PNWD-3206
WTP-RPT-043

Filtration, Washing, and Caustic Leaching of Hanford Tank AZ-101 Sludge

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Prepared for Bechtel National, Inc.
under Contract 24590-101-TSA-W000-0004

LEGAL NOTICE

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Test Specification: 24590-WTP-TSP-01-005
Test Plan: TP-RPP-WTP-099
Test Exceptions: Two
R&T Focus Area: Pretreatment
Test Scoping Statement(s): B-78a

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-01-005 and Test Plan TP-RPP-WTP-099. 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 H. Beeman, Manager
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Date

G. Todd Wright, Manager
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Date

Summary

Flowsheets developed for the River Protection Project-Waste Treatment Plant (RPP-WTP) call for the use of washing and/or caustic leaching to pretreat the Hanford Envelope D[a] sludge before it undergoes high-level waste (HLW) vitrification (DOE-ORP 2000). These pretreatment steps reduce the quantity of HLW generated, by removing components such as aluminum, chromium, sodium, and phosphorus that are soluble in water or high-temperature caustic solutions, or both, and often limit the waste loading in the glass.

The RPP-WTP flowsheets also specify crossflow filtration to separate the wash and leach solutions from the solids between each step. In crossflow filtration, the majority of the filter cake is swept away by the fluid flowing across it. This filtration method is especially beneficial when there are very fine particles and when system simplicity is required. Traditional dead-end filtration has a declining filtration rate caused by the growth of a filter cake on the surface of the filter medium.

This report summarizes testing performed in accordance with Test Specification 24590-WTP-TSP-01-005 and Test Plan TP-RPP-WTP-099, as part of Scoping Statement B-78a. The objective of this work was to gather data on the performance of crossflow filtration when processing solids from Hanford Tank AZ-101. The second objective of this work was to evaluate washing and leaching characteristics of Tank AZ-101 sludge, and determine the filterability of the treated sludge.

Approximately 4313 g of slurry from Hanford Tank AZ-101 were evaluated by the pretreatment processes of crossflow filtration, washing, caustic leaching, and rinsing. The filterability of diluted sludge was measured with a 0.1-µm sintered metal "Industrial Grade" Mott filter using a 24-in.-long, single-element, crossflow filtration system. Before washing and leaching, a 7.6 wt% solids[b] slurry was filtered using a matrix of thirteen 1-hour conditions of varying transmembrane pressure (TMP) (20 to 60 psid) and axial velocity (7 to 15 ft/s), with the permeate being recirculated. The system was backpulsed between each condition, and the slurry was tested for 10 hours at a single condition without backpulsing. The slurry was then concentrated to 17.9 wt% solids, and the 13-point test matrix and extended testing at a single condition without backpulsing were repeated.

The matrix with the low solids concentration (7.6 wt%) produced a permeate flux that ranged from 0.023 to 0.036 gpm/ft^2. This flux was primarily dependent on TMP, with little dependency on time or axial velocity. The matrix with the high solids concentration (17.9 wt%) produced a permeate flux that ranged from 0.011 to 0.025 gpm/ft^2. This flux was primarily dependent on axial velocity, with little dependency on time or TMP. These results indicate that the permeate flux in the high solids matrix was limited by back-transport of solids away from the membrane, but not in the low solids matrix. In both cases, the targeted[c] permeate flux of 0.014 gpm/ft^2 was attainable.

Once testing of these two matrices was completed, the material was washed twice in a cells unit filter (CUF) by batch additions of 1.0 liter of 0.01 M NaOH and by removing the permeate by filtration. The purpose of these washing steps was to displace the supernatant and remove any water-soluble

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[a] Envelope designations are explained in Specifications 7 and 8 of Contract No. DE-AC27-01RV14136 (DOE-ORP 2000).

[b] Solids concentrations are generally reported on an undissolved solids basis by mathematically subtracting out the dissolved solids from the total solids.

[c] Specified by the Contractor.

components. For example, the washing steps reduced the supernatant sodium molarity from 4.5 to 0.9. After the sludge was washed with dilute caustic, it was combined with a concentrated caustic leach solution to produce a slurry containing ~3 M NaOH. The slurry was contacted with the leaching solution for 8 hours at 85°C, and then filtered at 25°C. This leaching was followed by three batch rinses at 25°C using 0.01 M NaOH to displace remaining soluble analytes from the interstitial liquids.

Samples of permeate from each slurry washing were analyzed for chemical and radiochemical constituents. The percent removal for each step, provided in Table S.1, is based on the measured mass removed during the appropriate dewatering stages. Because of the sodium added during pretreatment of the slurry, the sodium removals are based on the measured component mass remaining in the slurry. The recovery column indicates how much of each component was accounted for by comparing the mass removed in the wash, leach, rinse, sampling, and residue with the mass in the initial slurry. In all cases the recovery is greater than the sum of the “total removed in the water wash and caustic leach” and the “fraction is solids residue,” because the recovery takes sampling into account.

The primary components in the initial tank sludge in order of decreasing concentration were sodium, aluminum, iron, and zirconium. Following washing and caustic leaching, these four components remained in the highest concentrations, but iron became the primary constituent, more than twice the concentration of sodium or aluminum.

The rheological properties of Tank AZ-101 slurries were determined with a Haake viscometer. All samples exhibited yield pseudoplastic and thixotropic behavior. Such rheological behavior has been observed in other tank waste slurries (Brooks et al. 2000) and was expected. Rheograms of the concentrated 17.9 wt% slurry material and the sludge washed and caustic leached material (10.9 wt%) are provided in this report.

Particle size distribution (PSD) analysis of the as-received slurry (prior to running the CUF); the CUF concentrated 17.9 wt% slurry; and the final sludge washed and caustic leached slurry were measured with a Microtrac X-100 particle analyzer and an ultrafine particle analyzer. For each sample, different flow rates and ultrasonic energy inputs were used to determine the shear sensitivity of the slurry. The volume mean particle size under low flow conditions was 5.4 µm. After running in the CUF for approximately 38 hours, the volume mean particle size decreased to 1.6 µm. This decrease in mean PSD

Table S.1. Removal of AZ-101 Sludge Key Components in 0.01 M NaOH Water Wash and 3 M NaOH Caustic Leach

Component	**Removed** in **Water Wash** (%)	**Total Removed in Water Wash and Caustic Leach (%)**	**Fraction in Solids Residue (%)**	**Recovery (%)**
Al	9	70	25	99
Cr	56	93	36	132
Fe	0	0	92	104
Na	85	91	9	110
P	42	60	60	128
Zr	0	0	108	121
^{90}Sr	0	0	92	104
^{137}Cs	100	100	7	137

is attributed to the extreme shear to which the particles in the CUF are exposed. The mean particle size increased to 2.8 μm after the sludge washing and caustic leaching treatment. It is surmised that some of the smaller particles dissolved, resulting in an increased mean.

Finally, a sample of the washed and caustic leached slurry was screened for reactivity using differential scanning calorimetry. The tested slurry exhibited only endothermic behavior; therefore, the sample did not meet the Hanford threshold criterion (-480 J/g dry waste) used to identify reactive wastes.

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. This work was conducted to the quality requirements of NQA-1-1989 and NQA-2a-1990, Part 2.7, as instituted through PNWD's *Waste Treatment Plant Support Project Quality Assurance Requirements and Description* (WTPSP) manual.

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 the reported work satisfied the Test Plan objectives.

References

Brooks, KP, PR Bredt, SK Cooley, GR Golcar, LK Jagoda, KG Rappe, and MW Urie. 2000. *Characterization, Washing, Leaching, and Filtration of AZ-102 Sludge.* BNFL-RPT-038 Rev. 0, PNWD-3045, Battelle, Pacific Northwest Division, Richland, WA.

U.S. Department of Energy, Office of River Protection (DOE-ORP). 2000. *Contract Between DOE Office of River Protection and Bechtel National, Inc. for the Design and Construction of the Hanford Tank Waste Treatment and Immobilization Plant.* DE-AC27-01RV14136, Richland, WA.

Contents

Figures

Tables

Acronyms

AEA	alpha energy analysis
CUF	cells unit filter
CWF	clean water flux
DF	decontamination factor
DI	deionized water
DOE-ORP	U.S. Department of Energy-Office of River Protection
DSC	differential scanning calorimetry
DTA	differential thermal analysis
DTG	differential thermogravimetric analysis
GEA	gamma energy analysis
HLRF	High Level Radiochemistry Facility
HLW	high-level waste
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectrometry
ID	inside diameter
MRQ	minimum reportable quantity
NIST	National Institute of Standards and Technology
PID	proportional-integral-derivative controller
PNWD	Battelle–Pacific Northwest Division
PSD	particle size distribution
RPL	Radiochemical Processing Laboratory
RPP-WTP	River Protection Project-Waste Treatment Plant
RSST	reactive system screening tool
SRTC	Savannah River Technology Center
TC	total carbon
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TMP	transmembrane pressure
TOC	total organic carbon
TRU	transuranic
UPA	ultrafine particle analyzer (Microtac)

Definitions

Dissolved solids	soluble solids. The solids remaining after complete drying of a liquid at 105°C. Typically reported as wt%. During drying, most mass loss is due to water but other volatile components (e.g. organics) may also be lost.
Undissolved solids	solids excluding all interstitial liquid. This can be thought of as the solids left if all the supernatant and associated dissolved solids could be drained from the bulk slurry. The undissolved solids will generally include some materials that can be washed or dissolved during pretreatment.
Total solids	solids remaining after drying to a stable mass at 105°C and includes dissolved and undissolved solids.
Inhibited water	0.01M $NaOH_{aq.}$

1.0 Introduction

Flowsheets developed for the River Protection Project-Waste Treatment Plant (RPP-WTP) call for the use of washing and/or caustic leaching to pretreat the Hanford Envelope D[a] sludge before high-level waste (HLW) vitrification (DOE-ORP 2000). These pretreatment steps reduce the quantity of HLW generated, by removing components such as aluminum, chromium, sodium and phosphorus that are soluble in water or high-temperature caustic solutions, or both, and often limit the waste loading in the glass.

