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Assessment of Abnormal Process Conditions for Sr/TRU Removal Using AN-102 Tank Waste Samples

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Test Specification: 24590-WTP-TSP-RT-02-014
Test Plan: TP-RPP-WTP-218
Test Exceptions: None
R&T Focus Area: Pretreatment
Testing Scoping Statement: B-40

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-02-014 and Test Plan TP-RPP-WTP-218. 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
WTP R&T Support Project

Date

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 sodium permanganate (0.05M), for TRU removal, at 50°C and 1M additional sodium hydroxide. Section 5 of the *Research and Technology Plan* [(a)] identifies further research needs. One need shown is to determine optimal conditions for the Sr/TRU precipitation reaction (SOW Ref.: Sec. C.6 Std.2(a)(3)(ii)(B) and WBS No.: 1.2.10.03 and .05). Abnormal process condition assessment for the Sr/TRU removal process is addressed in Scoping Statement B-40, which is included in Appendix C of the *Research and Technology Plan*. In accordance with Scoping Statement B-40, Test Specification 24590-WTP-TSP-RT-02-014, and Test Plan TP-RPP-WTP-218, studies were conducted with AN-102 tank waste samples to assess the impact of abnormal process conditions on the Sr/TRU removal process.

Objectives

This report discusses results of testing designed to assess the impact of abnormal process conditions for removing Sr-90 and TRU from Envelope C tank supernatant destined for immobilization as LAW. Experiments were conducted with actual waste samples from Tank AN-102. The purpose of these studies was to determine the impact of various process conditions on the overall Sr/TRU decontamination and on the treated supernatant composition. Of specific interest is the importance of varying reagent concentrations; precipitation temperature; reaction time; and deviation in process sequence, such as no mixing for 24h after reagent addition, reversing the order of reagent addition, and split reagent additions.

Previous studies with actual tank samples have shown that reagent concentration, precipitation temperature, and reaction time have significant impact on the Sr/TRU removal process. These conditions were varied over a wider range, with samples of the same diluted waste, to allow direct comparison of the results and assess the impact of the process variables. The matrix of tests conducted with actual waste samples was developed from results of tests with waste simulant solutions. The results from the actual waste tests are provided in this report.

Conduct of Testing

Small-scale radioactive tests (~20-60 mL) were conducted with tank waste samples from AN-102. In all experiments, both $Sr(NO_3)_2$ and $NaMnO_4$ were added for Sr/TRU removal, because earlier tests demonstrated that additions of both reagents are required for Sr-90 and TRU removal. The baseline process is addition of 0.075M strontium followed by 0.05M permanganate to well-mixed waste heated to 50°C. The treated waste is mixed for 4h at 50°C, and then cooled to 25°C before filtration. In the waste treatment plant, the cooling is expected to take approximately 18h. The overall process time, from start of

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(a) Bechtel National, Inc. (BNI). 2002. *Research and Technology Plan*. 24590-WTP-PL-RT-01-002, Rev. 1, U.S. Department of Energy, Office of River Protection, Richland, WA.

reagent addition to transferring the first waste to the filtration receipt vessel, would be approximately 24h. In the tests discussed here, reagent addition was reduced to 0.01M and 0.03M to assess the impact on Sr-90 and TRU decontamination. One variation of the baseline process conditions involved addition of permanganate first, followed by Sr addition 24h later. In one experiment, the reagents were added as separate additions of 0.01M, then 24h later, 0.02M reagents were added. One experiment was conducted where reagents were added without mixing for a period of 24h, then the mixture was well mixed and sampled after 24h of mixing. Waste samples were treated and digested at 15°C, 25°C, and 50°C to assess the impact of temperature. All samples were filtered at 25 ± 5°C (ambient hot cell temperature). Most experiments were sampled 24h after reagent addition; selected experiments were sampled at 4h, and one was sampled after 6 days. Two samples of filtrate were also stored for a week and then re-filtered and analyzed to examine if post-filtration precipitation had occurred. Samples of the initial waste mixture and baseline treated wastes were also analyzed for ammonia to assess the fate of ammonia in the Sr/TRU removal process.

The decontamination of Sr-90 was examined as a function of treatment conditions. The decontamination factors (DFs) were very high (>10) for baseline treatment conditions. Reducing the amount of added Sr, reducing the reaction time, and reducing the temperature all resulted in significant reductions in Sr-90 DF. Reducing the amount of added nonradioactive Sr reduces the isotopic dilution; more Sr-90 remains in solution. Reducing the reaction time and reaction temperature increases the [Sr] in solution, which results in lower Sr-90 decontamination. Other abnormal process conditions examined (reverse addition, no mixing for 24 hours, and split reagent addition) had little impact on Sr-90 removal. The results are consistent with the mechanistic understanding of the Sr removal process, Sr-90 removal by isotopic dilution and $SrCO_3$ precipitation.

The decontamination of TRU elements was not nearly as sensitive to the changes in process conditions as the Sr-90 DF. At the lowest level of added permanganate, 0.01M, reduced TRU removal was noted, but 0.03M and 0.05M permanganate addition yielded similar TRU DFs. Reaction time, temperature, and other changes to process conditions had little impact on TRU removal. TRU removal was also much less sensitive to abnormal process operations than Sr-90 removal.

Results and Performance Against Objectives

The experimental data can be used to predict the loading of Sr-90 and TRU (sum of alpha) expected in the immobilized low-activity waste (ILAW). The levels of both Sr-90 and TRU are well below the ILAW requirements of 20 Ci/m^3 of Sr-90 and 100 nCi/g of TRU for the baseline Sr/TRU removal process. The Sr-90 glass loading was most sensitive to variation of the process conditions. Reduced Sr addition of 0.01M, reduced temperature of 15°C, and reduced reaction time of 4h resulted in high Sr-90 ILAW loading. However, conditions less than baseline, 0.03M reagent addition at 25°C for 24h, met the target of 50% below the Sr-90 and TRU ILAW limits.

Quality Requirements

Testing began in January 2003 and continued through May 2003 to assess the impact of abnormal process conditions. Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements by performing work in accordance with the 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 procedure QA-RPP-WTP-604. 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.

Issues

None.

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Figures

Tables

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-102 and AN-107) 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. Entrained solids and Sr/TRU precipitate are to be removed via crossflow filtration; Cs-137 is to be removed by ion exchange.

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°C), provided adequate Sr-90 and TRU removal to meet immobilized low-activity waste (ILAW) requirements. However, the waste treatment plant's process baseline for the Sr/TRU remains as demonstrated in earlier work at PNWD and the Savannah River Technology Center (Hallen et al. 2000a,b; Nash et al. 2000a,b): addition of 0.075M $Sr(NO_3)_2$ followed by 0.05M $NaMnO_4$ at 50°C and precipitate digest time of 4h at 50°C.

The Sr-90 decontamination factors (DFs) have been shown to increase significantly with increased temperature (Hallen et al. 2002a) and time (Hallen et al. 2003a). However, this increased Sr-90 decontamination was not a result of increased isotopic exchange, 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 et al. 2002c). Therefore, the kinetics of the Sr precipitation reaction was shown to be important; more than 4h was required to approach the final equilibrium concentration. The Sr concentration is known to increase as temperature is reduced 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, temperature, and time. Envelope C wastes have such high levels of soluble carbonate (>0.5M) that small changes in soluble carbonate concentration have 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. 2002b,c) was consistent. The TRU decontamination in the AN-102/C-104 waste blend occurred after the permanganate was added. The TRU removal exceeded the requirements for ILAW glass by a factor of 5. These results suggested that reaction temperature and time had much less impact on TRU removal. TRU removal was also not impacted by no mixing for 1h after reagent addition (Hallen

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

et al. 2003a). Hydroxide levels in the waste feed have been examined, from none to as much as 1M added, with little noticeable impact on either Sr-90 or TRU DF.

The objective of the work reported here was to use a sample of actual tank waste to determine the impact of various important process conditions on Sr-90 and TRU decontamination. The experiments discussed in this report were performed in radioactive hot cells using approximately 20- to 60-mL samples of waste with various amounts of added reagents, reaction temperatures, and changes to other process conditions.

The results from the assessment of abnormal process conditions on treatment of actual waste samples from AN-102 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 the test matrix, and provide the analytical data.

2.0 Test Conditions and Experimental Procedures

Experiments conducted to assess the impact of abnormal process conditions on Sr-90 and TRU removal used actual samples of AN-102 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 the appendices.

2.1 Description of Waste Samples

PNWD received 27 bottles of tank waste from Hanford's 222-S Laboratory that were taken by grab sampling of AN-102 from riser 022 over the period August 7 through 11, 2000. Eight of the samples were designated for process testing. Only one, partially full, 125-mL bottle of waste remained after all other process testing had been completed. This volume was insufficient to run all of the tests required for this study, so two earlier samples (Caustic A and B) that had been caustic adjusted for solubility studies (Burgeson et al. 2002) were combined with approximately 100 mL of as-received waste. The as-received waste sample was assumed to be similar in composition to the earlier AN-102 samples characterized by Urie et al. (2002). The composition of Caustic A and B was determined by chemical analyses, including free hydroxide determination by titration (Burgeson et al. 2002). The sodium and free hydroxide concentrations of the combined waste samples were calculated. The concentration and quantity of NaOH added to the waste to give the target of 5.5M Na and 0.3M additional free hydroxide were also calculated. The waste was diluted accordingly with an appropriate amount of 0.3M NaOH to give a diluted feed of approximately 5.5M Na and 0.3M additional hydroxide prior to reagent addition. Free OH^- in the diluted waste was determined by titration to be 0.4M.

2.2 Development of Test Conditions

Experimental conditions were defined using the results from earlier tests with AN-102 simulant (Hallen et al. 2003b) and actual waste studies (Hallen et al. 2003a). The addition of 0.02M $Sr(NO_3)_2$ and permanganate showed adequate Sr-90 and TRU decontamination (Hallen et al. 2002a). Based on these studies, minimum levels of reagent addition were set at 0.01M. The mid-point concentration was defined as 0.3M reagent addition, and the baseline conditions were set as the maximum levels. The added free hydroxide was the same in all tests, 0.3M. Three temperatures were evaluated, 15°C, 25°C, and 50°C. Not all test conditions were run at all temperatures. 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 matrix shown in Table 2.1 for AN-102. The target concentrations listed in the test matrix are based on the final composition after addition of all reagents. The matrix had tests designed to assess processing conditions such as no mixing for the first 24h after reagent addition, reverse reagent addition with a 24h delay, and split reagent addition. 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

Sample ID	**Temp. (± 5°C)**	**Sr(II)**	**Mn(VII)**	**Other/Comments**	**Stir**	**Sample Time**
AC-01	25	None	None	Initial waste	No	24h; 168h
AC-02	50	0.03M	0.03M	Filter at 25°C	Yes	4h; 24h
AC-02	25	No Additional	No Additional	Continue to mix AC-02 for 6 days at 25°C	Yes	168h
PP-02	25	No Additional	No Additional	Post-filtration precipitation	Yes	>48h after AC-02 24h
AC-03	50	0.01M	0.01M	Minimum reagent at 0.3M OH^-	Yes	24h
AC-04	50	0.075M	0.05M	Maximum reagent at 0.3M OH^-	Yes	24h
AC-05	25	0.03M	0.03M	Proposed optimized conditions	Yes	4h; 24h
PP-05	25	No Additional	No Additional	Post-filtration precipitation	Yes	>48h after AC-05 24h
AC-06	25	0.03M	0.03M	No mix for 24h	No[a]	48h
AC-07	25	0.01M	0.01M	Add 0.01M Sr and 0.01M Mn stir 24h/sample, then add 0.02M more	Yes	4h; 24h
AC-07A	25	+ 0.02M	+ 0.02M	Add 0.02M more reagent to exp. 7, stir 24h/sample	Yes	48h (after start of AC-07)
AC-08	25	0.03M	0.03M	Reverse addition, add Mn stir 24h, then add Sr[b]	Yes	48h
AC-09	15	0.03M	0.03M	Filter at 25°C	Yes	4h; 24h

(a) Mix sample after 24h of reaction.
(b) Reverse addition, but delay second reagent addition for 24h.

