--	--		--	
1.000	Th	--	--	--		--	
0.025	Ti	--	--	--		--	
0.500	Tl	--	--	--		--	
2.000	U	--	--	--		--	
0.050	V	--	--	--		--	
0.500	W	--	--	[93]		[93]	
0.050	Y	--	--	[4.6]		[4.6]	
0.050	Zn	[6.3]	[6.2]	[9.6]		[9.3]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002
	Multiplier=	29.2	146.2	26.8	133.9	26.6	132.9
	RPL/LAB #=	03-00087	03-00087 @5	03-00088	03-00088 @5	03-00089	03-00089 @5
Instr. Det. Limit (IDL)	Client ID=	*SS-01-24*		*SS-02-4*		*SS-02-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	2,210		2,000		1,990	
0.250	Ca	428		308		311	
0.015	Cd	34.7		31.1		31.1	
0.200	Ce	[9.0]		--		--	
0.020	Cr	56.0		36.8		38.9	
0.100	Eu	--		--		--	
0.025	Fe	314		7.67		7.25	
2.000	K	929		874		870	
0.050	La	[7.5]		[1.4]		[1.4]	
0.100	Mg	--		--		--	
0.050	Mn	64.7		[5.7]		[1.7]	
0.150	Na	over range	129,000	over range	116,000	over range	112,000
0.100	Nd	[24]		[4.6]		[4.9]	
0.030	Ni	283		250		249	
0.100	P	384		345		343	
0.100	Pb	131		82.2		81.7	
0.015	Sr	[3.6]		209		93.8	
0.050	Zr	19.1		[3.5]		[3.0]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	19.0		18.0		17.7	
0.010	Ba	[1.3]		--		--	
0.010	Be	--		--		--	
0.100	Bi	--		--		--	
0.050	Co	[2.6]		[2.2]		[2.3]	
0.025	Cu	11.1		10.0		9.95	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	18.9		16.6		16.6	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[20]		[16]		[17]	
0.500	Sn	--		--		--	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Ti	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[96]		[84]		[85]	
0.050	Y	[4.7]		[1.8]		[1.8]	
0.050	Zn	[9.8]		[6.4]		[6.0]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002	10/29/2002
	Multiplier=	25.4	127.0	27.6	138.1	26.8	133.9
	RPL/LAB #=	03-00090	03-00090 @5	03-00091	03-00091 @5	03-00092	03-00092 @5
Instr. Det. Limit (IDL)	Client ID=	*SS-03-24*		*SS-04-24*		*SS-05-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	1,840		1,930		2,040	
0.250	Ca	293		308		336	
0.015	Cd	28.8		30.6		31.7	
0.200	Ce	--		--		--	
0.020	Cr	35.0		23.9		36.6	
0.100	Eu	--		--		--	
0.025	Fe	7.25		19.7		42.4	
2.000	K	808		849		893	
0.050	La	[1.4]		[2.0]		[1.6]	
0.100	Mg	--		--		--	
0.050	Mn	[2.2]		[13]		[11]	
0.150	Na	over range	108,000	over range	108,000	over range	113,000
0.100	Nd	[5.0]		[7.4]		[5.6]	
0.030	Ni	233		247		258	
0.100	P	320		342		354	
0.100	Pb	76.7		79.9		81.1	
0.015	Sr	92.9		116		108	
0.050	Zr	[2.9]		[4.6]		[3.8]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	18.4		17.5		18.4	
0.010	Ba	--		--		--	
0.010	Be	--		--		--	
0.100	Bi	--		--		--	
0.050	Co	[2.2]		[2.4]		[2.5]	
0.025	Cu	9.22		9.89		10.2	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	15.5		16.5		17.8	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[32]		[20]		[18]	
0.500	Sn	--		--		--	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Ti	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[79]		[84]		[86]	
0.050	Y	[1.6]		[2.1]		[1.6]	
0.050	Zn	[5.9]		[5.9]		[11]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/30/2002	10/30/2002	10/30/2002	10/30/2002	10/30/2002
	Multiplier=	26.4	26.1	130.3	26.2	131.0
	RPL/LAB #=	03-00093-B	03-00093	03-00093 @5	03-00093-DUP	03-00093-DUP @5
Instr. Det. Limit (IDL)	Client ID=	process blank	SS-06-24		SS-06-24-Dup	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	--	2,010		1,990	
0.250	Ca	--	298		299	
0.015	Cd	--	31.1		31.2	
0.200	Ce	--	--		--	
0.020	Cr	--	55.1		55.4	
0.100	Eu	--	--		--	
0.025	Fe	--	25.0		23.8	
2.000	K	--	2,860		2,820	
0.050	La	--	[1.6]		[1.7]	
0.100	Mg	--	--		--	
0.050	Mn	--	[2.6]		[2.4]	
0.150	Na	--	over range	114,000	over range	115,000
0.100	Nd	--	[5.7]		[6.3]	
0.030	Ni	--	258		259	
0.100	P	--	348		349	
0.100	Pb	--	94.9		96.0	
0.015	Sr	--	159		153	
0.050	Zr	--	[5.7]		[5.7]	
Other Analytes						
0.025	Ag	--	--		--	
0.250	As	--	--		--	
0.050	B	--	18.8		18.8	
0.010	Ba	--	--		--	
0.010	Be	--	--		--	
0.100	Bi	--	--		--	
0.050	Co	--	[2.2]		[2.3]	
0.025	Cu	--	10.0		9.97	
0.050	Dy	--	--		--	
0.030	Li	--	--		--	
0.050	Mo	--	17.0		17.1	
0.750	Pd	--	--		--	
0.300	Rh	--	--		--	
1.100	Ru	--	--		--	
0.500	Sb	--	--		--	
0.250	Se	--	--		--	
0.500	Si	--	[27]		[26]	
0.500	Sn	--	--		--	
0.500	Te	--	--		--	
1.000	Th	--	--		--	
0.025	Ti	--	--		--	
0.500	Tl	--	--		--	
2.000	U	--	--		--	
0.050	V	--	--		--	
0.500	W	--	[85]		[85]	
0.050	Y	--	[2.3]		[2.3]	
0.050	Zn	--	[6.8]		[6.7]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/30/2002	10/30/2002	10/30/2002	10/30/2002	10/30/2002	10/30/2002
	Multiplier=	26.4	131.8	26.6	133.1	26.0	129.8
	RPL/LAB #=	03-00094	03-00094 @5	03-00095	03-00095 @5	03-00096	03-00096 @5
Instr. Det. Limit (IDL)	Client ID=	*SS-07-24*		*SS-08-4*		*SS-08-24*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	1,840		1,910		1,920	
0.250	Ca	281		283		30€	
0.015	Cd	29.6		29.8		30.4	
0.200	Ce	--		--		--	
0.020	Cr	29.6		36.6		38.5	
0.100	Eu	--		--		--	
0.025	Fe	45.9		8.36		7.76	
2.000	K	796		819		815	
0.050	La	[1.6]		--		[1.3]	
0.100	Mg	--		--		--	
0.050	Mn	[6.7]		[6.0]		[2.0]	
0.150	Na	over range	108,000	over range	109,000	over range	111,000
0.100	Nd	[5.8]		[4.9]		[4.9]	
0.030	Ni	246		249		250	
0.100	P	323		333		334	
0.100	Pb	59.0		86.7		85.6	
0.015	Sr	110		186		105	
0.050	Zr	39.5		[3.4]		[3.0]	
Other Analytes							
0.025	Ag	--		--		--	
0.250	As	--		--		--	
0.050	B	18.0		18.6		18.2	
0.010	Ba	[0.32]		--		--	
0.010	Be	--		--		--	
0.100	Bi	--		--		--	
0.050	Co	[2.4]		[2.3]		[2.3]	
0.025	Cu	9.55		9.6		9.68	
0.050	Dy	--		--		--	
0.030	Li	--		--		--	
0.050	Mo	16.2		16.4		16.4	
0.750	Pd	--		--		--	
0.300	Rh	--		--		--	
1.100	Ru	--		--		--	
0.500	Sb	--		--		--	
0.250	Se	--		--		--	
0.500	Si	[17]		[21]		[20]	
0.500	Sn	--		--		--	
0.500	Te	--		--		--	
1.000	Th	--		--		--	
0.025	Tl	--		--		--	
0.500	Tl	--		--		--	
2.000	U	--		--		--	
0.050	V	--		--		--	
0.500	W	[79]		[80]		[81]	
0.050	Y	--		[1.7]		[1.7]	
0.050	Zn	[7.1]		[6.6]		[6.5]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