The RPP-WTP flowsheets also specify crossflow filtration for the initial dewatering and to separate the wash and leach solutions from the solids. In crossflow filtration, the majority of the filter cake is swept away by the fluid flowing across it. This filtration method is especially beneficial when there are very fine particles and when system simplicity is required. Traditional dead-end filtration has a declining filtration rate caused by the growth of a filter cake on the surface of the filter medium.

The first objective of the work discussed here was to test crossflow filtration using actual Envelope D waste (from Hanford Tank AZ-101) in a modified cells unit filter (CUF) system. Similar to the studies conducted with Envelope D wastes from Tanks AZ-102, and C-104 (Brooks et al. 2000a,b), the filtration of AZ-101 sludge was evaluated at both low and high solids concentrations as a function of transmembrane pressure (TMP), axial velocity, and time using a single-element, 0.1-μm Mott filter.

The second objective of this work was to evaluate washing and leaching characteristics of the Tank AZ-101 sludge. The AZ-101 slurry was dewatered and then washed twice with 0.01 M NaOH to determine the concentration of water-soluble components. The slurry was subsequently leached at ~3 M NaOH at elevated temperature (85°C) to determine the concentration of caustic-soluble components. The chemical and radiochemical compositions of the permeate and the final leached solids were measured to determine the efficiency of the filtration, washing, and leaching processes. Both test objectives were met.

This report describes the test apparatus, experimental approach, results of the tests, and chemical and radiochemical analyses of the sludge from Tank AZ-101 and permeates generated during the washing and caustic-leaching steps.[b] The testing was performed in accordance with Test Specification 24590-WTP-TSP-01-005, and Test Plan TP-RPP-WTP-099, as part of Scoping Statement B-78a. Exceptions to the test plan were 1) two dilute caustic washes of the AZ-101 sludge were conducted instead of three, and 2) three rinses were performed after leaching the AZ-101 sludge instead of two.

Section 2.0 of the report describes the test conditions. Section 3.0 discusses the results of the filtration, sludge washing, and caustic leaching tests. Section 4.0 gives the physical properties (including rheology, particle size distribution, and energetics screening measurements) of AZ-101 slurry samples. Conclusions from the testing and analyses are provided in Section 5.0. The appendices contain additional testing information, as well as details on analytical requirements, raw filtration data, statistical analysis, modeling, analytical results, rheology, and particle size distribution (PSD) measurements.

(a) Envelope designations are explained in Specifications 7 and 8 of Contract No. DE-AC27-01RV14136 (DOE-ORP 2000).

(b) Data recorded during the filtration testing are included in Laboratory Record Book #14048 and Test Instruction TI-RPP-WTP-149.

2.0 Test Conditions

Slurry samples from Tank AZ-101 were tested from November 12 through 16, 2001. The work was performed in the hot cells at the High Level Radiochemistry Facility (HLRF) located in the Radiochemical Processing Laboratory (RPL) in the Hanford 300 Area. Before testing, the material was homogenized, and sub-samples were pulled for analytical work. The material preparation and homogenization testing are described in Urie et al. (2002) [WTP-RPT-048, to be published].

This section outlines the testing and describes the test apparatus, the CUF verification testing, the experimental approach, and the samples and analyses.

2.1 Overview of Testing

The steps used to test the tank samples are outlined below:

1. Perform clean water flux (CWF) tests.
2. Perform flux tests on standard slurry (0.35 M $SrCO_3$).
3. Rinse the CUF, and repeat the CWF tests.
4. Run the AZ-101 slurry (nominally 8 wt% solids) through a test matrix of various TMPs and crossflow velocities to determine the optimal dewatering condition.
5. Run the slurry without backpulsing for ~10 hours.
6. Dewater the slurry to a pre-wash target of 20 wt% undissolved solids or to a concentration reasonably achievable based on the CUF equipment configuration.
7. Run the concentrated feed in a second matrix of TMPs and crossflow velocity conditions to determine the optimal dewatering conditions at higher solids loading.
8. Run the concentrated feed without backpulsing for ~10 hours.
9. Wash the slurry in two batches with 0.01 M caustic at 25 ± 5°C, dewatering after each wash to the minimum slurry volume achievable.
10. Leach slurry in CUF slurry reservoir tank at 85 ± 5°C with 3 M NaOH for 8 hours. Cool to 25°C and then use the CUF to dewater to the minimum slurry volume achievable.
11. Batch rinse slurry with 0.01 M NaOH, and then use the CUF to dewater to the minimum slurry volume achievable after each rinse.
12. Drain the slurry from the CUF. Clean CUF with inhibited (0.01 M NaOH) water to return the CWF to pre-operation (clean) levels. Perform $SrCO_3$ flux tests. If necessary, clean CUF with 2 M nitric acid and rinse to neutral pH.

The TMPs and crossflow velocity conditions are shown in Table 2.1. Except during the elevated-temperature leaching step, the slurry temperature was maintained at 25°C ± 5°C. During each test, permeate flux, axial velocity, filter inlet and outlet pressure, permeate pressure, and slurry temperature were monitored every 10 minutes.

Table 2.1. Test Matrix for Crossflow Filtration Tests of AZ-101 Sludge

Test No.	Event	TMP (psid)	Velocity (ft/s)
1.0	Clean water flux with 0.01 M NaOH	10	11
		20	11
		30	11
1.1	0.35 M $SrCO_3$ in 0.01 M NaOH	10	11
		20	11
		30	11
1.2	Clean $SrCO_3$ from CUF; measure clean water flux again	10	11
		20	11
		30	11
1.3	Test matrix at low solids loading	40	11
1.4		40	11
1.5		40	11
1.6		30	9
1.7		30	13
1.8		50	13
1.9		50	9
1.10		40	11
1.11		40	7
1.12		40	15
1.13		20	11
1.14		60	11
1.15		40	11
1.16a	Extended filtration at low solids loading (do not backpulse each hour for this test)	40	11
1.16b	Dewatering	40	11
1.17	Test matrix at high solids loading	40	11
1.18		40	11
1.19		40	11
1.20		30	9
1.21		30	13
1.22		50	13
1.23		50	9
1.24		40	11

Table 2.1. (contd)

Test No.	Event	TMP (psid)	Velocity (ft/s)
1.25		40	7
1.26		40	15
1.27		20	11
1.28		60	11
1.29		40	11
1.29a	Extended filtration at high solids loading (do not backpulse each hour for this test)	40	11
1.29b	Dewater to ~1000 mL	40	11
1.29.1	Wash 1 (0.01 M NaOH) and dewater @ 25°C	40	11
1.29.3	Wash 2 (0.01 M NaOH) and dewater @ 25°C	40	11
1.29.4	3M Caustic leach at 85°C (dewater at 25°C)	40	11
1.29.5	Rinse 1 – 0.01 M NaOH	40	11
1.29.5A	Rinse 2 – 0.01 M NaOH	40	11
1.29.6	Rinse 3 – 0.01 M NaOH	40	11
1.30	Dewater to minimum volume and recover solids	40	11
	Clean CUF, check clean water flux and $SrCO_3$ flux; acid clean if necessary	10	11
		20	11
		30	11

2.2 Testing Apparatus

Figure 2.1 is a process flow diagram of the CUF. The slurry feed is introduced into the CUF through the slurry reservoir. An Oberdorfer progressive cavity pump (powered by an air motor) pumps the slurry from the slurry reservoir through the magnetic flow meter and the filter element. The axial velocity and TMP are controlled by the pump speed (which is controlled by the pressure of the air supplied to the air motor) and the throttle valve position. Permeate that passes through the filter can be sent to the backpulse chamber; reconstituted with the slurry in the slurry reservoir; or removed. The permeate flow rate is measured by means of a graduated glass-flow monitor that is fill-and-drain operated. Higher permeate flow rates can be monitored with an in-line rotometer. Slurry samples are taken directly from the slurry reservoir by means of a 10-mL pipette.(a) Permeate samples are taken at the three-way valve upstream from the slurry reservoir. This is also the point at which permeate is removed for the dewatering step. Filter backpulsing is conducted by partially filling the backpulse chamber with permeate, pressurizing the backpulse chamber with air, and forcing the permeate in the chamber back through the filter.