2.3 Experimental

The waste samples, AN-102 AR, Caustic A, and Caustic B, were mixed and diluted with 0.3M NaOH just prior to waste testing in the Shielded Analytical Laboratory (SAL) hot cells (in the Radiochemical Processing Laboratory). The small-scale experiments were conducted in 120-mL sample jars using between approximately 20 mL and 60 mL of the diluted tank waste. The reagents were added rapidly to the wastes with an adjustable pipette, in the order listed in Table 2.1 (from left to right), at the specified temperature, and mixed with magnetic stir bars when specified. The 25°C experiments were conducted at ambient hot cell temperatures ranging from 26°C to 28°C on the days of these tests. The experiments were conducted over a 2-week period. Samples were collected at the specified times, allowed to equilibrate to ambient hot cell temperature, and filtered with a 0.2-µm disposable syringe filter. Multiple samples of initial waste, AC-01, were taken as controls and 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.

Stock solutions of the reagents were prepared for addition to the waste. The tests used 0.22M, 0.44M, or 1.65M solutions of $Sr(NO_3)_2$; and 0.22M, 0.44M, or 1.1M solutions of $NaMnO_4$. This allowed the addition of reagents to remain constant at 1 mL per 20 mL of initial waste. For AC-07, the split reagent addition test, the volume of reagent was adjusted to account for the removal of two samples (4h and 24h) before the remaining 0.02M reagent was added. The actual quantities of waste and reagents used are given in Appendix A.

The test specification stated the temperature for these tests as 15, 25, and 50 ± 5°C. For the 15°C and 50°C experiments, a shaker table in the hot cells was connected to an external, temperature-controlled thermostatic bath. A bottle was filled with water and fitted with a thermocouple to both record the temperature and control the set-point on the heater block. No external heating or cooling was provided for the 25°C samples during this testing, because the ambient hot cell temperature, 26°C to 28°C, was within the temperature requirement of 25 ± 5°C. All samples were filtered at ambient hot cell temperature, which was within the specified filtration temperature range, also 25 ± 5°C.

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. The Sr-90 concentration was determined by chemical separation followed by beta counting. Sodium concentration was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES), as were the other metals listed in the test instructions. Selected samples were 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). Two samples were submitted for ammonia analysis by an ion selective electrode (ISE) method. All of the analytical results are included in Appendix B.

The quality control limits for matrix spike (MS) recovery have been defined as ± 25%. The MS recovery for Sr-90 in batch 2 of the analytical samples was only 60%, indicating that the Sr-90 in the batch 2 samples could be biased low. However, on examination of the data, no data bias appears present. Consequently, the low MS recovery is most likely a result of the high uncertainty (10-30%) in the counting data, which was caused by a high Sr-90 concentration in the initial, untreated waste, relative to the size of the MS. The quality control limits were met for recovery of Sr-90 from the batch 1 matrix spike (117%). The samples were prepared and digested in two batches (in the SAL), but were analyzed (treated, separated, and counted) as one analytical batch. The Sr-90 data for the control (AC-01-24) in batch 1 = 2.7 µCi/g and the control (AC-01-168) in batch 2 = 2.8 µCi/g are essentially the same; no low bias noted for the batch 2 data. Also, the PP-0# samples are repeat analyses of AC-0#-24, to determine if post-filtration precipitation has occurred. The initial samples (AC) are in batch 1, and the PP samples are in batch 2. Comparing AC-02-24 = 1.4 µCi/g and PP-02 = 1.6 µCi/g; AC-05-24 = 1.4 µCi/g and PP-05 = 1.5 µCi/g, no low bias is seen in the batch 2 data. All other data in batch 2 are consistent with batch 1 data, based on known/expected impact of the various treatment conditions. We believe the data are appropriate to use for assessing the impact of the process conditions, because no data bias is observed. The low spike recovery is most likely a result of small spike relative to the initial high Sr-90 concentration and associated higher uncertainty (5% error in counting total Sr-90, then subtracting a large initial Sr-90 level results in a small value for the MS with a high overall uncertainty).

2.5 Quality Assurance Requirements

PNWD implements the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project 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 PNWD's *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements are implemented through PNWD's *Conducting Analytical Work in Support of Regulatory Programs*.

Experiments that are not method-specific were performed in accordance with PNWD's 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 (M&TE) to obtain quality results.

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.

PNWD addressed internal verification and validation activities by conducting an Independent Technical Review of the final data report in accordance with PNWD's 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. This review procedure is part of the *WTPSP Quality Assurance Requirements and Description Manual*.

3.0 Results and Discussion

The results of experiments with AN-102 waste to assess the impact of abnormal process conditions for the Sr/TRU removal process are discussed in this section.

3.1 Decontamination of Sr-90

Each series of experiments using AN-102 waste involved multiple samples, analyzed as two separate analytical batches, and provided analytical results to determine the change in waste composition upon treatment. Three samples of the starting waste were collected at various times, filtered, and analyzed 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:

$$\mathrm{DF} = [\mathrm{A}]_\mathrm{i} /([\mathrm{A}\} * \mathrm{MD})$$

where $[\mathrm{A}]_\mathrm{i}$ 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. The Sr-90 removal was determined for all experiments, and the DF varied from >50 for the baseline treatment conditions (AC-04) to <3 for 0.01M reagent addition, 25°C treatment temperature, and 4h reaction (AC-07). The results are consistent with the mechanism for Sr-90 removal involving isotopic dilution and $SrCO_3$ precipitation.

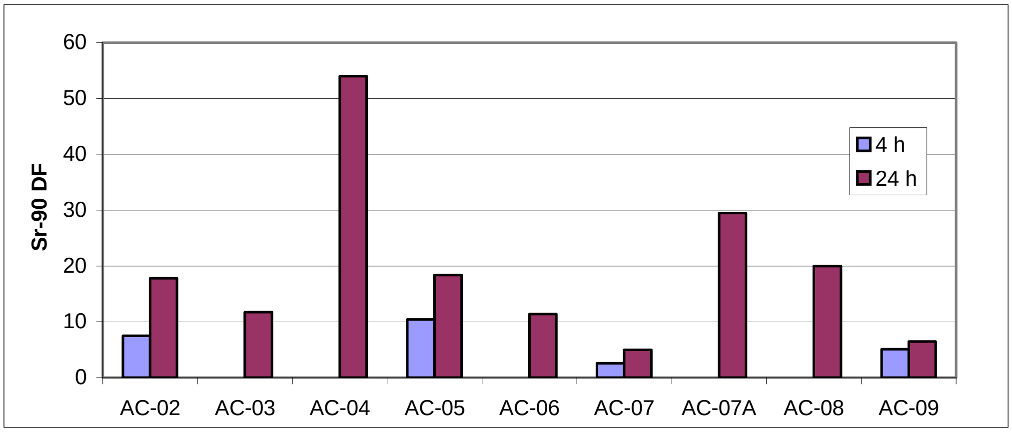

Figure 3.1. Strontium-90 Decontamination Factors for Treated AN-102 Samples as a Function of Reagent Concentration, Temperature, Time, and Process Variables

Isotopic dilution is based on the amount of added nonradioactive Sr. The highest level of reagent addition, 0.075M in AC-04, provides the greatest Sr-90 decontamination. Reducing the level of Sr addition to 0.03M (AC-02) and 0.01M (AC-03) results in proportionate reduction in Sr-90 DF. The amount of $SrCO_3$ precipitation is based on the reaction time and temperature, since the concentration of chelators and carbonate remain quite constant over the range of treatment conditions. The Sr-90 DFs are greatly increased by the solids digest time of 24h versus 4h and by increased treatment temperature of 15°C to 25°C (compare AC-09 to AC-05) and 25°C to 50°C (compare AC-05 to AC-02). The results are supported by the thermodynamic work of Felmy and Mason (2003), who studied the $SrCO_3$ solubility in detail.

The no-mix experiment (AC-06, no mixing for 24h of reaction followed by 24h of mixing) showed some reduction in DF compared to the same conditions (AC-05) with good mixing when the reagents were initially added. The equilibrium concentration of Sr was the same in both experiments, so the difference in DF must be a result of incomplete isotopic exchange, which may require more than 24h of mixing in the no-mix experiments because of the rapid $Sr(NO_3)_2$ addition (via adjustable pipette) and $SrCO_3$ precipitation, which only occurred in approximately 10% of the waste volume. The split reagent addition (AC-07A) did not reduce the Sr-90 DF when compared to addition of the same concentration of reagents all at once (AC-05). The reversed order of reagent addition with a 24h delay (AC-08) had little impact on Sr-90 DF (compare AC-08 to AC-05).

The time and temperature impacts on Sr-90 decontamination can be examined in more detail by comparing tests that were conducted with equivalent isotopic dilution ratios, 0.03M added $Sr(NO_3)_2$. Figure 3.2 shows the [Sr] as a function of both reaction time and temperature. Both the increase in temperature from 15°C to 50°C and the increase in reaction time from 4h to 24h resulted in a decreased [Sr]. The reduced [Sr] correlated directly to an increased Sr-90 DF. The highest Sr concentrations were

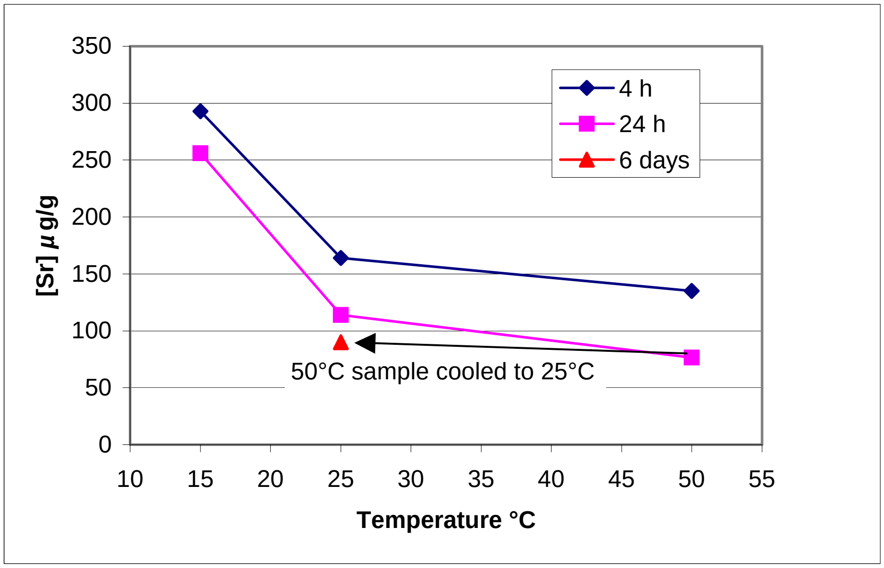

Figure 3.2. Total Strontium Solubility in the Treated AN-102 Samples as a Function of Temperature and Time (0.03M Reagent Addition)

noted for the 15°C experiments, two to three times higher than at 25°C and 50°C. The high [Sr] is likely a result of both the increased solubility at lower temperatures (retrograde solubility, Felmy and Mason 2003) and the slower precipitation kinetics at lower temperature. The filtration temperature must also be taken into consideration, since it will be 25°C in the plant regardless of the precipitation/reaction temperature. Thus, given enough time to reach equilibrium, the Sr will be the same for any precipitation temperature.

A test was conducted with the sample treated at 50°C for 24h to examine the change in [Sr] on cooling and allowing the mixture to equilibrate at 25°C for 6 days (25°C data point at 6 days). This test represents a potential plant scenario where the filtration circuit was plugged and not available to start the filtration immediately after precipitation and cooling was complete. After 6 days, the sample had a [Sr] of 90 μg/g, a slightly higher [Sr] than the sample at 50°C and 24h but lower than the sample at 25°C and 24h. This sample is probably most representative of the final equilibrium concentration of [Sr] to be expected in the filtrate at 25°C. The slow precipitation kinetics of $SrCO_3$ is consistent with the observation of Felmy and Mason (2003) that $SrCO_3$/chelator mixtures are slow to equilibrate for complex mixtures representative of tank waste solutions.