	Run Date=	10/30/2002	10/30/2002	10/30/2002	10/30/2002
	Multiplier=	26.1	653.7	27.3	681.5
	RPL/LAB #=	03-00097	03-00097 @25	03-00098	03-00098 @25
Instr. Det. Limit (IDL)	Client ID=	*AN-102 Diluted Feed*		*AN-102 Sr/TRU decon-24hr*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	8,800		8,690	
0.250	Ca	259		207	
0.015	Cd	38.1		37.7	
0.200	Ce	--		--	
0.020	Cr	151		117	
0.100	Eu	--		--	
0.025	Fe	31.0		[3.5]	
2.000	K	1,330		1,330	
0.050	La	[8.3]		[3.3]	
0.100	Mg	--		--	
0.050	Mn	[9.4]		[3.1]	
0.150	Na	over range	160,000	over range	154,000
0.100	Nd	[16]		[7.9]	
0.030	Ni	252		249	
0.100	P	1,170		1,160	
0.100	Pb	119		92.8	
0.015	Sr	[1.5]		177	
0.050	Zr	[5.3]		[1.4]	
Other Analytes					
0.025	Ag	--		--	
0.250	As	--		--	
0.050	B	32.3		30.3	
0.010	Ba	[1.5]		--	
0.010	Be	--		--	
0.100	Bi	[2.9]		[3.0]	
0.050	Co	[2.4]		[2.5]	
0.025	Cu	9.58		8.78	
0.050	Dy	--		--	
0.030	Li	--		--	
0.050	Mo	30.4		30.2	
0.750	Pd	--		--	
0.300	Rh	--		--	
1.100	Ru	--		--	
0.500	Sb	--		--	
0.250	Se	--		--	
0.500	Si	[58]		[54]	
0.500	Sn	--		--	
0.500	Te	--		--	
1.000	Th	--		--	
0.025	Ti	--		--	
0.500	Tl	--		--	
2.000	U	--		--	
0.050	V	--		--	
0.500	W	[77]		[76]	
0.050	Y	--		--	
0.050	Zn	[7.1]		[2.8]	

1) "--" indicates the value is < IDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated quantitation limit (EQL) = 10 times the MDL.