(a) Cold testing with a 5.0 wt% kaolin clay slurry indicated sampling with a 10-mL pipette provided slurry samples with a mean and average of 5.1 wt% and a standard deviation of 0.05 wt%. This method of sampling provided more accurate and repeatable results than sampling by means of a slurry sample trap, which had an average of 5.2 wt%, a mean of 5.3 wt%, and a standard deviation of 0.27 wt%. A sample trap is a two-valve arrangement on the pressurized loop. To obtain a sample, one valve is open to charge the sample trap and shut to isolate the sample from the pressurized line. The second valve is subsequently opened to drain the trap (shown in Brooks et al. 2000b).

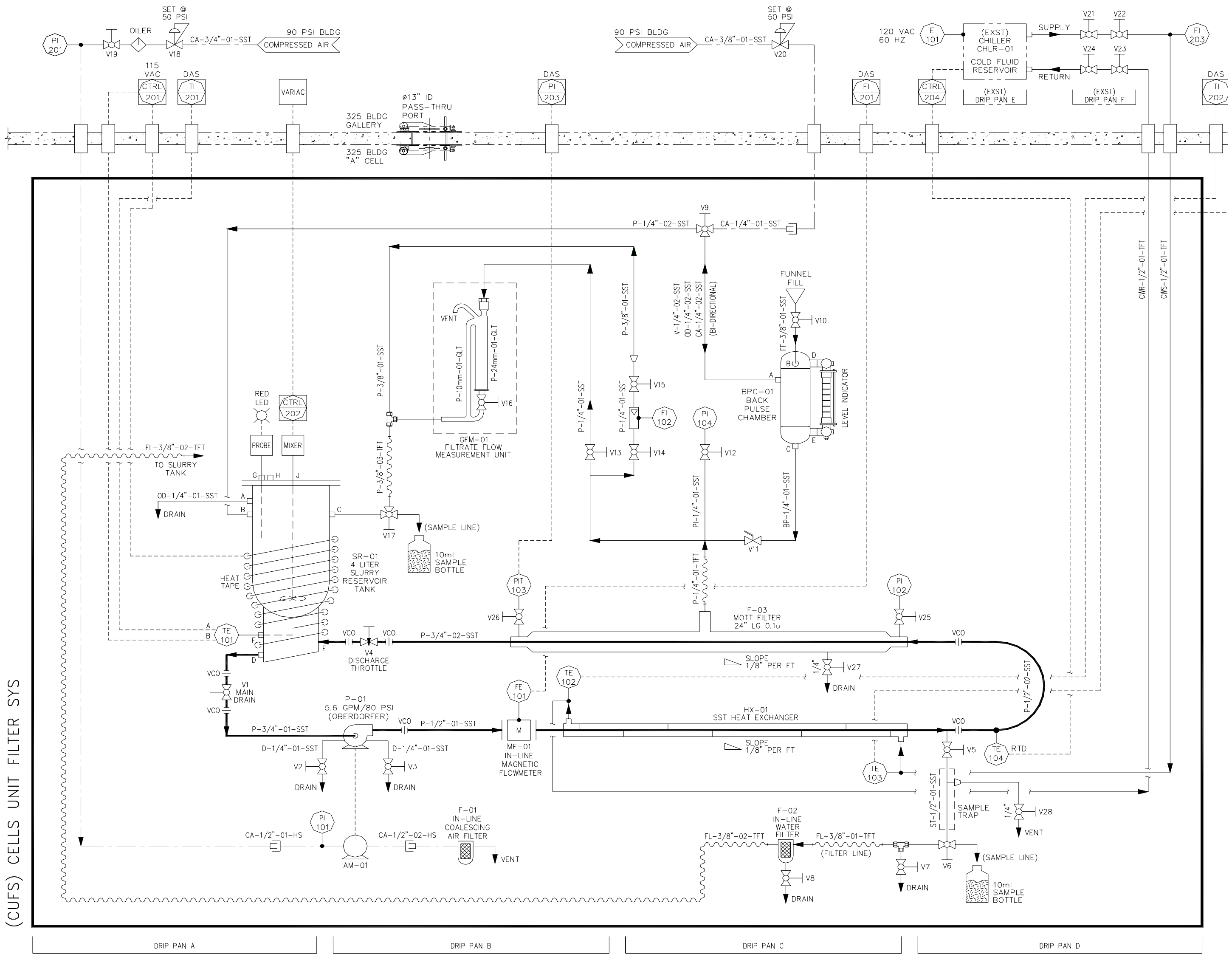


Figure 2.1. Flow Diagram of the Crossflow Filtration Process

The CUF was a new system (MOD3) fabricated for this testing, with minor changes from an earlier version (Brooks et al. 2000b) that had been removed from the hot cells.

- The maximum operating volume of the system was increased from 2.5 to 4.3 liters, while the same minimum operating volume of 1.0 liter was maintained. The increased capacity was needed to better accommodate the sludge washing, caustic leaching, and dewatering steps.

- A mixer was added to the slurry reservoir tank.

- A contact probe was added to the slurry reservoir to accurately determine the liquid level, and thereby the volume of sample in the CUF.

- To reduce areas where solids could hold up in the CUF, welded connections and VCO fittings were used on the slurry side instead of Swagelok fittings. In addition, the pressure relief valve and associated piping were removed. The effort required to clean the CUF has been greatly reduced by these changes.

- A data acquisition system was added so that pressure, temperature, and slurry flow rate would automatically be recorded. The permeate flow rate is still measured with a graduated site-glass and stopwatch.

- A funnel was added to the backpulse chamber so that, for example, cleaning chemicals could be added for backpulsing without sending them through the CUF and causing dilution.

- Drains were added to the filter housing and the suction side of the Oberdorfer pump to enhance recovery of solids and increase the ease of cleaning.

During the tests, the slurry temperature was maintained at 25 ± 5°C by pumping cooling water through the heat exchanger just downstream of the magnetic flow meter. The slurry temperature was measured by a thermocouple installed in the slurry reservoir and controlled by a proportional-integral-derivative (PID) temperature controller that was part of the chiller.

Deionized (DI) water and dilute caustic (0.01 M NaOH) were added to the CUF in measured volumes through a chemical addition tank located outside the hot cell. The chemical addition tank was hard piped into the cell where a long piece of flexible tubing was attached that could be gravity drained into the slurry reservoir. Concentrated caustic or acid solutions were added to the CUF using pre-filled bottles transferred manually into the cell.

The elevated-temperature caustic leaching was performed in the slurry reservoir. The slurry was drained from the CUF, and the CUF was rinsed three times with the leaching solution and drained. The slurry reservoir was then isolated by closing valves V1 and V4, and the slurry drained from the CUF was added back into the slurry reservoir. The slurry reservoir was heated with heat tape while being stirred continuously with the agitator. A thermocouple, immersed in the slurry, measured temperature and fed the data into the temperature controller, which allowed for automatic temperature control for the 8-hour wash cycle. To minimize evaporation loss, a stainless steel lid with a small hole for the mixer shaft was used.

All measuring equipment was calibrated. The instrument uncertainties are shown in Table 2.2.

Table 2.2. Instrument Uncertainties

Instrument	Uncertainty
Type K thermocouples	± 2.2°C
Pressure gauges (0-100 psi)	± 1% of full scale
Magnetic flow meter (0-8 gpm)	1% of rate at flows greater than 0.8 gpm.
Fill and drain graduated cylinder flow monitor (50-mL volume)	± 5% of measurement[(a)]
(a) The uncertainty was based on two items, the ability to measure the fill time of the graduated cylinder and the ability to read the volume. Based on an estimated fill time uncertainty of ± 0.3 seconds and a fill duration of 30 seconds, the ability to measure the time-to-fill is ± 1%. The ability to measure the level is estimated to ± 0.5 mL, as the graduated cylinder has 1-mL graduations. Based on a fill level of 10 mL, the ability to measure the volume is estimated to be ± 5%. Therefore, the cumulative error is ± 5.1%. The actual uncertainty will depend on duration of measurement and the operator.	

2.3 CUF System Verification Testing

A new, 0.1-µm, Mott "Industrial Grade" filter tube, manufactured for liquid service, was used. The filter had a 2-ft active length, 3/8-in.-ID bore, and 1/16-in. wall thickness. Before the filter was put into the hot cell, it was tested three times for 1 hour with a standard 0.35 M $SrCO_3$ slurry. The recipe for this slurry is given in Appendix A. The $SrCO_3$ slurry was used because when clean water flux (CWF) is measured, to a large extent the cleanliness of the entire CUF is being measured, rather than just the filter itself. Furthermore, it is very difficult to fully clean the CUF in the hot cell. Consequently, it has been speculated that using a standard slurry would serve to mask any particulate impurities in the CUF and would provide a better measure of the filter resistance.

In between each $SrCO_3$ slurry test, the CWF was measured to determine the associated fouling characteristics of the slurry and the amount of rinsing required after the test. Table 2.3 shows the conditions for the CWF testing and $SrCO_3$ testing. As can be seen in Figure 2.2, after each $SrCO_3$ test, the permeate flux dropped, as would be expected during the initial conditioning of a new filter. In the legend in Figure 2.2, CWF I denotes the flux of the new filter, and CWF II, CWF III, and CWF IV denote the flux after the first, second, and third $SrCO_3$ tests, respectively.