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 3.3. The DFs were significantly higher (>4) when more than 0.01M permanganate (AC-03 and AC-07) was added. At 50°C, 0.03M permanganate (AC-02) and 0.05M permanganate (AC-04) gave quite similar results, suggesting that reagent levels over 0.03M are not necessary for high TRU decontamination.

Treatment temperature, reaction time, and variation of the process scheme resulted in very little reduction in TRU DF. Little impact on TRU DF was noted in earlier tests with AN-102 between 25°C and 50°C (Hallen et al. 2002a). Reaction time also had little impact on TRU DF for 4h and 24h reaction (Hallen

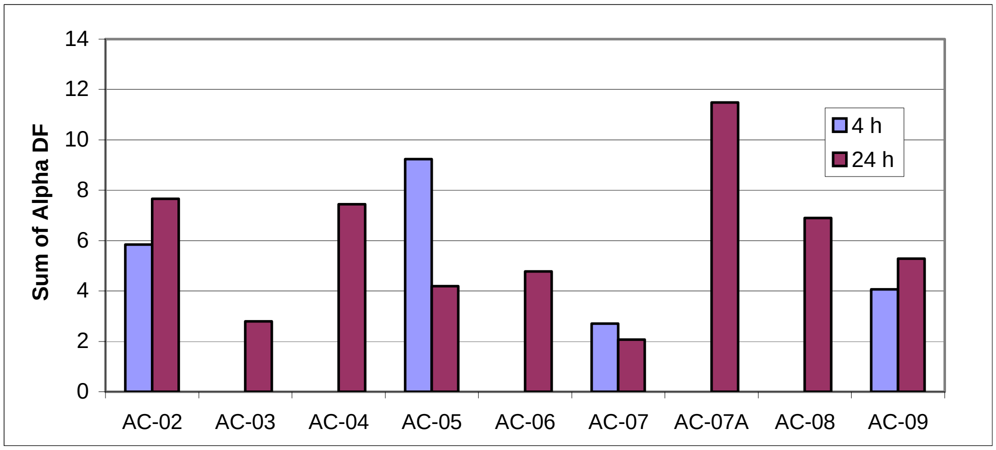

Figure 3.3. TRU (Sum of Alpha) Decontamination Factors for Treated Samples as a Function of Reagent Concentration, Temperature, Reaction Time, and Process Variables

et al. 2003a). The split addition experiment, where the 0.03M permanganate was added as two separate additions, first 0.01M, then 0.02M 24h later (AC-07A), gave the highest level of decontamination. No mixing for the first 24h (AC-06) appeared to have only a small impact (reduction), but DFs are still within the range of 4-6 noted for earlier experiments with AN-102. No mixing for 1h of reaction had no impact on TRU DF in studies by Hallen et al. (2003a). Reversing the order of reagent addition with a 24h delay (AC-08) appeared to have no impact on TRU DF.

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 3.1 shows the composition of the AN-102 starting waste, in μg/g, and the percent change that occurred for the various treatment conditions.

The 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 Al and P both 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 from the supernatant with treatment. The amount removed is relatively small, approximately 20%, and changes in treatment conditions appear to have only a minor impact on the Cr removal. Cr removal was not increased by the highest levels of reagent addition (baseline conditions).

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. The amount of Ca removed varies with the amount of added Sr; more Sr addition results in more Ca removal. This is likely a result of the competition of Sr and Ca for the complexing agents (EDTA/HEDTA) and precipitation of calcium carbonate. The changes in [Sr] were most impacted by time and temperature. Most other elements show little change with respect to changes in process conditions.

Two samples (AC-01-24h and AC-02-24h) from these tests were also analyzed for ammonia using an ISE method. Results gave similar estimated levels of ammonia in the waste and after treatment. It appears that ammonia does not react (is not oxidized) nor does it volatilize from the reaction mixture during the Sr/TRU removal process. This is consistent with model compound studies that showed that oxidation of glycine, a potential chelator aging product, gave oxalate and ammonia as the oxidation products (Gauger and Hallen 2001). The ammonia, a product of glycine oxidation, was not oxidized by permanganate but remained in the product mixture.

Table 3.1. Chemical Composition Data from ICP-AES for AN-102 Samples

Analyte	Initial Waste	0.03M Reagents and 50°C			0.01M and 50°C	0.075MSr, 0.05M MnO_4^- 50°C	0.03M and 25°C		No Mix, 25°C, 24h	0.01M and 25°C		Add 0.02M More to 7	Reverse Addition, 25°C	0.03M and 15°C	
		AC-02-4	AC-02-24	AC-02-168	AC-03-24	AC-04-24	AC-05-4	AC-05-24	AC-06-48	AC-07-4	AC-07-24	AC-07-48	AC-08-48	AC-09-4	AC-09-24
	Average (µg/g)	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal	% Removal
Al	5000	1	-2	-8	2	2	6	5	-2	5	5	0	-1	0	0
Ca	224	32	32	23	22	52	35	35	12	21	22	31	26	24	25
Cd	27.7	2	-4	-8	2	1	5	6	-2	6	6	-2	-2	-1	0
Co	[2.0]	-4	-4	-4	8	2	1	7	12	22	12	-10	1	-15	-15
Cr	80.8	17	31	22	15	13	26	20	28	21	19	9	7	12	11
Cu	11.2	1	-12	-8	-9	3	2	6	0	-2	4	0	6	0	1
Fe	9.7	73	76	70	77	74	73	77	71	76	68	69	78	73	72
K	1010	-5	-6	-7	5	0	5	5	5	15	7	-6	0	-4	-6
La	6.3	76	>78	>77	73	>77	>78	>79	74	69	54	>76	>77	76	69
Mn[(a)]	1.7	31	22	-34	55	59	-109	24	-69	-394	23	45	61	10	50
Mo	23.5	-1	-3	-10	3	2	6	5	-2	7	6	-1	-2	-2	-2
Na	101000	0	-2	-10	2	2	6	4	-3	4	4	-1	-3	-2	-2
Nd	[12]	>63	>60	58	61	>59	>61	>61	>57	>63	43	47	>58	44	37
Ni	189	-1	-3	-9	2	2	6	5	-2	5	5	-1	-3	-2	-2
P	814	1	-2	-8	2	3	7	5	-2	5	5	-1	-2	-1	-1
Pb	72.7	30	22	6	11	24	24	24	15	15	13	11	20	29	26
Sr[(a)]	[1.3]	-11319	-6362	-7521	-4730	-7529	-13854	-9600	-9603	-17221	-10373	-10049	-9330	-24794	-21650
W	59.5	1	-4	-3	1	7	7	7	3	4	6	2	2	6	1
Zr	[3.4]	69	81	86	84	85	72	76	79	67	64	71	75	65	62

(a) Reagent containing this element was added to some samples during testing, which resulted in increased concentration (a negative percent removal). The small negative percent removal values reported for other elements are consider to be zero within experimental and analytical error, since these elements were not added during the treatment process.

Note: ># = 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%.

3.4 Examination of Treated Samples for Evidence of Post-Filtration Precipitation

After the tests were completed, the filtrate samples that remained upon removal of the analytical samples were visually examined over approximately 1 week for evidence of post-filtration precipitation. Three different types of behavior were noted. In most samples, transparent solids were observed forming at the air-liquid interface. In one sample, AC-07-4h, a brown "bath tub" ring was noted at the air-liquid interface. In the samples from the 15°C experiment, AC-09-4 and 24h, white solids were noted at the bottom of the vial.

The transparent solids forming at the air-liquid interface are likely a result of evaporation. No chemical analyses were performed on these solids, but, visually, they are similar in appearance to oxalate and phosphate crystals that have been noted in the past. The fact that the transparent solids only form at the interface suggests the samples are near saturation and only a small amount of evaporation causes some solids to precipitate.

The brown "bath tub" ring noted for sample AC-07-4h after 5 days is most likely from Mn precipitation. The chemical analysis of this sample showed elevated levels of soluble Mn, approximately four times higher than in the initial waste. The elevated level of soluble Mn for low permanganate addition, 0.01M, is likely a result of incomplete oxidation of the organic complexant in solution. However, given time and exposure to air, the organic in the Mn-complex is oxidized and precipitates from solution. The amount of precipitation, although visible (dark ring on bottle), is likely less than 20 ppm (based on total sample weight), since the Mn concentrations are so low, <10 μg/g. After 24h of reaction for the AC-07 sample, the Mn was reduced to approximately 20% less than in the initial waste. This sample showed no distinct brown ring. Examining the Mn percent removal data (Table 3.2), sample AC-05-4h also showed increased [Mn], approximately two times higher than in the initial waste. AC-05-24h showed approximately 20% Mn removal. There was no visible brown ring in AC-05-4h, most likely because only 2 ppm of Mn solids are expected to form, less than can be visually observed.

Examining the composition of the 15°C test samples, both samples showed high [Sr], 250-300 μg/g. The final equilibrium [Sr] at ambient hot cell temperature, ~25°C, is approximately 100 μg/g. The white solids noted in these samples are most likely $SrCO_3$, and are in significant amounts (~300 ppm), since the [Sr] was so high in the original samples. These solids would contain significant Sr-90 from the additional $SrCO_3$ precipitation.

In addition to the visual observation, two filtrate samples, AC-02-24h and AC-05-24h, were chemically analyzed to determine if post-filtration precipitation occurs after filtration for removal of the Sr/TRU precipitate. Similar chemical analyses were conducted on the filtrate samples collected after 24h of reaction and after they had remained in the hot cell for 5 or 6 days, then were re-filtered and analyzed. Comparing the Sr-90 and TRU DFs of these samples, shown in Figure 3.4, very little change has occurred with no post-filtration precipitation. In examining the ICP-AES data in Table 3.2, the major difference between the samples taken at 24h, then approximately 6 days later, appears to be from evaporation. Elements that are not expected to precipitate, like K and P, have increased in this sample.

Table 3.2. Comparison of Supernatant Composition (ICP-AES Data) for Initial Treated Samples (AC Samples) and After Extended Sample Storage Time (PP Samples)

Analyte	AC-02-24 (µg/g)	PP-02 (µg/g)	Analyte	AC-05-24 (µg/g)	PP-05 (µg/g)
Al	4730	5020	Al	4370	4750
Ca	141	150	Ca	134	214
Cd	26.7	28.1	Cd	24.1	26.2
Co	[1.9]	[2.3]	Co	[1.7]	[1.6]
Cr	52	55.6	Cr	59.3	65.3
Cu	11.7	11.5	Cu	9.77	10
Fe	[2.2]	[2.7]	Fe	[2.1]	[2.1]
K	996	1040	K	[880.0]	[820.0]
La	<1.3	<1.3	La	<1.2	<1.4
Mn	1.24	[0.78]	Mn	1.21	1.35
Mo	22.4	24.2	Mo	20.5	22.2
Na	96200	104000	Na	89300	98600
Nd	<4.4	[4.7]	Nd	<4.3	<4.8
Ni	180	195	Ni	165	181
P	768	834	P	710	781
Pb	52.5	57.5	Pb	51	53.5
Sr	76.4	82.7	Sr	114	126
W	57.4	60.5	W	50.8	55.6
Zr	[0.61]	[0.9]	Zr	[0.75]	[0.47]

Note: ># = 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%.