2) Overall error for values > EQL is estimated to be within ±15%.

3) Values in brackets [ ] are > MDL but < EQL, with errors likely to exceed 15%.

QC Performance 10/29/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	03-00086 & 03-00086-D	LCS/BS	03-00086 & 03-00086-MS	03-00086 + Post Spike A	03-00086 + Post Spike B	03-00086 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	0.1	98	99	107		2.9
Ca	0.5	99	101	103		3.6
Cd	0.4	101	102	105		3.0
Ce		94	91		**100**	
Cr	0.4	94	98	104		4.6
Eu					103	
Fe	0.4	94	101	105		4.9
K	0.3	104	99	108		
La		97	95		101	
Mg		101	102	109		
Mn	0.5	99	100	107		
Na	0.7 (b)	88	nr	nr		6.2 (c)
Nd		97	93		100	
Ni	0.9	94	98	105		4.9
P	0.5	102	105	102		1.1
Pb	1.1	107	107	108		
Sr		101	97	103		
Zr	0.7	107	102	102		
Other Analytes						
Ag				**94**		
As				**99**		
B	0.5	104	101	104		
Ba		99	93	101		
Be		93	96	**97**		
Bi		104		98		
Co				105		
Cu	0.5	103	103	104		
Dy					103	
Li		105	102	110		
Mo	0.5	100	98	102		
Pd					**95**	
Rh					**100**	
Ru						
Sb				**100**		
Se				**99**		
Si		104	106	112		
Sn					**91**	
Te					**104**	
Th		97	96		**103**	
Tl		98	94	98		
Tl				**99**		
U		98	96		**101**	
V		92	92	**95**		
W		**97**				
Y				102		
Zn		94	103	105		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

(a) = RPD <10% for Na; (b) = Value for 5x dilutions; (c) = Value for 5x/25x dilutions.

QC Performance 10/30/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	03-00093 & 03-00093-D	LCS/BS	03-00093 & 03-00093-MS	03-00093 + Post Spike A	03-00093 + Post Spike B	03-00093 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	0.9	101	92	101		4.1
Ca	0.2	99	93	102		
Cd	0.1	101	93	103		4.8
Ce		95	89		**98**	
Cr	0.5	98	173	101		6.8
Eu					**98**	
Fe	4.9	103	289	104		
K	1.4	106	86	103		6.0
La		96	86		**99**	
Mg		101	95	107		
Mn		103	112	**109**		
Na	0.8 (b)	105	nr	nr		6.8 (c)
Nd		96	84		**99**	
Ni	0.4	100	137	100		5.9
P	0.1	103	97	99		0.3
Pb	1.1	111	105	112		
Sr	3.4	101	nr	100		5.5
Zr		112	94	103		
Other Analytes						
Ag				101		
As				**99**		
B	0.3	106	92	103		
Ba		100	85	98		
Be		93	88	**95**		
Bi		99		95		
Co				103		
Cu	0.4	103	93	102		
Dy					99	
Li		108	92	103		
Mo	0.3	100	90	100		
Pd					**92**	
Rh					**98**	
Ru						
Sb				**98**		
Se				**97**		
Si		108	99	114		
Sn					**96**	
Te					**104**	
Th		95	86		**98**	
Tl		98	86	95		
Tl				**94**		
U		100	89		**103**	
V		93	86	**93**		
W		**96**				
Y				**98**		
Zn		102	219	105		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

(a) = RPD <10% for Na; (b) = Value for 5x dilutions; (c) = Value for 5x/25x dilutions.

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Project No. 42365

Internal Distribution
File/LB

Date February 10, 2003

To R. T. Hallen

From L. R. Greenwood LRG

Subject Radiochemical Analyses for AN-107 and AN-102 Diluted Feed – ASR 6618

Samples of the diluted feed from tanks AN-107 and AN-102 were analyzed for alpha emitters and ^{90}Sr activities according to ASR 6618. The samples were acid digested in two batches in the RPL hot cells according to procedure PNL-ALO-128. However, both hot cell batches were analyzed in the laboratory in one batch. Aliquots of these preps were then delivered to the laboratory for analysis, as described below. Results are reported in uCi/g. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). Negligible levels of ^{90}Sr were found in the process and laboratory preparation blanks. A few of the samples had small (<5%) corrections for 137Cs in the separated Sr fractions. The LCS and matrix spike recoveries were 90% and 89%, respectively. RPD values were very low for sample duplicates. The ^{90}Sr activities were well above the requested MRQ value in all cases.

Total Alpha /Alpha Energy Analysis

Aliquots of the acid digestions were directly plated according to procedure RPG-CMC-4001 and counted with alpha spectrometers according to procedure PNL-ALO-422. Since no chemical separations were performed, peaks could not be uniquely identified and are labeled with the possible combinations in the attached table. Duplicate analyses met the requirements of our QA plan either with RPD values below 20% or MD (mean difference) values less than 1.96 in cases where the results had high counting uncertainties. The LCS and matrix spike recoveries were 109% and 108%, respectively. The lab blank was clean; however, the hot cell blanks for both batches show some alpha activity. Generally, the hot cell blank activities are below the limit of our QA plan (< 5% of the sample activity). However, four cases where identified where the 5% blank limit was exceeded. This was for Cm-243+Cm-244 for a few samples in the first hot cell batch (SS-02-4, SS-02-24, SS-03-24, and SS-04-24). The Cm-243+244 value measured in the first prep blank was 3.90E-5 uCi/g