Table 2.3. Summary of Conditions During System Verification Testing

Test	TMP (psid)	Velocity (ft/)	Comments
Clean Water Flux with 0.01 M NaOH	10, 20, 30[(a)]	11	Hold each condition for 20 minutes; backpulse between conditions
0.35 M $SrCO_3$ Slurry	10, 20, 30[(a)]	11	Hold each condition for 20 minutes; backpulse between conditions
(a) If the flux was too high, the pressures were decreased.			

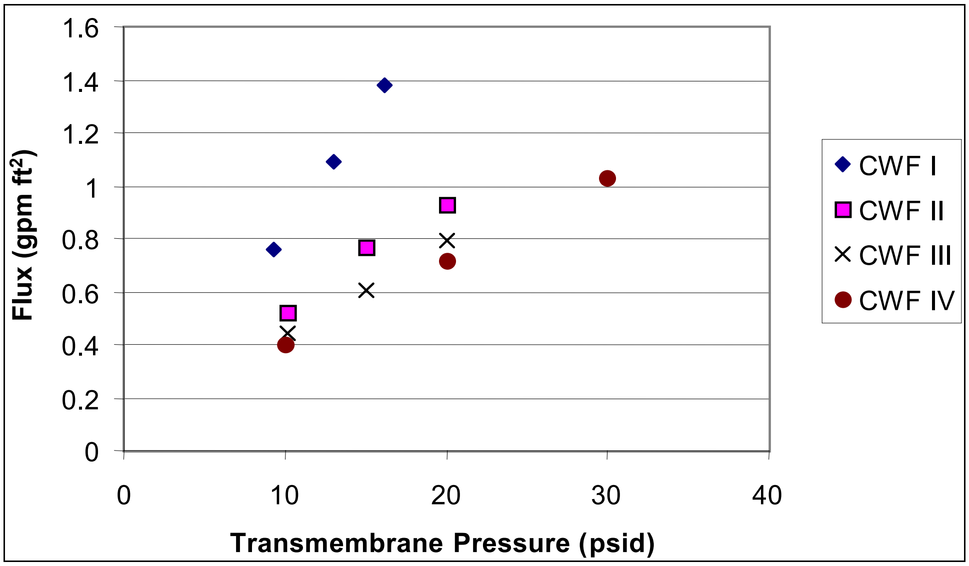


Figure 2.2. Clean Water Flux Before and After Each of Three $SrCO_3$ Tests

Figure 2.3 shows the permeate flux during the $SrCO_3$ slurry tests. In the first test, the flux decreased with pressure. This decrease is an artifact of the initial filter conditioning. In the subsequent tests, the permeate flux became fairly stable and repeatable.

After these initial tests, the filter was put into the hot cell, and the CWF test was repeated. The permeate flux was significantly lower than previously measured. Although the filter was new, the hot cell CUF

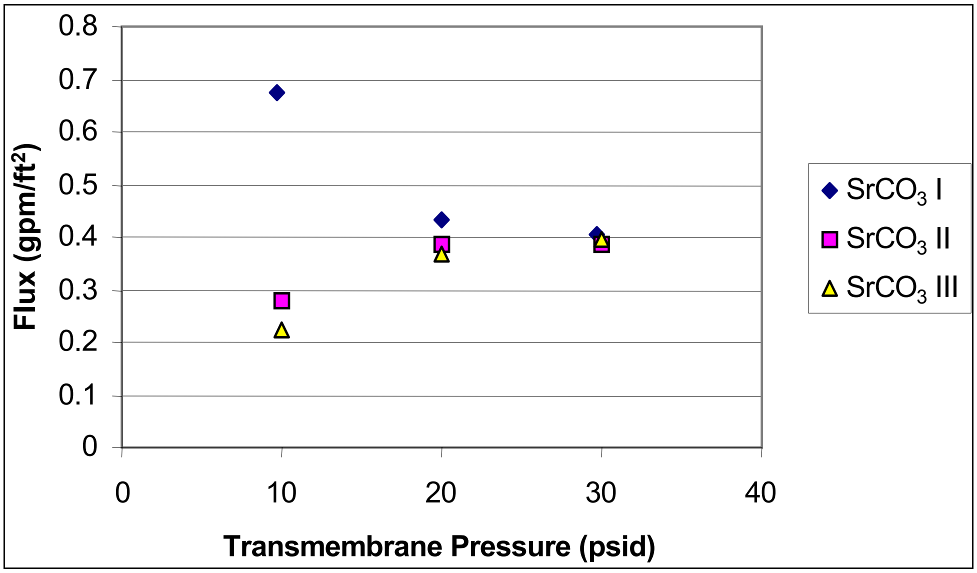


Figure 2.3. Tests with 0.35 M $SrCO_3$ Slurry

had previously been tested with AN-102/C-104 Sr/transuranic (TRU) precipitation slurry (reported in Hallen et al. 2002) [WTP-RPT-044, to be published]. Although the CUF apparatus was thoroughly cleaned after the AN-102/C-104 testing, and a new filter was installed, the CUF in the hot cell required further cleaning.

As a result, with the filter installed, the CUF was acid cleaned with 1 M HNO_3 and neutralized, and then the CWF was measured. (Refer to Figure 2.4 for a comparison of the CWF results.) The acid cleaning did not improve the rates, so the CUF was cleaned with 1 M NaOH and again neutralized. This caustic cleaning did little to improve the CWF. The filter was then tested with the standard $SrCO_3$ slurry, and the flux increased significantly. It is surmised that the effect of the $SrCO_3$ was to coat the filter surface with a permeable cake, essentially acting as a filter aid and thereby increasing the flux.

The improved flux after $SrCO_3$ testing was lower than that measured before the filter was installed in the hot cell. Nevertheless, the CWF was still high compared with previous CUF testing. For example, the CWF reported prior to testing Tank AN-102 by the Savannah River Technology Center (SRTC) was approximately 0.14, 0.20, and 0.31 gpm/ft^2 at 10, 15, and 20 psid, respectively (Nash et al. 2000). As a result, we decided to begin testing the AZ-101 slurry without further cleaning.

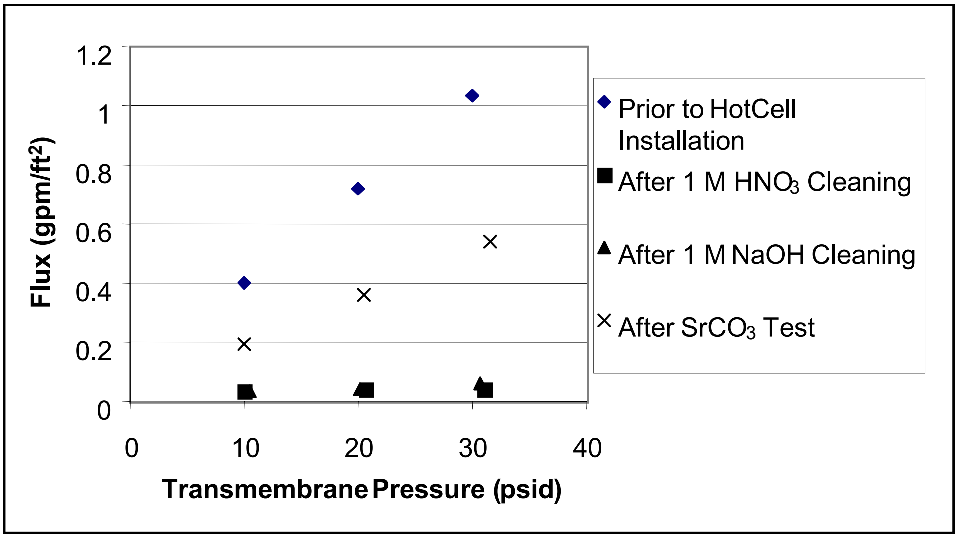
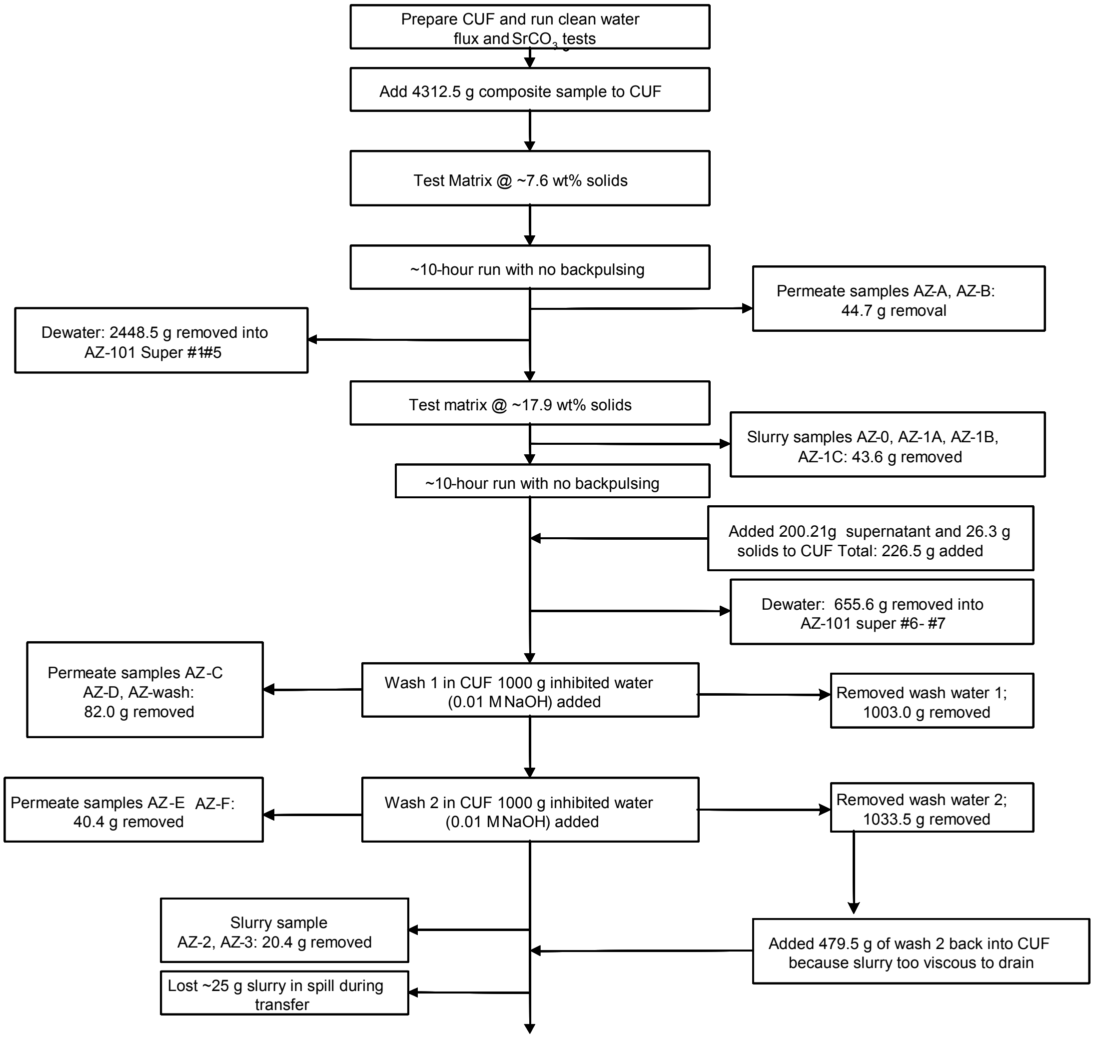