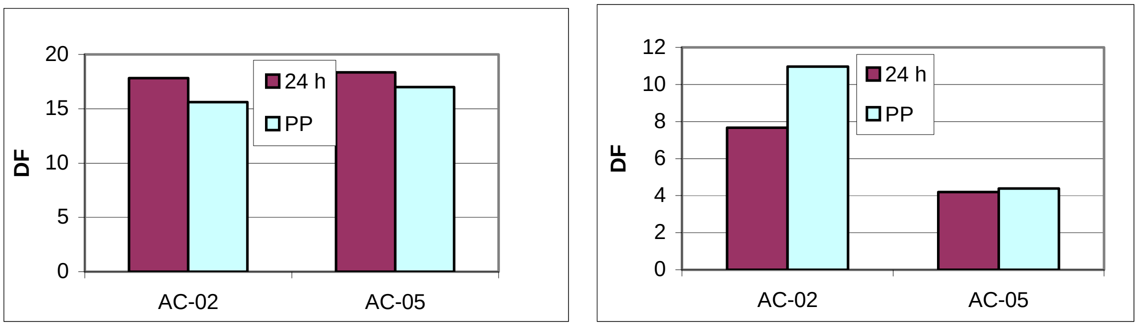

Figure 3.4. Sr-90 and TRU DFs for the Post-Filtration Precipitation Samples

3.5 Estimated Sr-90 and TRU Levels in ILAW Glass

The data from these experiments 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 (sum of alpha). The total sodium concentration determined by ICP-AES must be converted to waste sodium concentration for the ILAW calculation. Using the ICP-AES data for total [Na] and subtracting the amount of added Na from the caustic addition (0.3M added NaOH), the waste sodium was determined to be 95% of the total sodium. The calculated glass loadings are listed in Table 3.3 for the current baseline design waste glass concentration of 15 wt% waste Na_2O. The 15 wt% waste Na_2O is conservative because the contract limit is >10 wt% for Envelope C waste (DOE 2000). The results show that all treated samples were below the contract limits, 20 Ci/m^3 for Sr-90 and 100 nCi/g for TRU, for ILAW glass, except for Sr-90 for sample AC-07 at 4h of reaction, 0.01M reagent addition. The target level of 50% below the limit was not met for Sr-90 at 4h of reaction, low reagent addition, or at the lowest temperature tested, 15°C. However, 0.03M added $Sr(NO_3)_2$ and ambient temperature are adequate to meet the target levels of Sr-90 in ILAW if the reaction time is 24h.

Table 3.3. Sr-90 and TRU ILAW Glass Loadings for 15 wt% Waste Na_2O

Sample	**Sr-90 (Ci/m^3)**	**TRU (nCi/g)**
Contract Limit	**20**	**100**
Initial Waste	87	64
AC-02-4	12	11
AC-02-24	5	8
AC-02-168	3	5
AC-03-24	8	23
AC-04-24	2	9
AC-05-4	9	7
AC-05-24	5	16
AC-06-48	7	13
AC-07-4	35	25
AC-07-24	18	34
AC-07-24	18	31
AC-07-48	3	6
AC-08-48	4	9
AC-09-4	17	16
AC-09-24	13	12
PP-02	5	5
PP-05	5	14

TRU loadings were all below 50% of the contract limit regardless of treatment conditions. AN-102 waste actually is less than the contract limit initially. The baseline treatment conditions yield a TRU loading 10 times less than the contract requirement. Addition of 0.01M $NaMnO_4$ was adequate to give TRU levels approximately four times below the contract limit. The TRU removal process is very robust with regard to process variables of time, temperature, levels of reagent addition, and abnormal processing schemes.

4.0 Conclusions and Recommendations

Experiments were conducted with actual samples of diluted AN-102 waste with various Sr/TRU removal process conditions considered abnormal to the baseline process. These experiments provided a better understanding of the most critical processing parameters as they relate to Sr-90 and TRU removal. Conclusions from this work and recommendations to consider for plant operation are presented in this section.

Experiments were conducted to assess the impact of abnormal process conditions on Sr/TRU removal. A primary focus was to assess the impact of temperature, time, and reagent concentrations, since these conditions have been shown to have significant impact in earlier studies. The current studies were conducted with AN-102 waste samples that were adjusted to approximately 5.5M [Na] and had the addition of 0.3M NaOH. Reaction temperature was varied over a range that might be encountered in the waste treatment plant, 15°C to 50°C. Reaction times were 4h, the solids digest time defined in the baseline process, and 24h, the reasonable time the first waste would contact the filter with the precipitation temperature at 50°C. Reagent concentrations were varied from the baseline conditions of 0.075M Sr and 0.05M $NaMnO_4$, to new proposed concentrations of 0.03M Sr and sodium permanganate, to minimal reagent addition levels of 0.01M Sr and MnO_4^-. Earlier experiments had shown that 0.02M added reagent was adequate if the reaction time was 24h. Variation of the processing scheme was also evaluated. This included no mixing for 24h after reagent addition; reagent addition split into two separate events with 24h delay between additions; and reversing the order of reagent addition, i.e., permanganate first, then a 24h delay until $Sr(NO_3)_2$ is added.

The important factors for determining Sr-90 decontamination are the isotopic dilution ratio and the [Sr]. The baseline level of $Sr(NO_3)_2$ addition (0.075M) results in the highest isotopic dilution and greatest Sr-90 decontamination. The lowest level of $Sr(NO_3)_2$ addition, 0.01M, did not provide adequate isotopic dilution to meet target levels of Sr-90 in ILAW. An additional 0.02M Sr increased the DF to an acceptable level. The nonradioactive Sr addition should be 0.02M or higher to ensure low levels of Sr-90 in the ILAW.

The [Sr] is greatly affected by temperature and reaction time. $SrCO_3$ has retrograde solubility, higher solubility at lower temperature. Lower reaction temperatures also appear to slow the rate for equilibrium to be reached, which could lead to post-filtration precipitation at low temperatures and short reaction times (~24h). Attaining equilibrium [Sr] may take as long as a week. Strontium concentration, and therefore decontamination level, is very time dependent. In all experiments, the Sr concentrations decreased significantly between 4h and 24h, including the experiments at 50°C. In some cases, the concentration was halved over this time, i.e., a doubling of the Sr-90 DF. Although 50°C gave the highest Sr-90 decontamination levels, filtration will actually be conducted at 25°C and given enough time, all reaction temperatures will eventually give the same [Sr] and corresponding Sr-90 DF, based on the isotopic dilution ratio.

None of the various treatment schemes showed a significant impact to the Sr-90 DF. It is important that the Sr/TRU precipitate is well mixed before filtration, but the rapid, initial mixing during reagent addition, appears to be less important for the overall removal of Sr-90. However, it is preferred that the waste mixture be well mixed during reagent addition, because good mixing of the solids provides similar removal efficiency but is much slower and may require more than the 24h time between reagent addition and filtration. The sequence of reagent addition, order in which Sr and permanganate are added, appears to have little impact on decontamination. Sr-90 decontamination is independent of the process scheme, given adequate time for good mixing/distribution of the precipitate and $SrCO_3$ precipitation to reach near-equilibrium levels.

The TRU (sum of alpha) DFs were quite good for most abnormal process conditions evaluated. Reducing the permanganate concentration to 0.01M had a major impact on TRU DF. More reagent is required to obtain significant TRU removal, DF >3. Addition of more reagent gave good decontamination. Temperature appears to have little impact over the range tested, 15°C to 50°C. Time has very little impact on the TRU removal when comparing the 4h and 24h data. However, increased levels of Mn were noted for two of the samples after only 4h of reaction, whereas Mn was removed by 24h of reaction.

Some evidence of post-filtration precipitation was observed with abnormal process conditions, and not the baseline conditions. Low precipitation temperature had the most significant impact on post-filtration precipitation, where significant quantities of white solids were noted. This is most likely $SrCO_3$ precipitation because of the high [Sr] levels found in the 15°C test samples. Dark precipitation indicative of Mn precipitation was only noted for one sample treated with 0.01M reagents at 25°C and 4h of reaction. The precipitation was as a dark "bath tub" ring at the air-liquid interface and likely represented <10 ppm of solids. No ring was observed in a filtrate sample from this test at 24h of reaction.

These experimental results provide additional support to the recommendation that the treated waste should not be filtered after only 4h of reaction, because of the impact on Sr-90 DF and the possible post-filtration precipitation. It is recommended that at least 24h of reaction time be allowed before filtration begins, which is in line with the baseline treatment process, given that the time to cool the waste mixture from 50°C to 25°C would likely be 18h. The precipitation temperature should be higher than 15°C; it is recommended that the minimum reaction temperature be set to the filtration temperature. Given enough time, the treated waste would reach the same equilibrium levels corresponding to the filtration temperature; however, it may take up to a week to reach a near-equilibrium state.

The experimental data can be used to predict the loading of Sr-90 and TRU (sum of alpha) expected in the ILAW. The baseline treatment conditions provided levels of both Sr-90 and TRU that are well below the ILAW requirements of 20 Ci/m^3 of Sr-90 and 100 nCi/g of TRU at 15 wt% waste Na_2O. Most "abnormal" conditions tests met the requirements. The removal of Sr-90 for the supernatant is more sensitive to process conditions, reagent level, temperature, and reaction time. The permanganate treatment for TRU removal is quite robust, with variation in process conditions having little impact on meeting the ILAW requirement.

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	Initial Waste		Sr Addition			MnO_4^- Addition			MDF
	Target, mL	Actual, g	[Sr], M	Target, mL	Actual, g	$[MnO_4^-]$, M	Target, mL	Actual, g	
AC-01	20	25.318	--	None	--	--	None	--	1.000
AC-02	60	76.513	0.66	3	2.972	0.66	3	3.03	1.174
AC-03	20	25.506	0.22	1	0.777	0.22	1	0.942	1.163
AC-04	20	25.652	1.65	1	1.049	1.1	1	0.991	1.265
AC-05	40	51.153	0.66	2	2.132	0.66	2	2.208	1.189
AC-06	20	25.076	0.66	1	(a)	0.66	1	2.137	1.218
AC-07	40	50.856	0.66	0.63	0.709	0.66	0.63	0.672	1.078
AC-07 cont.	~30	36.443	0.66	1	1.095	0.66	1	1.041	1.239
AC-08	20	25.505	0.66	1	1.046	0.66	1	1.075	1.213
AC-09	40	51.012	0.66	2	2.199	0.66	2	2.049	1.188

(a) No individual weight on no-mix experiment.

Appendix B

Analytical Data

Battelle
. . . Putting Technology To Work

Client: Rich Hallen
Date: 3/12/03

Subject: Hydroxide Analyses for: AN-102 Treated Waste (5)
ASR: 6701

Direct sample aliquots of five **AN-102 Treated Waste samples** 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 provided in ug/g and ug/mL as well as converted to Molarity to be comparable to the MRQ units specified in the analytical service request (ASR). All sample concentrations were well above the required MRQ value of 0.05M hydroxide. The sample and replicate were in good agreement with RPD's of 14% or less. The hydroxide standard recovery was 96%, the matrix spike recovery on 03-0664 was 92% and the matrix spike recovery on 03-0677 was 87%.. 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 7% for all the samples. The results are accepted based on the QC data meeting the acceptance criteria as specified in the ASR.

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.