whereas four of the samples had Cm-243+244 activities ranging from 5.47E-4 to 7.18E-4, meaning that the activity in the blank ranged from 5.4% to 7.1% of the sample activities in these cases. To put this in perspective, the MRQ value was listed as 7.24E-4 uCi/ml (or about 5.8E-4 uCi/g if we use the measured density of about 1.25 g/ml). So the blank contamination was very low. The TRU removal process was also very effective for the samples with failed QC, reducing the Cm isotopes to very low levels, near the MRQ value. Other factors also need considered. The success criteria are evaluated using total alpha and/or sum of alpha data. The alpha activity is >90% Am-241. The Cm isotopes only contribute approximately 5% of the alpha activity. Also the AEA is never corrected for the hot-cell (prep) blank. Potential contamination of reagents, vials, etc. would result in a conservative alpha activity, since no blank subtraction or correction is made.

The WTP project was notified of this QC problem as soon as it was detected. Based on the information stated above, the project made the determination that the QC failure will not adversely impact the data analyses and reporting. This analytical report describes the failure and re-preparation and analysis will not be performed.

File: 03-00086.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

01/30/03

Client : R. Hallen

Cognizant Scientist: P R Heasmond Date : 1/30/03

Concur : T Trang-Le Date : 1/30/03

Reference Date: Jan. 21, 2003 for AEA
Procedure: PNL-ALO-4001 & 422 for AEA

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Alpha Energy Analysis U-234+ Np-237 ± 1s	 Pu-239+ Pu-240 ± 1s	 Pu-238+ Am-241 ± 1s	 Cm-243+ Cm-244 ± 1s	 (Am-242m)* Cm-242 ± 1s	 Sum of alpha emitters	Hot Cell Batch**
03-00086PB Process Blank	<2.E-7	2.67E-5 ± 5%	1.81E-4 ± 2%	3.90E-5 ± 4%	<5.E-7	2.47E-4 ± 2%	1
03-00086 SS-01-4	<4.E-5	1.07E-2 ± 4%	1.10E-1 ± 2%	3.51E-3 ± 7%	4.72E-4 ± 18%	1.25E-1 ± 2%	1
03-00086DUP SS-01-4	<4.E-5	8.85E-3 ± 4%	1.06E-1 ± 2%	4.10E-3 ± 6%	4.34E-4 ± 18%	1.19E-1 ± 2%	1
RPD MD		19% 1.67	4% 0.65	16% 0.85	8% 0.16	4% 0.85	
03-00087 SS-01-24	<3.E-5	9.81E-3 ± 3%	1.15E-1 ± 2%	3.68E-3 ± 5%	4.46E-4 ± 14%	1.29E-1 ± 2%	1
03-00088 SS-02-4	<2.E-5	1.02E-3 ± 7%	1.24E-2 ± 2%	5.47E-4 ± 9%	8.90E-5 ± 22%	1.41E-2 ± 2%	1
03-00089 SS-02-24	<2.E-5	1.80E-3 ± 5%	1.43E-2 ± 2%	6.50E-4 ± 8%	1.01E-4 ± 20%	1.69E-2 ± 2%	1
03-00090 SS-03-24	<2.E-5	1.41E-3 ± 5%	1.27E-2 ± 2%	5.00E-4 ± 9%	9.51E-5 ± 21%	1.47E-2 ± 2%	1
03-00091 SS-04-24	<2.E-5	1.41E-3 ± 6%	1.39E-2 ± 2%	7.18E-4 ± 8%	5.32E-5 ± 29%	1.61E-2 ± 2%	1
03-00092 SS-05-24	<2.E-5	1.79E-3 ± 5%	1.97E-2 ± 2%	7.89E-4 ± 7%	1.63E-4 ± 16%	2.24E-2 ± 2%	1
03-00093PB Process Blank	<2.E-7	5.61E-6 ± 9%	3.90E-5 ± 3%	2.51E-5 ± 4%	<2.E-7	6.97E-5 ± 2%	2
03-00093 SS-06-24	<2.E-5	4.54E-3 ± 3%	2.48E-2 ± 2%	1.07E-3 ± 6%	1.15E-4 ± 19%	3.05E-2 ± 2%	2
03-00093 DUP SS-06-24	<2.E-5	4.42E-3 ± 3%	2.38E-2 ± 2%	9.41E-4 ± 7%	1.64E-4 ± 16%	2.93E-2 ± 2%	2
RPD MD		3% 0.32	4% 0.73	13% 0.70	35% 0.72	4% 0.83	
03-00094 SS-07-24	<2.E-5	2.52E-3 ± 4%	1.81E-2 ± 2%	7.39E-4 ± 8%	1.09E-4 ± 20%	2.15E-2 ± 2%	2
03-00095 SS-08-4	3.66E-4 ± 19%	1.11E-3 ± 7%	1.29E-2 ± 2%	6.45E-4 ± 9%	9.14E-5 ± 24%	1.51E-2 ± 2%	2