Figure 2.4. Clean Water Flux Prior to Tank AZ-101 Testing

2.4 Experimental Approach

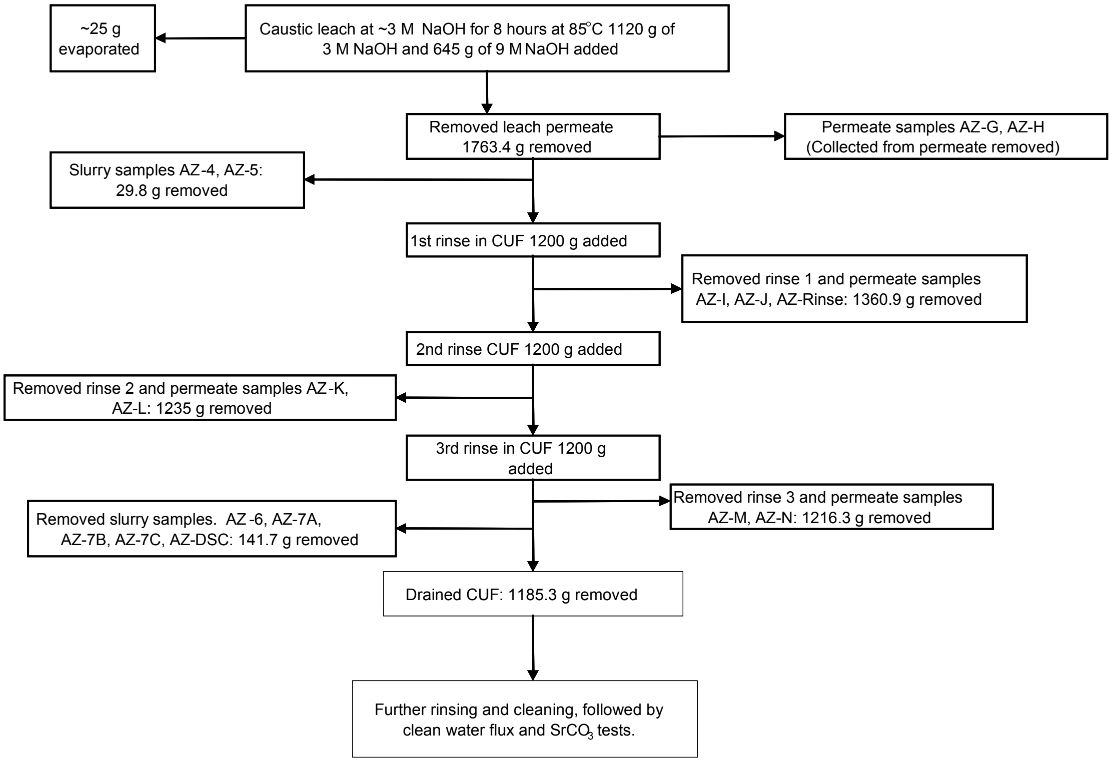
A schematic of the testing procedure is shown in the flowsheet in Figure 2.5. Appendix B shows the additions to and removals from the CUF during testing.

The Tank AZ-101 material that was used for these tests was very cohesive/adhesive in nature. It was hard to transfer because the material would form a thick clinging layer on all the tools and sides of the vessels. Similar observations were also noted for material tested from Tank AZ-102 (Brooks et al. 2000b).



(a)

Figure 2.5. Summary of AZ-101 Experimental Steps



(b)

Figure 2.5. (contd)

Before the AZ-101 testing began, a CWF test was run with 0.01 M NaOH. Following this test, $SrCO_3$ slurry was tested in the CUF. After the $SrCO_3$ slurry testing, the CUF was thoroughly rinsed and the CWF was again measured. At the conclusion of these tests, 4313 g of 7.6 wt% undissolved solids AZ-101 slurry were loaded into the CUF. The sodium molarity of the slurry supernatant was measured to be 4.5. Table 2.4 indicates the feed source. Refer to Urie et al. (2002) for the history of the samples listed in Table 2.4.

For approximately the first 7 hours after the start of testing, the system showed large variations in flow and pressure. These problems could have been due to large particles in the slurry or just high solids loading and the cohesive nature of the material. Ultimately, steady-state pressures and velocities were more easily achievable. This same phenomenon was seen during tests with waste from Tank AZ-102 (Brooks et al. 2000b).

As shown in Figure 2.6, the test matrix consists of 13 combinations of TMPs and crossflow velocity. The first condition (center point) was held for 3 hours before conditions were changed with a backpulse each hour. The center point was then repeated in the middle and at the end of testing to assess the effect of filter fouling over the course of testing. The system was backpulsed once between each condition.

Table 2.4. Tank AZ-101 Feed Source

After Compositing and Mixing; Sub-Sample ID	Mass (Gross) (g)	Bottle Tare Weight (g)	Net Weight (g)	Approx. Density (g/mL)	Original Bottle Tare (g)	Residue Left in Bottle (g)
AZ-101-RHEO-3%	186.00	144.40[(a)]	41.60	1.25	133.22	11.18
AZ-101-RHEO-13%	421.74	325.64	96.10	1.27	320.66	4.98
AZ-101-RHEO-20%	419.31	361.10[(a)]	58.21	1.28	318.97	42.13
AZ-101-AR-A	845.35	322.55	522.80	1.26	318.09	4.46
AZ-101-AR-B	888.25	324.88	563.37	1.24	320.94	3.94
AZ-101-AR-C	884.33	325.18	559.15	1.24	318.72	6.46
AZ-101-AR-D	900.48	323.71	576.77	1.27	319.85	3.86
AZ-101-AR-E	863.26	325.51	537.75	1.23	319.35	6.16
AZ-101-AR-F	905.00	322.10	582.90	1.24	318.48	3.62
AZ-101-AR-G	897.92	326.57	571.35	1.23	320.83	5.74
AZ-101-AR-H	528.50	326.03	202.47	1.19	320.57	5.46
Total	7740.14	3427.67	4312.47[(a)]	Not applicable	3329.68	97.99

(a) This is the mass before a second rinsing with AZ-101 supernatant that occurred between tests 1.29a and 1.29b. For the second rinse, 200.21 g of AZ-101 supernatant were added to jars AZ-101-RHEO-3% and AZ-101-RHEO-20% in an attempt to remove more solids. After rinsing, the jar mass was 140.19 g and 339.23 g respectively; the total added to the CUF was (4312.5 g in CUF plus 200.21 g supernatant and 26.25 g of settled solids) 4539 g.