Prepared by: Date: 3/12/03
Reviewed by: Date: 3-18-03

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

ASR 6701

WP# W65819

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

Report Summary for ASR # -- 6701

					Concentration, moles / Liter					
RPG #	**Client ID**		**OH conc ug/g**	**OH conc ug/ml**	**First Point**	**RPD**	**Second Point**	**RPD**	**Third Point**	**RPD**
03-0662	**AC-01-24**		5.1E+03	6.5E+03	**0.38**		**1.13**		**0.72**	
03-0662	**AC-01-24**	**Rep**	5.8E+03	7.3E+03	**0.43**	12%	**1.08**	4.1%	**0.72**	1.0%
03-0664	**AC-02-24**		3.9E+03	5.0E+03	**0.29**		**1.15**		**0.75**	
03-0664	**AC-02-24**	**Rep**	4.5E+03	5.7E+03	**0.34**	14%	**1.12**	2%	**0.70**	7%
03-0668	**AC-05-24**		4.7E+03	5.9E+03	**0.35**		**0.99**		**0.67**	
03-0668	**AC-05-24**	**Rep**	4.6E+03	5.8E+03	**0.34**	3%	**1.02**	2%	**0.64**	5%
03-0671	**AC-01-168**		5.4E+03	6.9E+03	**0.40**		**1.13**		**0.72**	
03-0671	**AC-01-168**	**Rep**	5.8E+03	7.3E+03	**0.43**	7%	**1.10**	2%	**0.72**	0%
03-0677	**AC-09-24**		5.5E+03	6.8E+03	**0.40**		**0.93**		**0.64**	
03-0677	**AC-09-24**	**Rep**	6.0E+03	7.5E+03	**0.44**	10%	**0.89**	5%	**0.64**	1%

	MRQ Molarity	**Estimated Molarity MDL**	**MRQ ug/ml**
OH conc (ug/mL) = M (g/L) * 17,000	**0.05**	**0.0105**	**8.5E+02**

Reag. Blk.1		**0**
Standard 1		**96%**
MS 03-0664	**Matrix spike**	**92%**
MS 03-0677	**Matrix spike**	**87%**

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 free hydroxide concentration. The second inflection point generally represents carbonate or a combination of aluminate and carbonate. The third inflection point is usually indicative of bicarbonate or other weak acids or possibly the continued protonation of alumina.

Analyst: 3/12/03

Reviewer: C Soderquist 3-18-03

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR #	6701	File: R:\radchem\hydroxide\asr	6701
Client:	Rich Hallen	Analysis Date:	3/11/03
WP#	W65819	Report Date:	3/12/03

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

Analyst: Bwoler 3/12/03

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
03-0662	AC-01-24		na	0.300	0.3798	1.266	5	11.774	0.547	10.904	0.115	0.38	
03-0662	AC-01-24	Replicate	na	0.300	0.3824	1.275	6	12.020	0.618	10.714	0.130	0.43	12.19%
03-0664	AC-02-24		na	0.300	0.3831	1.277	7	12.063	0.418	11.030	0.088	0.29	
03-0664	AC-02-24	Replicate	na	0.300	0.3853	1.284	8	11.608	0.482	10.662	0.101	0.34	14.22%
03-0668	AC-05-24		na	0.300	0.3780	1.260	10	11.936	0.500	10.839	0.105	0.35	
03-0668	AC-05-24	Replicate	na	0.300	0.3786	1.262	11	11.975	0.485	10.957	0.102	0.34	3.05%
03-0671	AC-01-168		na	0.300	0.3835	1.278	12	11.941	0.577	10.907	0.121	0.40	
03-0671	AC-01-168	Replicate	na	0.300	0.3812	1.271	13	11.668	0.618	10.558	0.130	0.43	6.86%
03-0677	AC-09-24		na	0.300	0.3712	1.237	14	11.543	0.572	10.277	0.120	0.40	
03-0677	AC-09-24	Replicate	na	0.300	0.3749	1.250	15	11.460	0.634	10.031	0.133	0.44	10.28%
	MDL			5.000					0.25		0.05245	0.0105	
											OH % Recovery		
Reag. Blk.1				5.00	5.0029		1	8.169					
Standard 1	0.1018N NaOH			5.000	5.0213	1.004	3	12.405	2.321	10.309	0.4869	95.7%	
MS 03-0664	+ 2mL 0.1N NaOH			0.100	0.1282	1.282	9	12.019	1.047	10.917	0.2197	92.4%	MS
MS 03-0677	+ 2mL 0.1N NaOH			0.100	0.1221	1.221	16	11.873	0.995	10.818	0.2088	87.1%	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 =	6.981
Continuing	pH 7.0 reading =	7.003

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

Pipet #	Vol.	Wt.	Pipet #	Vol.	Wt.
F04171	5.00	5.0016	92501	0.200	0.2988
F04171	5.00	4.9929	92501	0.200	0.2997
F04171	5.00	4.9971	92501	0.200	0.2991
	Ave	4.9972		Ave	0.2992
	Std. Dev.	0.0044		Std. Dev.	0.0005
	% Pipet error	0.09%		% Pipet error	0.15%

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6701
WP# W65819

File: R:\radchem\hydroxide\asr

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: 3/12/03

Titrant	Molarity
HCl	0.2098

RPG #	0	Sample Vol. (mL	2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	illimole RPD	3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
03-0662	0	0.300	2.160	7.525	0.338	1.128		3.184	4.847	0.215	0.72	
03-0662	Replica	0.300	2.166	7.439	0.325	1.083	4.11%	3.200	4.801	0.217	0.72	1.0%
03-0664	0	0.300	2.057	7.528	0.344	1.146		3.134	4.836	0.226	0.75	
03-0664	Replica	0.300	2.089	7.414	0.337	1.124	1.97%	3.095	4.902	0.211	0.70	6.8%
03-0668	0	0.300	1.918	7.556	0.297	0.992		2.879	4.768	0.202	0.67	
03-0668	Replica	0.300	1.938	7.521	0.305	1.016	2.44%	2.852	4.859	0.192	0.64	5.0%
03-0671	0	0.300	2.187	7.509	0.338	1.126		3.215	4.935	0.216	0.72	
03-0671	Replica	0.300	2.194	7.559	0.331	1.102	2.13%	3.221	4.870	0.215	0.72	0.1%
03-0677	0	0.300	1.907	7.506	0.280	0.934		2.818	4.927	0.191	0.64	
03-0677	Replica	0.300	1.910	7.414	0.268	0.892	4.52%	2.827	4.864	0.192	0.64	0.7%
						2-nd Recovered						
Standard 1		5.000	2.401	7.841	0.01678	3.3%	sample	2.463	4.102			

MS 03-0664		0.100	1.648	7.627	0.12609	6.2%		2.046	4.601			
MS 03-0677		0.100	1.551	7.645	0.11665	5.3%	sample	1.945	4.596			

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.

Chem Rec_86 Prep date: 7/15/02

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 $KHC_8H_4O_4$ (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 84j, Potassium Acid Phthalate $KHC_8H_4O_4$ (KAP) --CMS# 52232
Technique used will be via hand-titration to the phenopthalein endpint. Project titration for about 20-25 mL of a 50 mL burrette.
----- $KHC_8H_4O_4$ = 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 r. g. Swoboda 3/12/03

Expiration Date on Stds.
7/15/04

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

Project / WP#: 42365 / W65819
ASR#: 6701
Client: R. Hallen
Total Samples: 18 (liquid)

	First	Last
RPL#:	03-00662	03-00679
Client ID:	AC-01-24	PP-05
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): 03-06-2003 (A0891)
03-07-2003 (A0892)

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. Oliver 3/13/03
Preparer

3/13/03
Review and Concur

Eighteen aqueous samples submitted under Analytical Service Request (ASR) 6701 were analyzed by ICPAES. The samples were prepared by acid extraction per PNL-ALO-128 in the RPL 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 was conducted in two separate batches.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (6 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.

As specified in the ASR, the results are given as μg/g for each detected analyte, and have been adjusted for all laboratory processing factors. Processing factors for each sample were determined from the calculated final diluent volume, measured sample mass, and instrument dilution(s). Minimum Reportable Quantity (MRQ) values were specified in the ASR for the AOIs. To meet this requirement, method detection limits (MDL) for the ICPAES analyses need to be ≤ ⅓ MRQ. The required MRQ levels were met for all AOIs

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For each extraction processing batch, 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. All AOIs were included in the combined spike solution.

Process Blank:

A process blank (reagents only) was prepared with both sample batches. The concentrations of all AOIs for both process blanks were within the acceptance criteria of <EQL (estimated quantitation level) or ≤ 5% of the concentration in the samples.

Blank Spike:

A blank spike (reagents and spike solution) was prepared with both sample batches. Recovery values for both blank spikes were within the acceptance criterion of 80% to 120% for all AOIs.

Duplicate RPD (Relative Percent Difference):

Duplicates were prepared for Samples 03-00662 and 03-00671. RPDs are listed for all analytes that were measured at or above the EQL. The RPDs for both duplicates were within the acceptance criteria of ≤ 20% for all AOIs meeting the above requirement.

Laboratory Control Standard (LCS):

No LCS samples were provided for analysis.

Matrix Spiked Sample:

Matrix spikes were prepared with Samples 03-00662 and 03-00671. Recovery values are listed for all analytes in the spikes that were measured at or above the EQL, and that had a spike concentration ≥ 20% of that in the sample. The recovery values for both matrix spikes were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirements. Recovery values for analytes with a spike concentration < 20% of the concentration in the sample are listed as "nr".

Post-Spiked Samples (Spike A Elements):

A post-spike A was conducted on Samples 03-00662 and 03-00671. Recovery values are listed for all analytes in the spikes that had a concentration ≥20% of that in the sample. Except for aluminum in the second batch of samples analyzed, the recovery values were within the acceptance criterion of 75% to 125% for all AOIs meeting the above requirement. Recovery values for analytes with a spike concentration < 20% of the concentration in the sample are listed as "nr".

Aluminum was recovered in the second batch at a level of 132%. However, as aluminum was present in the samples at levels greater than 1000 µg/g, no matrix spike, and therefore no post spike, was required for this analyte (Laboratory QA Plan, Chapter 5). It should be noted, that all other QC for aluminum (i.e., duplicate, blank spike, and serial dilution) were well within acceptance criteria. As a result, the over recovery for aluminum in the post spike is not considered a QC failure.

Post-Spiked Samples (Spike B Elements):

A post-spike B was conducted on Samples 03-00662 and 03-00671. 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. Recovery values for analytes with a spike concentration < 20% of the concentration in the sample are listed as "nr".

Serial dilution (Percent Difference):

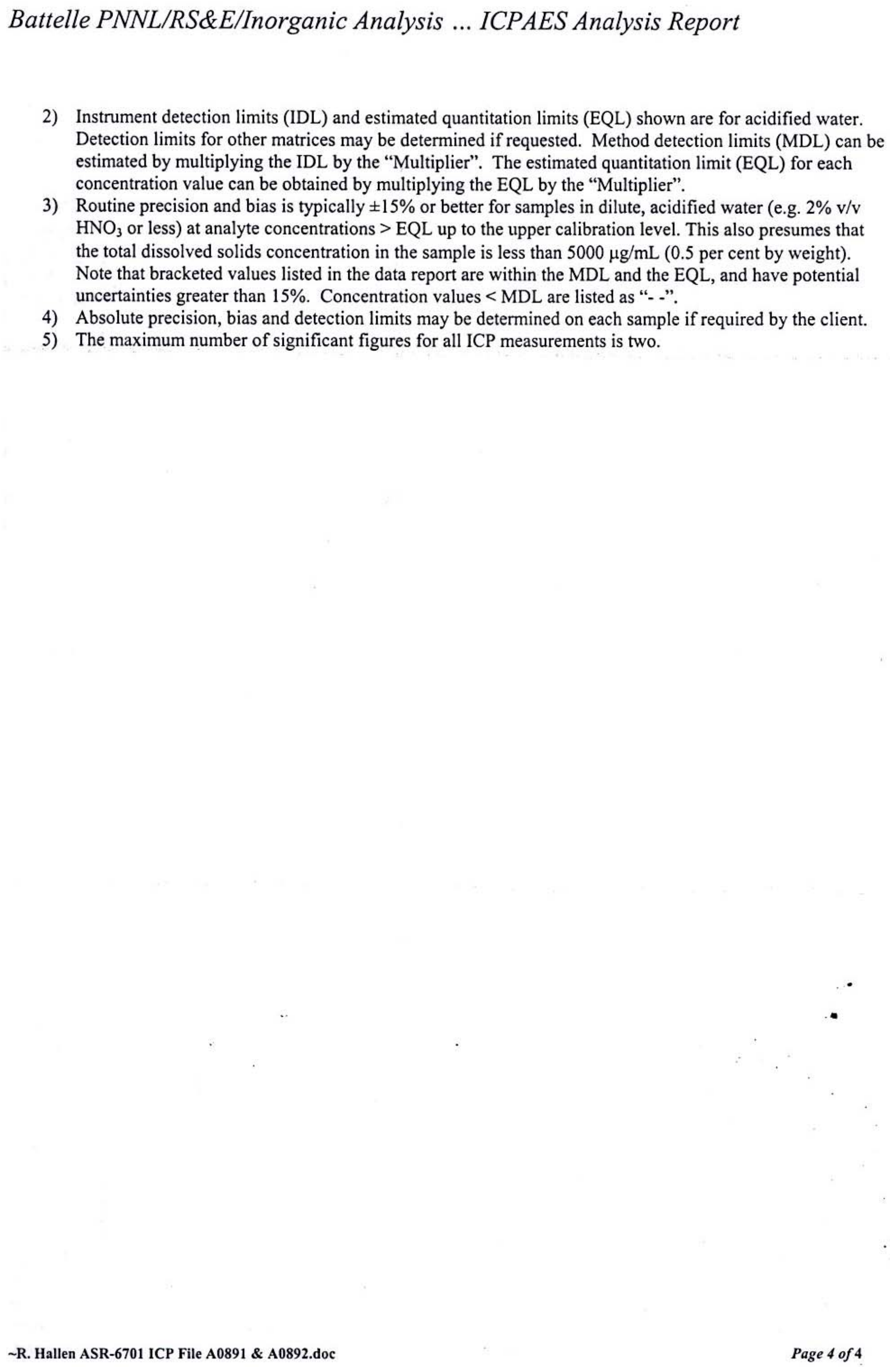
Five-fold serial dilution was conducted on Samples 03-00662 and 03-00671. Percent differences (%Ds) are listed for all analytes that had a concentration at or above the EQL in the diluted sample. The %Ds were within the acceptance criterion of ≤ 10% for all AOIs meeting the above requirement, including aluminum which was over recovered in the post spike.