File: 03-00086.xls

Measured Activities (µCi/g) with 1-sigma error

ALO ID Client ID	Alpha Energy Analysis U-234+ Np-237 ± 1s	 Pu-239+ Pu-240 ± 1s	 Pu-238+ Am-241 ± 1s	 Cm-243+ Cm-244 ± 1s	 (Am-242m)* Cm-242 ± 1s	 Sum of alpha emitters	Hot Cell Batch**
03-00096 SS-08-24	<2.E-5	1.59E-3 ± 5%	1.40E-2 ± 2%	6.96E-4 ± 8%	1.29E-4 ± 19%	1.64E-2 ± 2%	2
03-00096L Dup SS-08-24	<2.E-5	1.44E-3 ± 7%	1.29E-2 ± 2%	5.97E-4 ± 11%	1.01E-4 ± 29%	1.50E-2 ± 2%	2
RPD MD		10% 0.58	8% 1.44	15% 0.57	24% 0.37	9% 1.67	
03-00097 AN-102 Diluted Feed	<4.E-5	3.90E-3 ± 6%	7.62E-2 ± 2%	2.86E-3 ± 7%	3.54E-4 ± 20%	8.33E-2 ± 2%	2
03-00098 AN-102 Sr/TRU decon-24 hr.	<2.E-5	1.09E-3 ± 8%	2.53E-2 ± 2%	1.08E-3 ± 8%	1.29E-4 ± 25%	2.76E-2 ± 2%	2
Matrix Spike00096		108%					N/A
Blank Spike		109%					N/A
Blank	<2.E-7	<8.E-7	<2.E-6	<2.E-7	<2.E-7		N/A

*Cm-242 is in equilibrium with the parent isotopes Am-242 (16 h) and Am-242m (141 y).

File: 03-00086.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

01/30/03
Rev. 1

Client : R. Hallen
ASR: 6618

Cognizant Scientist: L R Greenwood Date : 1/30/03

Concur : T Trang-Le Date : 1/30/03

Procedure: PNL-ALO-476/408 for Sr-90
Reference Date: Jan. 21, 2003

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Hot Cell Batch*	Sr-90 Error +/-	ALO ID Client ID	Hot Cell Batch*	Sr-90 Error +/-
03-00086PB Process Blank	1	2.59E-2 ± 3%	03-00093PB Process Blank	2	<3.E-4
03-00086 SS-01-4	1	3.11E+1 ± 3%	03-00093 SS-06-24	2	2.75E+0 ± 4%
03-00086DUP SS-01-4	1	2.94E+1 ± 3%	03-00093 DUP SS-06-24	2	2.62E+0 ± 4%
RPD		6%	RPD		5%
03-00087 SS-01-24	1	3.00E+1 ± 3%	03-00094 SS-07-24	2	1.91E+0 ± 5%
03-00088 SS-02-4	1	3.35E+0 ± 4%	03-00095 SS-08-4	2	3.22E+0 ± 4%
03-00089 SS-02-24	1	1.55E+0 ± 6%	03-00096 SS-08-24	2	1.71E+0 ± 5%
03-00090 SS-03-24	1	1.58E+0 ± 5%	03-00096L Dup SS-08-24	2	1.66E+0 ± 5%
03-00091 SS-04-24	1	2.00E+0 ± 5%	RPD MD		3% 0.21
03-00092 SS-05-24	1	2.42E+0 ± 4%	03-00097 AN-102 Diluted Feed	2	2.62E+1 ± 3%
			03-00098 AN-102 Sr/TRU decon-24 hr.	2	2.57E+0 ± 4%
Matrix Spike 00096	N/A	89%			
Blank Spike	N/A	90%			
Blank	N/A	<2.E-4			

*The samples were prepared in the hot cells in 2 different batches and analyzed in the lab in 1 batch.

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Client: Rich Hallen

Date: 11/6/2002
Rev Date 6/17/2003

Subject: Hydroxide Analyses for: AN-107 Diluted Feed (6)
ASR: 6618-Rev-1 AN-102 Diluted Feed (1)

-- Rev- 1 issued for 2 minor issues, neither of which affected the data as reported.
issue 1 --- strictly editorial -- NIST KAP-- SRM 84k was used instead of 84j as stated on original Standard Prep File ChemRec_86. -- Molecular Wt. did not change in either SRM certificate.
issue 2 -- Although performed, the weight check support data on the 10mL pipet, used to standardized the 0.2M HCl, was not located in original Chem Rec 86 file. Recently, re-certification of this acid titrant was conducted and the comparative results were within 0.29%. Supporting data appears in ChemRec_97.--- see copy attached.

Direct sample aliquots of **six AN-107 DF (diluted feed) and one AN-102 DF (diluted feed)** were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228 and using a Brinkman 636 Auto-Titrator. A 0.1018 N NaOH solution was used as a standard and sample spike and the titrant was a 0.2098 M HCl prepared solution for the all the samples (see ChemRec_86 attached).
The attached Report Summary indicates good RPD on the OH molarity (1st inflection point) on the sample and replicate results. The hydroxide results are also shown converted to ug/g to be comparable to the MRQ units specified in the analytical service request (ASR). The MRQ value required equivalent to 0.05M hydroxide concentration was 850 **ug/ml** and in all cases concentrations well above this value were detected and the RPD's were 12% or less. The hydroxide recovery on the standards were 97% and 96% respectively, the matrix spike recovery on 03-0088 was 96% and the matrix spike recovery on 03-0097 was 88%.. No hydroxide was detected in the reagent blank. The second and third inflection points, generally associated with carbonate and bicarbonate respectively, showed excellent RPD's, less than 8% for all the samples. A fourth inflection point, somtimes associated with formate, was indicated on the curve (although not clearly visible) on four of the samples and associated replicates with RPD's 11% or less, therefore these results are also listed on the attached summary.
Following is the report summary, the calculation spreadsheet including the data from titration curves, and the record file for the standardized acid and base used. Copies of the titration curves are available upon request.