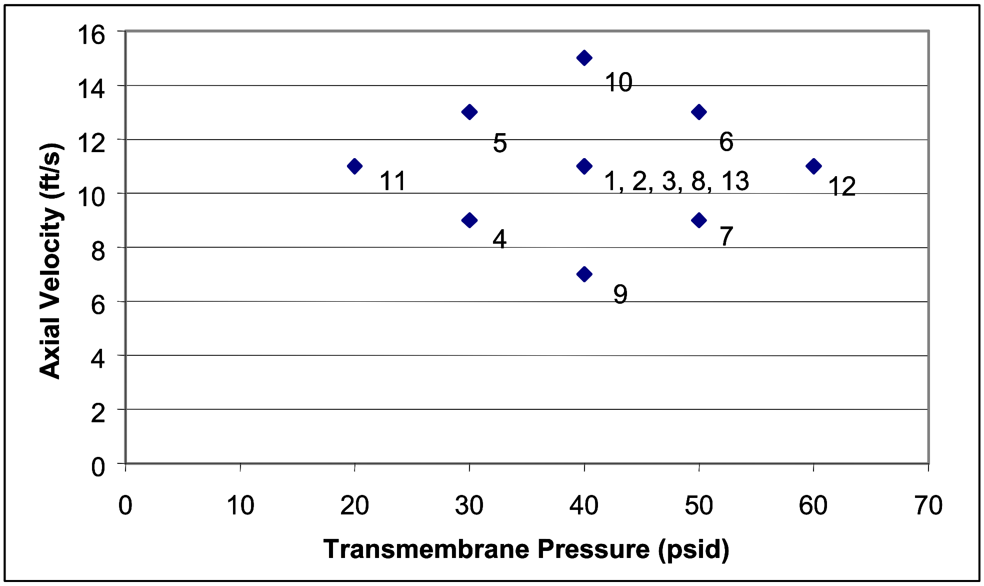


Figure 2.6. AZ-101 Crossflow Filtration Test Experimental Steps (Conditions 1 – 13)

After testing with the first matrix was completed, the system was run for ~10 hours at 40 psid TMP and 11 ft/s axial velocity without backpulsing. After the extended run, representative permeate samples were taken; the slurry was dewatered, and 2448 g of permeate were collected in five bottles labeled AZ-101 Super #1 through #5. At this point the solids concentration in the CUF was 17.9 wt% undissolved solids.

The test matrix and the extended run were repeated at 17.9 wt% undissolved solids loading, and representative samples of the slurry were taken for analysis. After the extended run, additional AZ-101 solids and supernatant were added to the CUF, because two of the jars holding the AZ-101 feed (AZ-101-RHEO-3% and AZ-101-RHEO-20%) still contained appreciable solids. Two hundred grams of AZ-101 supernatant, available in the Shielded Analytical Laboratory (in the RPL), were transferred to the HLRF and used to further rinse these jars. After the rinsing, 26.25 g of settled solids and 200.21 g of AZ-101 supernatant were added to the CUF.

After the transfer, the slurry was further dewatered, and 656 g permeate were collected in two bottles labeled AZ-101 Super #6 and #7. The slurry was 24.7 wt% undissolved solids. The solids were washed with two batches of 1000 g of 0.01 M NaOH solution. The slurry was dewatered after each batch addition, and a total of 2159 g of solution were removed. Permeate samples were taken during each wash, and slurry samples were taken after the second wash. After the two washes, the measured undissolved solids concentration in the slurry was 27.0 wt%.

The CUF should then have been drained so that all of the slurry would be contained in the slurry reservoir to prepare for caustic leaching. However, the slurry was so thick that it would not gravity drain out of the 1/2-in. tubing (3/8-in. ID). Because the slurry was so viscous, 480 g of the second wash permeate were added, and the CUF was then drained and rinsed with the leaching solution (i.e., 1120 g of 3 M NaOH solution). During the transfer, some of the slurry (~40 mL) spilled onto the catch pan, and approximately 20 mL could not be recovered. The catch pan had been cleaned prior to running the CUF. The drained slurry was added back into the isolated slurry reservoir, and 645 g of 9 M NaOH leaching solution were added. The slurry was heated with agitation to 85°C for 8 hours. The calculated hydroxide concentration during the leach was 2.8 M (targeted value was 3.0 M). After leaching, the slurry was dewatered, and 1793 g of leach solution were removed.

The slurry was then batch rinsed three times with 0.01 M NaOH. Each rinse consisted of 1200 g of 0.01 M NaOH added to the slurry. A total of 3600 g of rinse solution were added during the three rinses, and 3812 g were removed, including permeate samples taken between each rinse. After dewatering following the third rinse, representative slurry samples were taken for physical, chemical, radiochemical, and rheological analysis. The final concentration of undissolved solids measured 10.9 wt%.

The CUF was then drained, and 1185.3 g of final washed and leached AZ-101 sludge were collected into a 2-liter bottle. The CUF was rinsed three times with a total of 1950 g of 0.01 M NaOH solution, and the solids were collected to be settled and recovered. The final washed sludge was transferred into a storage container for melter feed rheological studies and vitrification tests.

2.5 Sample Analyses

The samples taken during the filtration testing are shown in Table 2.5. The samples and their analyses are described below. All sample designations actually include the prefix "AZ," which is generally not listed here for brevity.

1. Appendix C, Table 1, provides the analytes, analysis methods, and the minimum reportable quantities (MRQs) for both the solid and liquid samples (except sample 6). The liquid samples were acid digested and analyzed. The slurry was analyzed following drying and separate fusions with both KOH and NaOH.

2. Appendix C, Table 2, provides the analytes, analysis methods, and the MRQs for slurry sample 6. The slurry was analyzed following drying and separate fusions with both KOH and NaOH. Appendix C, Table 2, is the combined analyte list agreed upon by the Contractor Pretreatment and Waste Form Qualification leads.

3. Analyses were performed to determine weight % total solids, weight % undissolved solids, and slurry density on slurry samples 1, 3, 5, and 7. The centrifuged solids volume was measured on all samples prior to physical analyses.

4. Particle size distribution and rheological analyses were performed on samples 1 and 7.

5. The permeates were sampled approximately midway through the first dewatering step (sample a). Wash, leach, and rinse solutions were also taken (samples c, e, g, i, k, and m,). Duplicate sub-samples were acquired.

Table 2.5. Sampling and Analyses During Testing

Process Step	Condition	Liquid Sample	Slurry Sample	Analysis Description
As-received material	Prior to 1.3	--	Note 1	Appendix C, Table 1, Physical Properties 1
Dewatering	1.16A	a, (b)	--	Appendix C, Table 1
High solids matrix at ~17.9 wt% solids	1.29	--	0, 1a, 1b, 1c	Physical Properties 1
Wash 1 (0.01 M NaOH)	1.29.1	c, (d)	--	Appendix C, Table 1
Wash 2 (0.01 M NaOH)	1.29.3	e, (f)	--	Appendix C, Table 1
Wash 2 (0.01 M NaOH)	1.29.3	--	2, 3	Appendix C, Table 1, Physical Properties 2, vol centrifuged solids
3 M NaOH Leach	1.29.4	g, (h)	--	Appendix C, Table 1
3 M NaOH Leach	1.29.4	--	4, 5	Appendix C, Table 1, Physical Properties 2, vol centrifuged solids
Rinse 1 (0.01 M NaOH)	1.29.5	i, (j)	--	Appendix C, Table 1
Rinse 2 (0.01 M NaOH)	1.29.5A	k, (l)	--	Appendix C, Table 1
Rinse 3 (0.01 M NaOH)	1.30	m, (n)	--	Appendix C, Table 1
Rinse 3 (0.01 M NaOH)	1.30	--	6, 7a, 7b, 7c	Appendix C, Table 2, Physical Properties 1

The samples in ( ) were archived.

Physical Properties 1: wt% undissolved solids, wt% total solids, slurry density, particle size distribution, viscosity. Note that the Physical Properties 1 required three samples (a, b, and c). The first sample (a) was ~10 mL and was used to determine wt% undissolved solids, wt% total solids, and slurry density. The second sample (b) was ~2 mL and was used to measure the PSD. The third sample (c) was ~40 mL and was used to measure the viscosity. Analysis of the third sample was completed during testing, and the sample was returned to the CUF after analysis.

Physical Properties 2: wt% undissolved solids, wt% total solids, slurry density.

Note 1: Physical properties and analytical performed as part of the homogeneity work were used in lieu of a separate analysis.

3.0 Results from Filtration, Sludge Washing, and Caustic Leach Testing

This section provides the results for crossflow filtration, sludge washing, and caustic leaching. Section 3.1 discusses the permeate fluxes measured during testing, and Section 3.2 discusses the chemical and radiochemical analyses obtained from the slurry washing and caustic leaching tests

3.1 Crossflow Filtration Results

The low and high solids loading matrix consisted of 13 conditions, as indicated in Figure 2.6. The low solids matrix was performed at 7.6 wt% undissolved solids concentration, and the high solids matrix was performed at 17.9 wt% undissolved solids concentration. Each condition in the matrix was 1 hour in duration. All the flux data presented in this section have been corrected to 25°C using the following formula to correct for viscosity and surface tension changes:

$$\mathrm{Flux}_{25C} = \mathrm{Flux}_{T}\, e^{2500\left(\frac{1}{273+T}-\frac{1}{298}\right)} \tag{3.1}$$

where Flux_{25C} is the corrected permeate flux, and T is the temperature (in °C) at the flux measurement (Flux_T). Analysis indicates that the Contractor design basis of 0.014 gpm/ft^2 can be met for both the low and high solids slurries. All of the raw data for the permeate flux measurements are included in Appendix D. This appendix also graphs the flux versus time for the entire run with the AZ-101 slurry.