Other QC:

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

Comments:

1) The "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) and estimated quantitation limits (EQL) 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 IDL by the "Multiplier". The estimated quantitation limit (EQL) for each concentration value can be obtained by multiplying the EQL by the "Multiplier".
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 > EQL 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 the MDL and the EQL, and have potential uncertainties greater than 15%. Concentration values < MDL are listed as "- -".
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.

Battelle PNNL/RSE/Inorganic Analysis.... ICPAES Report

Page 1 of 6

		Run Date=	3/6/2003	3/6/2003	3/6/2003	3/6/2003	3/6/2003	3/6/2003
		Multiplier=	20.7	95.9	95.1	92.4	97.7	93.0
		RPL/LAB #=	03-00662-B	03-00662 @5	03-00662-DUP @5	03-00663 @5	03-00664 @5	03-00665 @5
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID=	process blank	AC-01-24	AC-01-24-Dup	AC-02-4	AC-02-24	AC-03-24
(ug/mL)	(ug/mL)	(Analyte)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
0.0310	0.446	Al	--	4,850	4,740	4,600	4,730	4,590
0.0100	0.100	Fe	[0.29]	[9.1]	[8.8]	[2.4]	[2.2]	[2.1]
0.0130	0.130	La	--	[5.8]	[6.2]	[1.4]	--	[1.6]
0.0870	0.870	Na	--	97,500	96,000	93,800	96,200	93,100
Other Analytes								
0.0050	0.069	Ag	--	--	--	--	--	--
0.0360	0.360	As	--	--	--	--	--	--
0.0100	0.031	B	--	20.1	17.5	18.4	19.1	18.4
0.0011	0.010	Ba	[0.051]	[0.49]	[0.37]	[0.19]	[0.23]	[0.18]
0.0002	0.002	Be	--	--	--	[0.016]	--	--
0.0250	0.250	Bi	11.0	[4.4]	--	[2.6]	[6.5]	[2.5]
0.0450	0.450	Ca	[1.6]	250	191	141	141	163
0.0038	0.038	Cd	[0.23]	27.0	26.1	25.3	26.7	25.5
0.0400	0.400	Ce	--	--	--	--	--	--
0.0050	0.050	Co	--	[1.9]	[1.9]	[1.9]	[1.9]	[1.7]
0.0060	0.060	Cr	--	77.7	75.9	62.3	52.0	64.5
0.0070	0.070	Cu	[0.37]	11.1	10.9	10.3	11.7	11.5
0.0100	0.100	Dy	--	--	--	--	--	--
0.0050	0.050	Eu	--	--	--	--	--	--
1.0000	10.000	K	--	961	989	980	996	[900]
0.0058	0.058	Li	--	[0.82]	--	[0.69]	[0.79]	[0.81]
0.0250	0.335	Mg	--	--	--	--	--	--
0.0006	0.012	Mn	--	1.68	1.63	[1.1]	1.24	[0.72]
0.0057	0.057	Mo	--	23.2	22.1	22.0	22.4	21.3
0.0450	0.400	Nd	--	[11]	[12]	--	--	[4.4]
0.0130	0.130	Ni	--	183	179	176	180	174
0.0240	0.236	P	--	788	765	745	768	749
0.0230	0.264	Pb	[0.66]	70.4	67.8	47.5	52.5	60.7
0.1250	1.250	Pd	--	--	[14]	--	--	--
0.0510	0.510	Rh	--	--	[5.6]	[5.7]	[6.2]	--
0.0200	0.200	Ru	--	[11]	[12]	[12]	[12]	[11]
0.0280	0.280	Sb	--	--	--	--	--	--
0.0360	0.360	Se	--	--	--	--	--	--
0.0300	0.298	Si	--	[17]	[6.4]	[10]	[10]	[15]
0.1300	1.234	Sn	--	--	[14]	[15]	[17]	--
0.0015	0.015	Sr	--	[1.3]	[1.1]	135	76.4	57.7
0.0500	0.500	Te	--	--	--	--	--	--
0.0250	0.278	Th	--	[2.7]	--	--	--	--
0.0025	0.025	Ti	--	--	--	--	--	--
0.0210	0.210	Tl	--	[2.6]	--	--	--	--
0.5400	4.971	U	--	--	--	--	--	--
0.0039	0.040	V	--	--	[0.58]	[0.61]	[0.39]	--
0.0380	0.130	W	--	57.4	55.0	54.8	57.4	54.9
0.0019	0.020	Y	--	[0.58]	[0.64]	[0.23]	--	[0.23]
0.0070	0.070	Zn	[0.59]	[2.4]	[1.8]	--	[0.72]	[1.2]
0.0043	0.043	Zr	--	[3.0]	[3.4]	[1.0]	[0.61]	[0.5]

1) "--" indicates the value is ≤MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values > EQL is estimated to be within ±15%.

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

ASR 6701 Final (2) - ~A0892 R. Hallen ASR-6701 ICP03 hi.XLS

		Run Date=	3/6/2003	3/6/2003	3/6/2003	3/6/2003	3/6/2003
		Multiplier=	101.6	96.9	95.2	95.4	94.0
		RPL/LAB #=	03-00666 @5	03-00667 @5	03-00668 @5	03-00669 @5	03-00670 @5
Instr. Det. Limit (IDL)	Est. Quant. Limit (EQL)	Client ID=	AC-04-24	AC-05-4	AC-05-24	AC-07-4	AC-07-24
(ug/mL)	(ug/mL)	(Analyte)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
0.0000	2.000	Al	4,520	4,330	4,370	4,640	4,650
0.0000	2.000	Fe	[2.3]	[2.4]	[2.1]	[2.3]	[3.0]
0.0000	0.200	La	--	--	--	[1.9]	[2.8]
0.0000	10.000	Na	92,300	88,300	89,300	94,600	94,900
Other Analytes							
0.3000	0.100	Ag	--	--	--	--	--
2.0000	0.500	As	--	--	--	--	--
0.0000	0.400	B	17.8	16.6	16.0	17.8	17.3
0.2000	0.100	Ba	[0.22]	[0.27]	--	--	[0.17]
0.0200	0.020	Be	--	--	--	--	--
0.0000	2.000	Bi	[3.2]	[3.2]	[2.7]	--	--
0.0000	3.000	Ca	99.8	134	134	172	169
0.6000	0.100	Cd	25.3	24.2	24.1	25.3	25.5
0.0000	0.400	Ce	--	--	--	--	--
0.0000	0.200	Co	[1.8]	[1.8]	[1.7]	[1.5]	[1.7]
0.4000	0.200	Cr	65.4	55.2	59.3	62.2	64.0
0.0000	0.200	Cu	10.1	10.1	9.77	11.1	10.5
0.0000	0.400	Dy	--	--	--	--	--
0.0000	0.400	Eu	--	--	--	--	--
0.0000	10.000	K	[940]	[880]	[880]	[840]	[910]
0.0000	0.400	Li	[0.88]	--	[0.63]	--	--
0.0000	3.000	Mg	--	--	--	--	--
0.1000	0.100	Mn	[0.65]	3.32	1.21	8.3	1.3
0.0000	0.200	Mo	21.4	20.3	20.5	21.4	21.6
0.0000	0.400	Nd	--	--	--	--	[6.7]
0.0000	0.200	Ni	171	164	165	174	174
0.0000	1.000	P	728	696	710	756	753
2.0000	0.500	Pb	51.0	50.7	51.0	60.3	61.4
0.0000	2.000	Pd	--	--	--	--	--
0.0000	1.000	Rh	--	--	--	--	--
0.0000	1.000	Ru	[11]	[11]	[11]	[11]	[11]
1.5000	0.500	Sb	--	--	--	--	--
2.0000	0.500	Se	--	--	--	--	--
10.0000	4.000	Si	[14]	[6.2]	[5.0]	[5.0]	[6.1]
0.0000	4.000	Sn	--	[14]	--	--	--
0.0000	0.100	Sr	90.1	164	114	215	130
0.0000	3.000	Te	--	--	--	--	--
0.0000	4.000	Th	--	--	--	--	--
0.0000	0.200	Ti	--	--	--	--	--
0.0000	0.500	Tl	--	--	--	--	--
0.0000	10.000	U	--	--	--	--	--
0.5000	0.200	V	--	--	--	--	--
0.0000	0.500	W	51.2	50.8	50.8	55.6	54.2
0.0000	0.200	Y	--	--	[0.22]	[0.25]	[0.39]
0.0000	0.300	Zn	[1.1]	[0.97]	--	[0.98]	[0.71]
0.0000	0.400	Zr	[0.47]	[0.89]	[0.75]	[1.1]	[1.2]

1) "--" indicates the value is ≤MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values > EQL is estimated to be within ±15%.