Rev-1

Prepared by: _____ Date: 6/17/03

Reviewed by: _____ Date: 6/17/03

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6618-Rev-1

WP# W63934

Hydroxide and Alkalinity Determination
Procedure: **PNL-ALO-228** Equip # **WB76843**

Report Summary for ASR # -- 6618-Rev-1

RPG #	Client ID		OH conc ug/ml	Concentration, moles / Liter: First Point	RPD	Second Point	RPD	Third Point	RPD	Fourth Point	RPD
03-0086	SS-01-4		5.9E+03	0.35		1.23		1.05			
03-0086	SS-01-4	Rep	6.7E+03	0.39	12%	1.20	2.5%	1.05	0.6%		
03-0087	SS-01-24		7.0E+03	0.41		1.19		1.04		0.56	
03-0087	SS-01-24	Rep	7.5E+03	0.44	7%	1.15	4%	1.01	3%	0.50	11%
03-0088	SS-02-4		6.2E+03	0.37		1.05		0.92		0.54	
03-0088	SS-02-4	Rep	6.1E+03	0.36	2%	1.05	0%	0.90	2%	0.56	4%
03-0089	SS-02-24		5.7E+03	0.34		1.06		0.92		0.58	
03-0089	SS-02-24	Rep	5.9E+03	0.35	2%	1.06	1%	0.93	1%	0.60	3%
03-0095	SS-08-4		6.5E+03	0.38		1.06		0.86			
03-0095	SS-08-4	Rep	6.3E+03	0.37	3%	1.05	1%	0.94	8.0%		
03-0096	SS-08-24		5.9E+03	0.35		1.05		0.91		0.59	
03-0096	SS-08-24	Rep	5.7E+03	0.34	3%	1.07	1.9%	0.91	1%	0.63	7%
03-0097	AN-102 Diluted feed		1.1E+04	0.64		1.48		0.88			
03-0097	AN-102 Diluted feed	Rep	1.1E+04	0.64	1%	1.49	0.4%	0.90	2%		
		MRQ Molarity	MRQ ug/ml								
OH conc (ug/mL) = M (g/L) * 17,000		0.05	8.5E+02								
Reag. Blk.1				0							
Standard 1				97%							
Standard 2				96%							
MS 03-0088	Matrix spike			96%							
MS 03-0097	Matrix spike			88%							

Note: Results are presented for the first, second, third, and sometimes a fourth inflection point on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second, third, and fourth points generally represent the carbonate, bicarbonate, and formate concentrations respectively.

Analyst: [signature] 6/17/03

Reviewer: [signature] 6/17/03

6/17/03

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6618-Rev-1 | File: R:\radchem\hydroxide\asr 6618-Rev-1
Client: Rich Hallen | Analysis Date: 11/5/2002
WP# W63934 | Report Date: 11/6/2002

Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Analyst: RJS 6/17/03

Equip # **WB76843** Lab Loc. 525

	Titrant	Molarity	Chem Rec#		Std. & Spike	Molarity
Strong	HCl	**0.2098**	**86**		NaOH	**0.1018**
Weak	HCl					

RPG #	Sample ID		Dilution Factor	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Diluted Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	OH 1st Equivalence pH	Found millimoles base	Molarity base	millimole RPD
03-0086	SS-01-4		na	0.200	0.2545	1.273	9	11.784	0.332	10.914	0.070	0.35	
03-0086	SS-01-4	Replicate	na	0.200	0.2549	1.275	10	11.484	0.376	10.119	0.079	0.39	12.43%
03-0087	SS-01-24		na	0.200	0.2563	1.282	11	11.867	0.391	10.851	0.082	0.41	
03-0087	SS-01-24	Replicate	na	0.200	0.2537	1.269	12	11.409	0.418	10.088	0.088	0.44	6.67%
03-0088	SS-02-4		na	0.200	0.2479	1.240	13	11.900	0.348	10.851	0.073	0.37	
03-0088	SS-02-4	Replicate	na	0.200	0.2494	1.247	14	11.929	0.341	10.855	0.072	0.36	2.03%
03-0089	SS-02-24		na	0.200	0.2499	1.250	16	11.756	0.322	10.697	0.068	0.34	
03-0089	SS-02-24	Replicate	na	0.200	0.2493	1.247	17	11.796	0.329	10.738	0.069	0.35	2.15%
03-0095	SS-08-4		na	0.200	0.2508	1.254	18	11.647	0.364	9.117	0.076	0.38	
03-0095	SS-08-4	Replicate	na	0.200	0.2513	1.257	19	11.613	0.353	10.624	0.074	0.37	3.07%
03-0096	SS-08-24		na	0.200	0.2497	1.249	20	11.852	0.333	10.788	0.070	0.35	
03-0096	SS-08-24	Replicate	na	0.200	0.2487	1.244	21	11.871	0.322	10.829	0.068	0.34	3.36%
03-0097	AN-102 Diluted feed		na	0.200	0.2612	1.306	22	11.804	0.611	11.078	0.128	0.64	
03-0097	AN-102 Diluted feed	Replicate	na	0.200	0.2630	1.315	23	12.141	0.607	10.979	0.127	0.64	0.66%
Reag. Blk.1				5.00	5.0029		6	3.957				**OH % Recovery**	
Standard 1	0.1018N NaOH			5.000	5.0531	1.011	7	12.348	2.354	10.368	0.4939	**97.0%**	
Standard 2	0.1018N NaOH			5.000	5.0422	1.008	8	12.353	2.341	10.402	0.4911	**96.5%**	
MS 03-0088	+ 2mL 0.1N NaOH			0.100	0.1191	1.191	15	11.789	1.101	10.788	0.2310	**95.7%**	MS
MS 03-0097	+ 2mL 0.1N NaOH			0.100	0.1310	1.310	24	12.331	1.016	10.883	0.2132	**87.8%**	MS