3.1.1 Low Solids Loading (7.6 wt% Solids) Matrix

The average permeate flux (excluding the first 10 minutes of operation) from the 13 test conditions is shown in Table 3.1. A graph of the permeate flux as a function of time for conditions 1, 2, 3, 8, and 13 (the center points of the matrix) is shown in Figure 3.1. The benefits from backpulsing in terms of increased flux are short in duration, as can be seen by the small initial decline in flux. The flux immediately after backpulsing decreases with run order (i.e., condition), but the flux beyond ~30 minutes after backpulsing shows little dependency with run order. This lack of dependency with run order is in contrast with previous crossflow filtration studies on Hanford tank wastes (Brooks et al. 2000a,b; Geeting and Reynolds 1997).

Figures 3.2 and 3.3 show the average flux plotted as a function of TMP and axial velocity, respectively. The flux is principally dependent on the TMP, which is typical of low solids slurries. In contrast, the axial velocity shows almost no influence on the flux. The lines in Figures 3.2 and 3.3 represent a linear regression through the data. In Figure 3.3 the linear regression is meant to highlight the lack of trend. These lacks of trend are so pronounced that the experimental design (test matrix) can be seen in Figure 3.3, since velocity has so little impact on flux and TMP is so highly correlated with flux. The scatter in the data may be an artifact of how difficult it was to maintain the axial velocity and TMP at the targeted conditions during the first ~7 hours of testing. It is somewhat surprising that a slurry of 7.6 wt% solids behaved in a manner typical of much lower solids slurries.

Table 3.1. Average Permeate Flux for Low Solids Matrix

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Permeate Flux (gpm/ft^2)
1	9.9	44.5	0.033
2	10.6	43.5	0.030
3	10.8	42.2	0.030
4	8.1	29.8	0.028
5	13.3	30.4	0.023
6	12.7	51.5	0.031
7	8.9	51.9	0.029
8	11.0	41.3	0.031
9	7.0	41.2	0.024
10	14.9	42.5	0.026
11	10.8	21.6	0.018
12	10.6	61.3	0.036
13	11.0	41.5	0.031

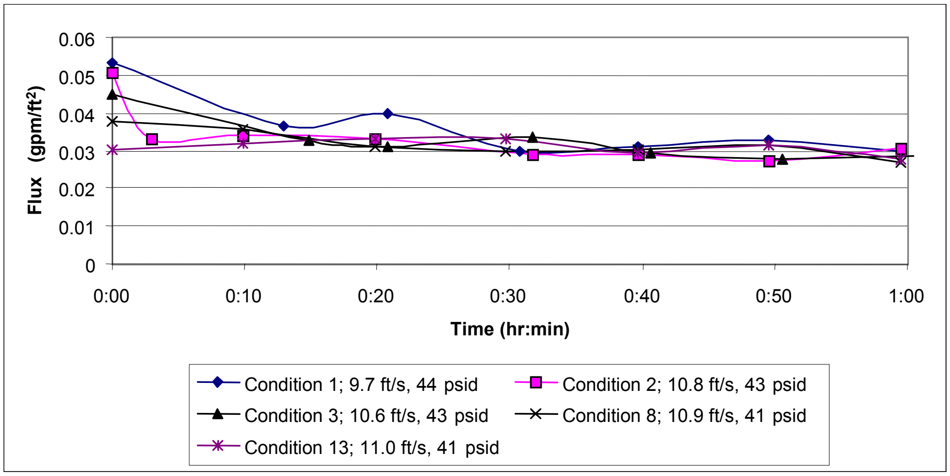


Figure 3.1. Permeate Flux as a Function of Time for the Low Solids Matrix

The whole model (linear) fit shows an Rsquare of 0.707; that is, roughly 71% of the variation in permeate flux is captured by the model. The largest contributor to the model is TMP, which, by itself, captures 64% of the variation in permeate flux. Adding time to the model increases Rsquare to 0.706. The model is:

$$\text{Flux} = 0.0154 + 3.81 \times 10^{-4} \times \text{TMP} - 6.7 \times 10^{-5} \times \text{Velocity} - 3.02 \times 10^{-4} \times \text{Time} \tag{3.2}$$

With Flux in gpm/ft^2, TMP in psid, Velocity in ft/s, and Time in hours.

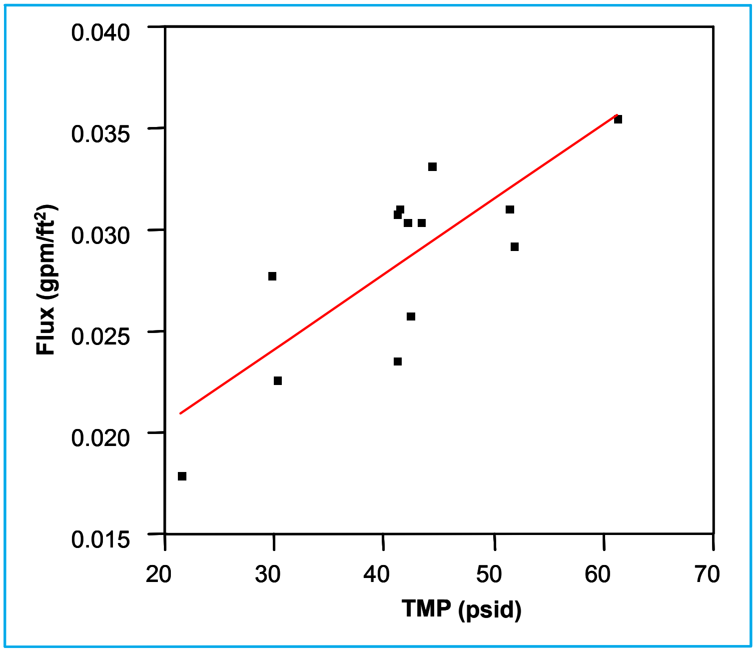


Figure 3.2. Effect of Transmembrane Pressure on Permeate Flux

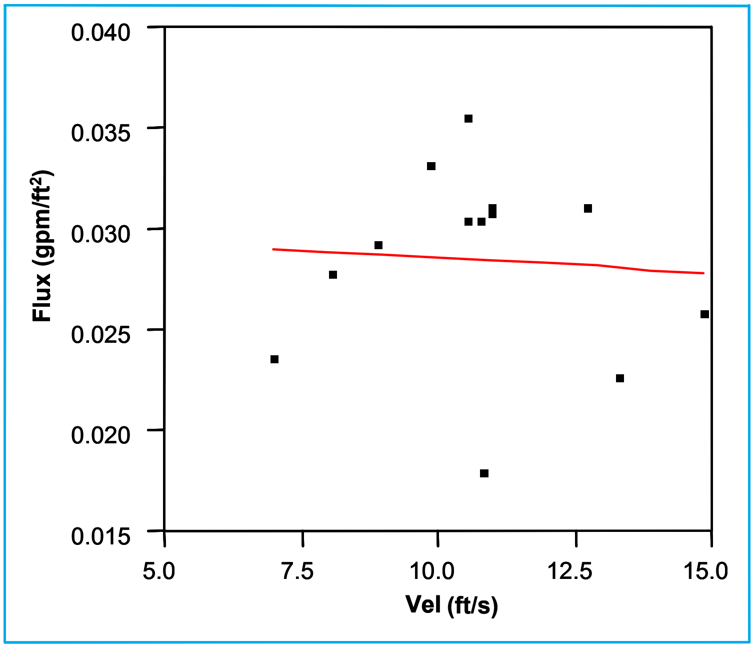


Figure 3.3. Effect of Axial Velocity on Permeate Flux

3.1.2 High Solids Loading (17.9 wt% Solids) Matrix

The average permeate flux (excluding the first 10 minutes of operation) from these conditions is shown in Table 3.2. A graph of the permeate flux as a function of time for conditions 1, 2, 3, 8, and 13 (the center points of the matrix) is shown in Figure 3.4. As seen for the low solids slurry, the flux displayed little dependency on run order, as the flux was consistent between conditions. The benefits from backpulsing in terms of increased flux again are minor and short in duration.

Figures 3.5 and 3.6 show the average flux plotted as a function of TMP and axial velocity, respectively. In contrast with the low solids slurry, the flux is principally dependent on the axial velocity with almost

Table 3.2. Average Permeate Flux for High Solids Matrix

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Permeate Flux (gpm/ft^2)
1	11.1	40.6	0.022
2	10.7	40.8	0.022
3	11.0	41.5	0.022
4	8.8	31.3	0.016
5	13.2	29.5	0.023
6	12.7	49.1	0.023
7	9.2	47.3	0.014
8	11.0	39.6	0.020
9	7.3	42.8	0.011
10	14.8	39.3	0.025
11	11.4	21.4	0.019
12	10.7	59.6	0.018
13	10.9	41.3	0.019