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

ASR 6701 Final (2) - ~A0892 R. Hallen ASR-6701 ICP03 hi.XLS

		3/7/2003	3/7/2003	3/7/2003	3/7/2003	3/7/2003	3/7/2003
		20.3	103.8	104.7	104.9	105.0	105.4
		03-00671-B	03-00671 @5	03-00671-DUP @5	03-00672 @5	03-00673 @5	03-00674 @5
Instr. Det. Limit (IDL)	**Est. Quant. Limit (EQL)**	**process blank**	**AC-01-168**	**AC-01-168-Dup**	**AC-02-168**	**AC-06-48**	**AC-07-48**
(ug/mL)	(ug/mL)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
0.0000	0.000	--	5,150	5,270	5,010	4,690	4,580
0.0000	0.000	[0.71]	[9.7]	11.3	[2.7]	[2.6]	[2.8]
0.0000	0.000	--	[6.2]	[6.9]	--	[1.5]	--
0.0000	0.000	--	104,000	108,000	103,000	96,600	93,900
Other Analytes							
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	18.6	19.4	19.0	22.6	20.1
0.0000	0.000	[0.035]	[0.34]	[0.47]	[0.56]	[0.28]	[0.28]
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	15.2	[9.4]	[5.7]	[2.9]	--	[3.0]
0.0000	0.000	[0.92]	227	227	160	181	143
0.0000	0.000	[0.51]	28.6	29.2	27.7	26.1	25.9
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	[1.9]	[2.2]	[1.9]	[1.6]	[2.0]
0.0000	0.000	--	83.7	85.9	58.8	53.4	67.9
10.0000	0.000	--	10.9	12.0	11.2	10.3	10.3
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	[1,000]	1,090	[1,000]	[880]	[980]
10.0000	0.000	--	--	[0.88]	[0.79]	--	[0.76]
0.0000	0.000	--	--	[3.3]	--	--	--
10.0000	0.000	--	1.71	1.88	2.14	2.68	[0.87]
0.0000	0.000	--	23.9	24.9	24.0	22.1	21.9
0.0000	0.000	--	[11]	[14]	--	--	[5.8]
0.0000	0.000	--	194	199	190	178	175
0.0000	0.000	--	838	865	815	767	755
10.0000	0.000	[1.1]	74.7	77.9	63.1	56.7	59.2
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	[6.4]	--	--	[6.6]
0.0000	0.000	--	[12]	[13]	[12]	[10]	[12]
0.0000	0.000	--	--	[3.7]	--	--	--
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	[10]	[13]	[13]	[15]	[12]
0.0000	0.000	--	--	[25]	--	--	[14]
0.0000	0.000	[0.08]	[1.3]	[1.4]	90.1	114	119
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	[3.3]	[2.9]	--	[2.4]
0.0000	0.000	--	--	--	--	--	--
0.0000	0.000	--	--	[0.59]	--	--	[0.5]
0.0000	0.000	--	61.3	64.1	57.0	53.4	53.7
0.0000	0.000	--	[0.63]	[0.72]	--	--	--
10.0000	0.000	[0.81]	[4.1]	[5.6]	[1.4]	[0.84]	[0.93]
0.0000	0.000	--	[3.5]	[3.8]	[0.46]	[0.66]	[0.92]

1) "--" indicates the value is ≤MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values > EQL is estimated to be within ±15%.

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

		Run Date=	**3/7/2003**	**3/7/2003**	**3/7/2003**	**3/7/2003**	**3/7/2003**
		Multiplier=	**104.2**	**103.7**	**102.8**	**102.5**	**107.1**
		RPL/LAB #=	**03-00675 @5**	**03-00676 @5**	**03-00677 @5**	**03-00678 @5**	**03-00679 @5**
Instr. Det. Limit (IDL)	**Est. Quant. Limit (EQL)**	**Client ID=**	**AC-08-48**	**AC-09-4**	**AC-09-24**	**PP-02**	**PP-05**
(ug/mL)	**(ug/mL)**	**(Analyte)**	**(ug/g)**	**(ug/g)**	**(ug/g)**	**(ug/g)**	**(ug/g)**
0.0000	0.000	Al	4,680	4,620	4,620	5,020	4,750
0.0000	0.000	Fe	[2.0]	[2.4]	[2.5]	[2.7]	[2.1]
0.0000	0.000	La	--	[1.4]	[1.8]	--	--
0.0000	0.000	Na	96,300	95,200	95,200	104,000	98,600
Other Analytes							
0.0000	0.000	Ag	--	--	--	--	--
0.0000	0.000	As	--	--	--	--	--
0.0000	0.000	B	19.9	17.7	17.1	24.4	20.5
0.0000	0.000	Ba	[0.17]	[0.18]	[0.34]	[0.2]	[0.58]
0.0000	0.000	Be	--	--	--	--	--
0.0000	0.000	Bi	--	--	--	--	--
0.0000	0.000	Ca	153	156	154	150	214
0.0000	0.000	Cd	26.2	25.8	25.5	28.1	26.2
0.0000	0.000	Ce	--	--	--	--	--
0.0000	0.000	Co	[1.8]	[2.1]	[2.1]	[2.3]	[1.6]
0.0000	0.000	Cr	69.4	65.4	66.6	55.6	65.3
0.0000	0.000	Cu	9.77	10.4	10.3	11.5	10.0
0.0000	0.000	Dy	--	--	--	--	--
0.0000	0.000	Eu	--	--	--	--	--
0.0000	0.000	K	[930]	[970]	[990]	1,040	[820]
0.0000	0.000	Li	--	--	--	[0.79]	--
0.0000	0.000	Mg	--	--	--	--	--
0.0000	0.000	Mn	[0.62]	1.44	[0.79]	[0.78]	1.35
0.0000	0.000	Mo	22.2	22.1	22.2	24.2	22.2
0.0000	0.000	Nd	--	[6.2]	[7.0]	[4.7]	--
0.0000	0.000	Ni	179	177	177	195	181
0.0000	0.000	P	767	758	762	834	781
0.0000	0.000	Pb	53.8	47.5	49.5	57.5	53.5
0.0000	0.000	Pd	--	--	[13]	--	--
0.0000	0.000	Rh	--	[6.9]	[7.6]	[7.9]	--
0.0000	0.000	Ru	[12]	[12]	[12]	[13]	[9.9]
0.0000	0.000	Sb	--	--	[3.4]	[3.4]	--
0.0000	0.000	Se	--	--	--	--	--
0.0000	0.000	Si	[12]	[11]	[8.7]	[19]	[13]
0.0000	0.000	Sn	--	[15]	[17]	[20]	--
0.0000	0.000	Sr	111	293	256	82.7	126
0.0000	0.000	Te	--	--	--	--	--
0.0000	0.000	Th	--	--	--	--	--
0.0000	0.000	Ti	--	--	--	--	--
0.0000	0.000	Tl	--	[3.0]	[4.1]	[4.0]	--
0.0000	0.000	U	--	--	[57]	--	--
0.0000	0.000	V	--	[0.49]	[0.55]	[0.53]	--
0.0000	0.000	W	53.8	51.8	54.4	60.5	55.6
0.0000	0.000	Y	--	[0.3]	[0.37]	[0.23]	--
0.0000	0.000	Zn	[1.1]	--	--	[1.3]	[1.4]
0.0000	0.000	Zr	[0.78]	[1.1]	[1.2]	[0.9]	[0.47]

1) "--" indicates the value is ≤MDL. The method detection limit (MDL) = IDL times the "multiplier" near the top of each column. The estimated sample quantitation limit = EQL (in Column 2) times the "multiplier". Overall error for values > EQL is estimated to be within ±15%.

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

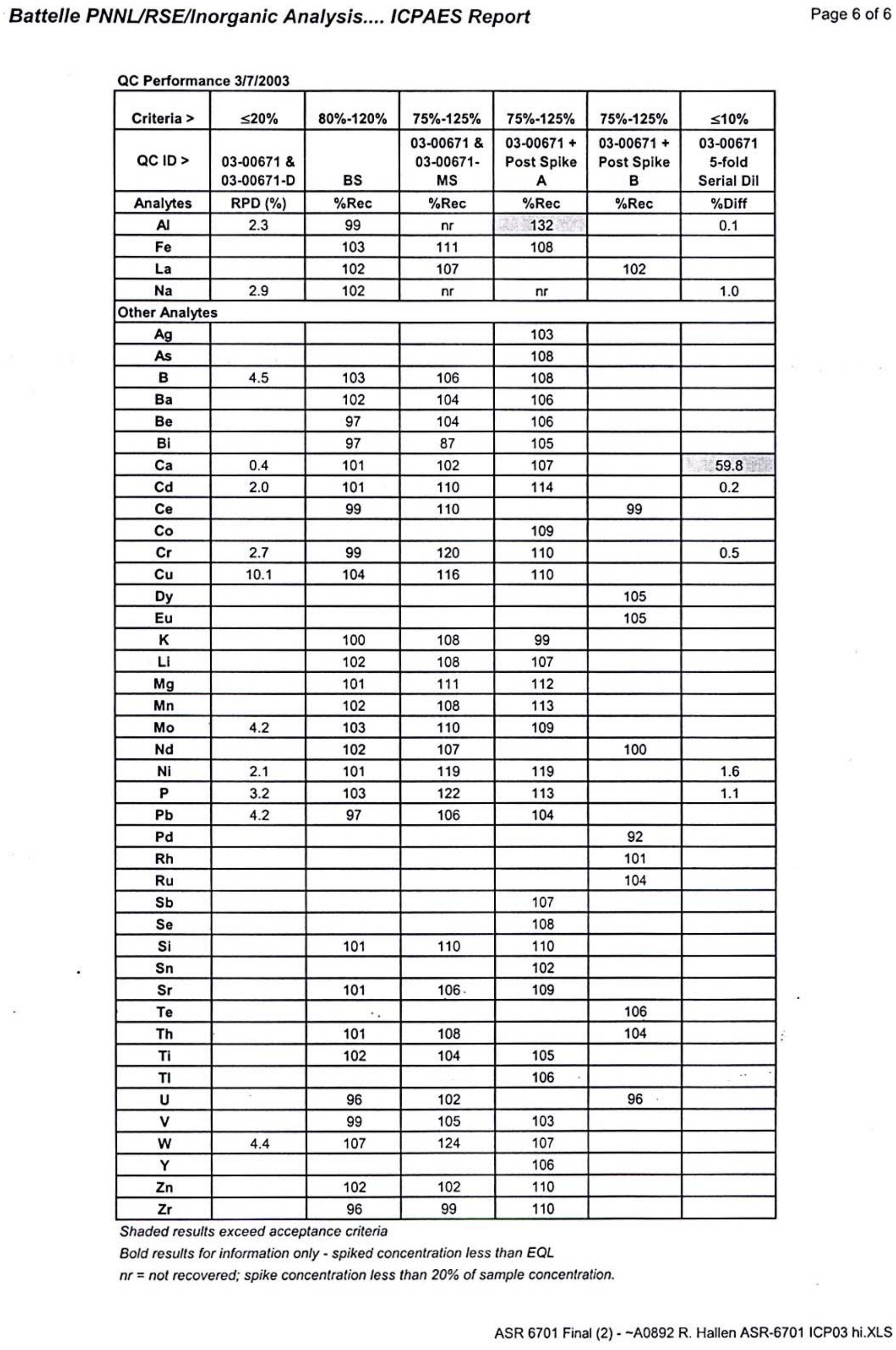
QC Performance 3/6/2003

Criteria >	**≤20%**	**80%-120%**	**75%-125%**	**75%-125%**	**75%-125%**	**≤10%**
QC ID >	**03-00662 & 03-00662-D**	**BS**	**03-00662 & 03-00662-MS**	**03-00662 + Post Spike A**	**03-00662 + Post Spike B**	**03-00662 5-fold Serial Dil**
Analytes	**RPD (%)**	**%Rec**	**%Rec**	**%Rec**	**%Rec**	**%Diff**
Al	2.4	101	102	nr		2.5
Fe		104	105	106		
La		100	98		101	
Na	1.5	104	nr	nr		4.6
Other Analytes						
Ag				99		
As				106		
B	13.8	104	100	104		
Ba		102	97	102		
Be		96	96	102		
Bi		100	102	101		
Ca	26.8	101	73	101		8.0
Cd	3.4	102	98	109		2.3
Ce		97	99		96	
Co				107		
Cr	2.4	99	103	105		2.3
Cu	1.6	104	102	106		
Dy					103	
Eu					103	
K	2.9	102	99	99		
Li		104	96	105		
Mg		103	105	110		
Mn		103	101	110		
Mo	4.7	103	100	106		
Nd		100	97		99	
Ni	2.5	102	105	109		2.9
P	3.0	104	106	104		0.1
Pb	3.8	98	97	101		
Pd					96	
Rh					99	
Ru					104	
Sb				104		
Se				106		
Si		103	103	110		
Sn				99		
Sr		103	97	107		
Te					107	
Th		100	99		103	
Ti		101	97	102		
Tl				105		
U		98	96		97	
V		96	96	98		
W	4.2	104	98	102		
Y				100		
Zn		103	102	108		
Zr		103	101	107		

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.