Buffer	ORION Lot #	CMS#	Expire Date
10	910110-GY-1	186909	Apr-04
4	910104-GX-1	186908	Mar-04
7	910107-GY-2	186907	Feb-04

Initial	pH 7.0 reading =	6.875
Continuing	pH 7.0 reading =	7.002

Performance checks using Balance # 360--01-06-037

Pipet #	Vol.	Wt.	Pipet #	Vol.	Wt.
F04171	5.00	5.0004	92501	0.200	0.20019
F04171	5.00	4.9981	92501	0.200	0.19989
F04171	5.00	5.0012	92501	0.200	0.20123

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 618-Rev-1 File: R:\radchem\hydroxide\asr

WP# W63934

Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: 6/17/03

Titrant	Molarity
HCl	0.2098

RPG #	0	Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	4-th Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
03-0086	0	0.200	1.501	7.618	0.245	1.226		2.500	4.735	0.210	1.05						
03-0086	Replicat	0.200	1.516	7.750	0.239	1.196	2.51%	2.521	4.730	0.211	1.05	0.6%					
03-0087	0	0.200	1.522	7.655	0.237	1.186		2.514	4.685	0.208	1.04		3.561	2.550	0.220	0.56	
03-0087	Replicat	0.200	1.510	7.769	0.229	1.146	3.51%	2.475	4.747	0.202	1.01	2.8%	3.474	2.616	0.210	0.50	11.4%
03-0088	0	0.200	1.351	7.672	0.210	1.052		2.226	4.672	0.184	0.92		3.129	2.613	0.189	0.54	
03-0088	Replicat	0.200	1.344	7.595	0.210	1.052	0.00%	2.199	4.730	0.179	0.90	2.3%	3.116	2.584	0.192	0.56	3.6%
03-0089	0	0.200	1.328	7.555	0.211	1.055		2.203	4.730	0.184	0.92		3.094	2.642	0.187	0.58	
03-0089	Replicat	0.200	1.343	7.636	0.213	1.064	0.79%	2.227	4.796	0.185	0.93	1.0%	3.161	2.633	0.196	0.60	2.6%
03-0095	0	0.200	1.379	7.345	0.213	1.065		2.203	4.835	0.173	0.86						
03-0095	Replicat	0.200	1.353	7.660	0.210	1.049	1.49%	2.246	4.689	0.187	0.94	8.0%					
03-0096	0	0.200	1.330	7.765	0.209	1.046		2.200	4.824	0.183	0.91		3.138	2.626	0.197	0.59	
03-0096	Replicat	0.200	1.338	7.623	0.213	1.066	1.89%	2.201	4.776	0.181	0.91	0.8%	3.170	2.560	0.203	0.63	6.6%
03-0097	0	0.200	2.026	7.499	0.297	1.484		2.868	4.890	0.177	0.88						
03-0097	Replicat	0.200	2.027	7.460	0.298	1.490	0.35%	2.887	4.854	0.180	0.90	2.1%					
						2-nd Recovered											
Standard 1		5.000	2.433	7.908	0.01657	3.3%	sample	2.499	4.101								
Standard 2		5.000	2.433	7.84	0.0193	3.8%	sample	2.507	3.905								
MS 03-0088		0.100	1.660	7.367			sample	2.122	4.520								
MS 03-0097		0.100	1.875	7.635			sample	2.454	4.747								

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2041 M HCl is on following page.

KS 6/11/03

Chem Rec_86-rev-1 Prep date: 7/15/2002

Preparation and Standardization of 0.1 M, and 0.01M NaOH
and **Preparation and Standardization of 0.2 M HCl and dilutions**

WP# K88426 Prepared by: rg Swoboda

Request: *I need more NaOH and HCl solutions made up for the OH- analysis procedure --- rgs*
Preparation: Prepared ~ 0.1M NaOH and 0.2M HCl from reagent grade stock . Standardize the ~0.1M NaOH solution against NIST Potassium Acid Phthalate KHC8H4O4 (KAP) . Then prepare 0.2M HCl and standardize against the calibrated 0.1M NaOH. Do a verification check on all the subsequent dilutions of NaOH and HCl.

Standardization : Use NIST SRM 84k, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 186903
Technique used will be via hand-titration to the phenopthalein endpint. Project titration for about 20-25 mL of a 50 mL burrette.
----- KHC8H404 = 204.23 g/mole or mg/meq
Hence, ~20 mL * 0.1M NaOH = 2 meq. and ~2 meq of KAP = 204.22 mg/meq * 2 = ~ 400 mg KAP weighed on 5-place balance --- All preparations will be certified for 2 yrs beyond calibration date --- rgs.