QC Performance 3/7/2003

Criteria >	≤20%	80%-120%	75%-125%	75%-125%	75%-125%	≤10%
QC ID >	03-00671 & 03-00671-D	BS	03-00671 & 03-00671-MS	03-00671 + Post Spike A	03-00671 + Post Spike B	03-00671 5-fold Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	2.3	99	nr	132		0.1
Fe		103	111	108		
La		102	107		102	
Na	2.9	102	nr	nr		1.0
Other Analytes						
Ag				103		
As				108		
B	4.5	103	106	108		
Ba		102	104	106		
Be		97	104	106		
Bi		97	87	105		
Ca	0.4	101	102	107		59.8
Cd	2.0	101	110	114		0.2
Ce		99	110		99	
Co				109		
Cr	2.7	99	120	110		0.5
Cu	10.1	104	116	110		
Dy					105	
Eu					105	
K		100	108	99		
Li		102	108	107		
Mg		101	111	112		
Mn		102	108	113		
Mo	4.2	103	110	109		
Nd		102	107		100	
Ni	2.1	101	119	119		1.6
P	3.2	103	122	113		1.1
Pb	4.2	97	106	104		
Pd					92	
Rh					101	
Ru					104	
Sb				107		
Se				108		
Si		101	110	110		
Sn				102		
Sr		101	106	109		
Te					106	
Th		101	108		104	
Ti		102	104	105		
Tl				106		
U		96	102		96	
V		99	105	103		
W	4.4	107	124	107		
Y				106		
Zn		102	102	110		
Zr		96	99	110		

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.

ASR 6701 Final (2) - ~A0892 R. Hallen ASR-6701 ICP03 hi.XLS

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Project No. 42365

Internal Distribution
File/LB

page 1 of 2
7/18/03

Date May 5, 2003

To R. T. Hallen

From L. R. Greenwood LRGreenwood

Subject Analyses for AN102 Treated Waste – ASR 6701

Samples of the treated waste from tank AN102 were analyzed for ammonia, ^{90}Sr, and alpha emitters according to ASR 6701. The ammonia analyses were performed on diluted samples. However, for the radiochemical analyses, the samples were acid leached in the hot cells in two batches according to procedure PNL-ALO-128. The samples were analyzed in the laboratory in one batch. Aliquots of these preps were then delivered to the laboratory for analysis, as described below. The radiochemistry data are reported in uCi/g and the ammonia results are in ug/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.

Alpha Emitters by Alpha Energy Analysis

Aliquots of the leachates from the hot cells were prepared for alpha counting by precipitation plating according to procedures RPG-CMC-4001 and PNL-ALO-496. Alpha counting was then performed according to procedure PNL-ALO-422. Three peaks were observed in the samples due to the unresolved isotopes of ^{239}Pu+^{240}Pu, ^{238}Pu+^{241}Am, and ^{243}Cm+^{244}Cm. No significant alpha activities were seen in the hot cell process blanks or the laboratory blanks. Matrix spike and LCS recoveries were 95% to 100%. Duplicate results are generally in good agreement (RPD < 20%) except in a few cases where the counting statistics are high. However, mean difference (MD) calculations show that all of the duplicate results are in good statistical agreement (MD < 1.96) when the counting statistics are taken into account. All of the samples had alpha activities well above the MRQ value of 7.2E-4 uCi/ml.

Strontium-90

Strontium separations were performed according to procedure PNL-ALO-476 and counted by liquid scintillation counting according to procedure PNL-ALO-474. 90Sr was detected in all of the samples well above the MRQ value of 1.5E-1 uCi/ml. Duplicate results were in good agreement and no significant ^{90}Sr activities were seen in the hot cell or lab blanks. Two LCS recoveries were 98% and 113%. Duplicate matrix spike recoveries were 117% and 60%. However, the uncertainties on the matrix spike recoveries are high (10-30%) due to the high sample activities relative to the spike activities.

Because of the low matrix spike recovery for the Batch #2 samples, results for AC-01-168, AC-06-48, AC-07-48, AC-08-48, AC-02-1 AC-09-4, AC-09-24, PP-02, and PP-05 are all flagged with J, indica estimated values. 7/18/03 per KM 5/19/03

Ammonia

Ammonia was measured in two of the samples according to procedure RPG-CMC-226 using samples that were diluted with water and 1M sulphuric acid. Ammonia was seen in both samples, although no MRQ value was requested. Duplicate results show good agreement for sample AC-01-24. No ammonia could be detected in the hot cell blank. The LCS recovery was 112%. However, the matrix spike recovery could not be determined since the spike concentration was much lower than the sample concentration.

Because of matrix spike recovery – too small of spike used, all NH_3 data marked with "J" (estimated). RTHallen 7/18/03

Review: rnKM 5/19/03

C. Soderquist 5-8-03

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 03-0662
3/24/2003

Client: R. Hallen
ASR 6701

Prepared by: C Soderquist 3-24-03
Concur: L.R. Greenwood 3-24-03

Procedure RPG-CMC-226, *Measurement of Ammonia in Aqueous Samples*

Sample	Lab ID	**Measured Ammonia Concentration, µg/g** **Result ± 1s**	**EQL**
AC-01-24	03-0662	**1.05E+2**(J)**± 3%**	5E+0
AC-01-24	03-0662 Dup	**9.78E+1**(J)**± 3%**	5E+0
	RPD	7%	
AC-02-24	03-0664	**1.06E+2**(J)**± 3%**	5E+0
	Hot cell blank	< 4E+0	
	Matrix spike 662	*spike concentration was too low for the sample	
	LCS	112%	

*Because of spike concentration too low, all data marked as "J" - estimated.
7/18/03
per KM 5/19/03

J = estimated value

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 03-0662
05/05/03
Rev. 1

Client: R. Hallen
ASR 6701

Prepared by: L.R. Greenwood Date: 5/5/03

Concur: C. Soderquist Date: 5-8-03

Reference date: March 21, 2003 for AEA
Procedure: RPG-CMC-4001and PNL-ALO-496 & 422 for AEA
Reference date: April 1, 2003 for Sr-90
Procedure: PNL-ALO-476 & 408 for Sr-90

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

ALO ID Client ID	Hot Cell Batch *	Sr-90 Error %	Pu-239+ Pu-240 Error %	Pu-238+ Am-241 Error %	Cm-243+ Cm-244 Error %	Sum of alpha emitters
03-0662B	1	1.64E-4	<3.E-6	<4.E-6	<4.E-6	
Process Blank		± 27%				
03-0662	1	2.75E+1	2.11E-3	5.14E-2	2.68E-3	5.62E-2
AC-01-24		± 5%	± 14%	± 3%	± 12%	± 3%
03-0662 DUP	1	2.56E+1	2.08E-3	5.08E-2	2.04E-3	5.49E-2
AC-01-24		± 4%	± 14%	± 3%	± 14%	± 3%
RPD		7%	1%	1%	27%	2%
MD		0.40	0.03	0.10	0.53	0.20
03-0663	1	3.36E+0	8.58E-4	7.36E-3	5.42E-4	8.76E-3
AC-02-4		± 5%	± 24%	± 8%	± 29%	± 7%
03-0664	1	1.42E+0	6.64E-4	5.67E-3	3.52E-4	6.69E-3
AC-02-24		± 5%	± 24%	± 9%	± 33%	± 8%
03-0665	1	2.18E+0	1.07E-3	1.64E-2	1.03E-3	1.85E-2
AC-03-24		± 5%	± 18%	± 5%	± 19%	± 5%
03-0666	1	4.68E-1	8.93E-4	4.58E-3	1.40E-3	6.87E-3
AC-04-24		± 5%	± 21%	± 9%	± 17%	± 7%
03-0667	1	2.41E+0	5.84E-4	4.54E-3	3.89E-4	5.51E-3
AC-05-4		± 5%	± 33%	± 12%	± 41%	± 11%
03-0668	1	1.37E+0	8.86E-4	1.07E-2	5.70E-4	1.22E-2
AC-05-24		± 5%	± 27%	± 8%	± 33%	± 7%
03-0669	1	1.02E+1	9.59E-4	1.82E-2	7.03E-4	1.99E-2
AC-07-4		± 5%	± 27%	± 6%	± 35%	± 6%
03-0670	1	5.23E+0	8.73E-4	2.47E-2	1.61E-3	2.72E-2
AC-07-24		± 4%	± 30%	± 4%	± 20%	± 4%
03-0670 Lab Dup	1	5.40E+0	7.89E-4	2.30E-2	1.17E-3	2.50E-2
AC-07-24		± 5%	± 24%	± 4%	± 20%	± 4%
RPD		3%	10%	7%	32%	9%
MD		0.18	0.09	0.45	0.40	0.55

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

ALO ID Client ID	Hot Cell Batch *	Sr-90 Error %	Pu-239+ Pu-240 Error %	Pu-238+ Am-241 Error %	Cm-243+ Cm-244 Error %	Sum of alpha emitters
03-0671 B Process Blank	2	5.19E-4 ± 10%	3.60E-6 ± 33%	2.32E-5 ± 13%	9.20E-6 ± 21%	3.60E-5 ± 10%
03-0671 AC-01-168	2	2.87E+1 (J) ± 5%	2.15E-3 ± 15%	5.45E-2 ± 3%	2.59E-3 ± 13%	5.92E-2 ± 3%
03-0671 DUP AC-01-168	2	2.73E+1 (J) ± 4%	2.20E-3 ± 18%	4.64E-2 ± 3%	2.10E-3 ± 17%	5.07E-2 ± 3%
RPD MD		5% 0.28	2% 0.03	16% 1.34	21% 0.35	16% 1.34
03-0672 AC-02-168	2	8.98E-1 (J) ± 4%	4.19E-4 ± 32%	3.90E-3 ± 10%	4.19E-4 ± 32%	4.74E-3 ± 9%
03-0673 AC-06-48	2	2.21E+0 (J) ± 4%	8.06E-4 ± 22%	9.22E-3 ± 7%	6.44E-4 ± 25%	1.07E-2 ± 6%
03-0674 AC-07-48	2	8.51E-1 (J) ± 4%	4.03E-4 ± 32%	3.78E-3 ± 10%	2.42E-4 ± 41%	4.43E-3 ± 9%
03-0675 AC-08-48	2	1.26E+0 (J) ± 4%	6.28E-4 ± 33%	6.21E-3 ± 11%	5.58E-4 ± 35%	7.40E-3 ± 10%
03-0676 AC-09-4	2	4.93E+0 (J) ± 4%	1.24E-3 ± 24%	1.07E-2 ± 8%	6.20E-4 ± 33%	1.26E-2 ± 7%
03-0677 AC-09-24	2	3.88E+0 (J) ± 4%	5.51E-4 ± 40%	8.61E-3 ± 9%	4.82E-4 ± 38%	9.64E-3 ± 9%
03-0678 PP-02	2	1.62E+0 (J) ± 4%	6.45E-4 ± 29%	4.03E-3 ± 10%	<5.E-4	4.68E-3 ± 10%
03-0679 PP-05	2	1.52E+0 (J) ± 4%	7.81E-4 ± 32%	9.46E-3 ± 7%	6.99E-4 ± 28%	1.09E-2 ± 7%
03-0679 Lab Dup PP-05	2	1.44E+0 (J) ± 4%	5.41E-4 ± 32%	1.13E-2 ± 6%	5.83E-4 ± 29%	1.24E-2 ± 6%
RPD MD		5% 0.34	36% 0.28	18% 0.69	18% 0.16	13% 0.51
Matrix Spike 670	1	117%	100%			
Matrix Spike 679	2	60%	100%			
Reagent Spike #1		113%	100%			
Reagent Spike #2		98%	95%			
Lab Blank #1		<5.E-5	<5.E-4	<4.E-4	<3.E-4	
Lab Blank #2		<5.E-5	<3.E-4	<5.E-4	<2.E-4	

Low matrix recovery (<75%) resulting in data flag (J) for Batch 2 Sr-90 values

7/18/0

per KM 5/19/03

*The samples were prepped in two batches in the hot cells and analyzed in the lab in one batch.

J = estimated Value (due to low matrix recovery)

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