0.1M NaOH and dilutions

Verification Test #	Wt. of **KAP**	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s	% error
1	0.43336	20.85	0.10177		
2	0.49981	24.05	0.10176		
3	0.63432	30.50	0.10183		
	Standardized Average NaOH Molarity =		**0.10179**	0.00004	0.04%

10X cut of ~ 0.1M NaOH

Verification Test #	Wt. of **KAP**	Vol. Of ~ **0.01M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s	% error
1	0.06842	33.05	0.01014		
2	0.07756	37.42	0.01015		
3	0.07141	34.42	0.01016		
	Standardized Average NaOH Molarity =		**0.01015**	0.00001	0.11%

0.2M HCl and dilutions

Titration Id.	aliquot of acid	Vol. of **0.10179M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	20.00	41.20	0.2097		
2	20.00	41.25	0.2099		
3	20.00	41.20	0.2097		
	Standardized Average HCl Molarity =		**0.2098**	0.00015	0.07%

Titration Id.	aliquot of acid	Vol. of **0.01015M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	20.00	41.05	0.0208		
2	20.00	41.10	0.0209		
3	20.00	41.00	0.0208		
	Standardized Average HCl Molarity =		**0.0208**	0.00003	0.12%

0.0052 M HCl was prepared by making an exact 40X cut of 0.2098 M HCl -- Error ~ 0.5%

Analyst/Date 6/27/07

Expiration Date on Stds.
7/15/2004

Chem Rec_97 Prep date: 06/11/03

Preparation of Standardized 1.0 M NaOH Verification of 1 M HCl, 0.2 M HCl and 0.1 M NaOH

Prep'd by: 6/12/03

Verif. By: 6/12/03

WP# Requester: rg swoboda

Request: Need new NaOH and HCL standardized solutions prepared for Hydroxide procedure.

Preparation Prepared~ 1.0M NaOH from reagent grade solid NaOH -- then standardize vs KAP.

Standardization : Use NIST SRM 84k, Potassium Acid Phthalate, HC8H4O4 (KAP) --CMS# 186903. Dried KAP in oven @ 50 deg C for 30 min., cool and then weigh on 5-place balance. (from Chemrec_94)

Technique used will be via hand-titration to the phenopthalein endpint using Rainin motorized pipets in titration mode.

NaOH Molarity verification performed on 1 M NaOH prep. See Chemrec_94

Verification Test #	(target = 1g) Wt. of KAP	Vol. Of 1M NaOH to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.58126	2.88	0.98823	
2	1.19083	5.91	0.98660	
3	1.24888	6.18	0.98949	
4	1.22452	6.08	0.98615	
		4	**0.98762**	0.0015
			certified value	0.16%

~ 0.2M HCL Standardization Check

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	8.00	1.745	0.2154		
2	8.00	1.740	0.2148		
3	8.00	1.751	0.2162		
	Standardized Average Acid Molarity =		**0.2155**	0.00068	0.32%

~ 1.0M HCl Standardization

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	8.50	8.71	1.0120		
4	8.00	8.30	1.0247		
5	9.00	9.27	1.0172		
2	8.50	8.69	1.0097		
3	9.00	9.30	1.0205		
	Standardized Average Acid Molarity =		**1.0168**	0.00611	0.60%

~ 0.1M NaOH Standardization

Titration Id.	aliquot of base	Vol. of 0.2155 M HCl to neutralize	**Molarity of Base** in Sample	Molarity Error +/- @ 1 s	% error
1	10.00	4.77	0.1028		
2	10.00	4.73	0.1019		
3	10.00	4.77	0.1028		
	Standardized Average Acid Molarity =		**0.1025**	0.00050	0.49%

~ 0.2M HCL Standardization Check (from Chemrec 86)

Titration Id.	aliquot of acid	Vol. of 0.988 M NaOH to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s	% error
1	8.00	1.694	0.2091		
2	8.00	1.693	0.2090		
3	8.00	1.697	0.2095		
	Standardized Average Acid Molarity =		**0.2092**	0.00026	0.12%

Recheck of 0.2m HCl

Date	Balance M&TE#		Calib. Expire Date:		Temp in deg C	cm3/g H2O
06/10/03	**360-06-01-017**		**August 31, 2003**		**24.0**	1.0027
Balance check	200 mg =	0.2000 g				
	2 g =	2.0001 g				
	20 g =	20.0009 g				

Rainin Pipet ID:	Delivery setting in mL	pipeted mass in g.	Average mass (g)	1s Std. Dev.	Temp. corrected ave.	% of Nominal	% pipet error @ 1s
H30213	1.000	0.9975					
H30213	1.000	0.9956					
H30213	1.000	0.9968	0.99663	0.0010	0.9993	99.93%	0.0962%
H30973	8.000	8.0084					
H30973	8.000	8.0095					
H30973	8.000	8.0001	8.00600	0.0051	8.0276	100.34%	0.0640%

Date	Balance M&TE#		Calib. Expire Date:		Temp in deg C	cm3/g H2O
06/11/03	**360-06-01-017**		**August 31, 2003**		**22.5**	1.0023
Balance check	2 g =	2.0002 g				
	20 g =	20.0008 g				

Rainin Pipet ID:	Delivery setting in mL	pipeted mass in g.	Average mass (g)	1s Std. Dev.	Temp. corrected ave.	% of Nominal	% pipet error @ 1s
H30973	4.75	4.7291					
H30973	4.75	4.7409					
H30973	4.75	4.7278	4.73260	0.0072	4.7453	99.90%	0.1521%

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