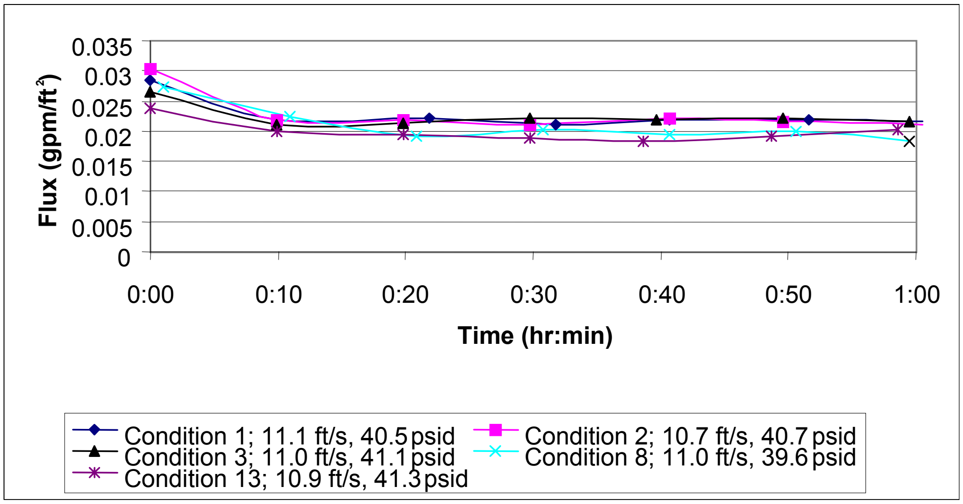


Figure 3.4. Permeate Flux as a Function of Time for the High Solids Matrix

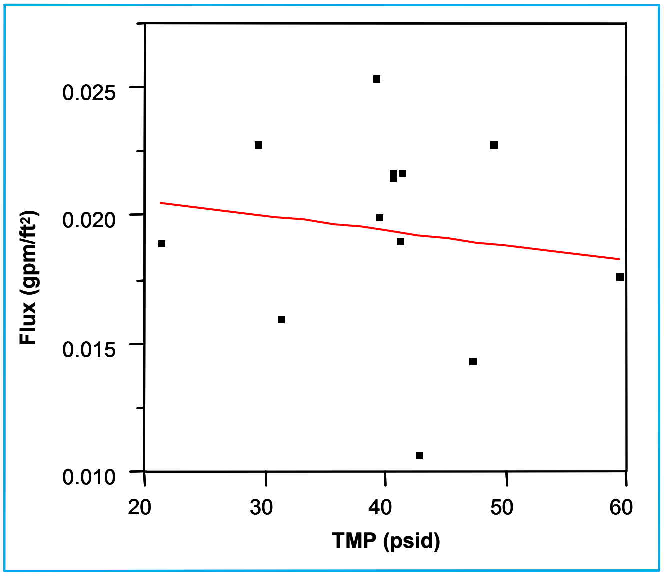


Figure 3.5. Effect of Transmembrane Pressure on Permeate Flux

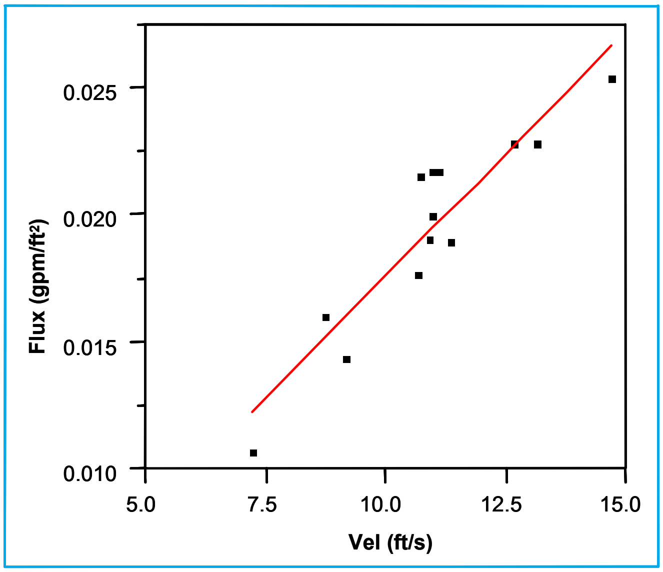


Figure 3.6. Effect of Axial Velocity on Permeate Flux

no dependency on the TMP. The lines in Figures 3.5 and 3.6 represent a linear regression through the data. In Figure 3.5 the linear regression shown is meant to highlight the lack of trend. Because TMP has so little impact on flux and the axial velocity is so highly correlated, the experimental design (test matrix) can actually be seen in Figure 3.5.

The whole model (linear) fit shows an Rsquare of 0.944; that is, roughly 94% of the variation in permeate flux is captured by the model. The largest contributor to the model is axial velocity, which, by itself, accounts for 86% of the variation in permeate flux. Adding time to the model increases Rsquare to 0.943. The model is:

$$\text{Flux} = -2.83 \times 10^{-4} + 8 \times 10^{-6} \times \text{TMP} + 1.96 \times 10^{-3} \times \text{Velocity} - 3.02 \times 10^{-4} \times \text{Time} \qquad (3.3)$$

With Flux in gpm/ft^2, TMP in psid, Velocity in ft/s, and Time in hours.

3.1.3 Extended Runs and Dewatering of Untreated AZ-101

After the tests with the low and high solids matrices, the slurry was tested for ~10 hours at 11 ft/s axial velocity and 40 psid TMP, without backpulsing. The results are shown in Figure 3.7. After the first 100 to 200 minutes the slurry flux generally stopped decreasing and held within a range. The range for the high solids slurry showed less variability. The average permeate flux (excluding the first 200 minutes) was 0.026 and 0.016 gpm/ft^2 for the low and high solids slurry, respectively.

After each of the extended runs, the slurry was dewatered at 11 ft/s axial velocity and 40 psid TMP. The first dewatering brought the slurry from 7.6 to 17.9 wt% undissolved solids. The second dewatering, just prior to the first sludge washing, brought the slurry from 17.9 to 24.7 wt% undissolved solids. The system was not backpulsed during either dewatering. Figure 3.8 displays the average flux from each of the extended runs together with the instantaneous dewatering flux measured as a function of the log of the solids concentration, Cs. This same information together with the measured axial velocity and TMP is provided in Table 3.3.

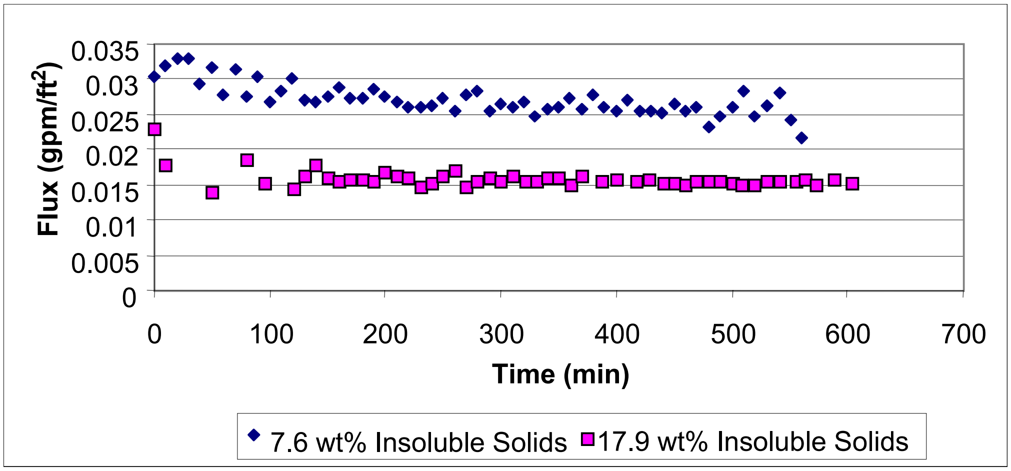


Figure 3.7. Extended Run Without Backpulsing

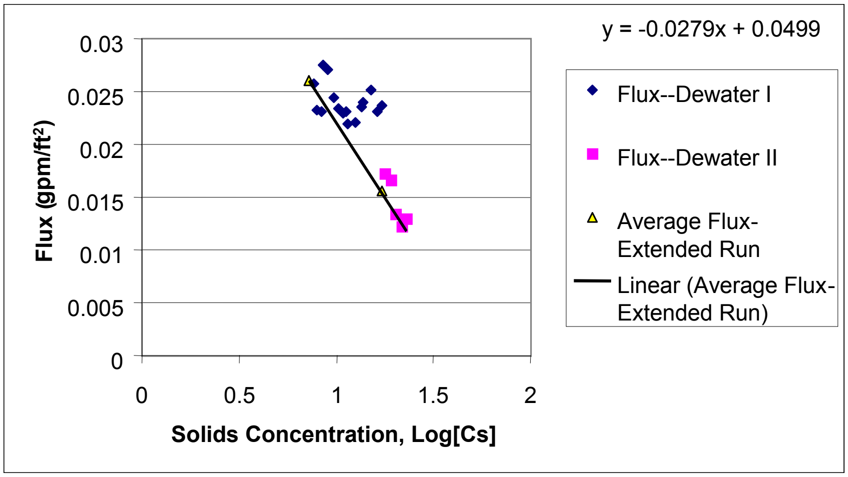


Figure 3.8. Untreated Tank AZ-101 Flux at Various Solids Loadings

Table 3.3. Permeate Flux of Untreated AZ-101 at Various Solids Loadings

Wt% Undissolved Solids	TMP (psid)	Axial Velocity (ft/s)	Flux (gpm/ft^2)
7.6	41.0	10.5	0.0258
7.6[(a)]	39.8[(a)]	11.1[(a)]	0.0260[(a)]
8.0	37.0	10.7	0.0232
8.4	40.0	11.0	0.0231
8.6	41.0	10.7	0.0275
9.1	42.0	10.5	0.0270
9.7	42.0	11.0	0.0244
10.2	40.0	11.0	0.0234
10.8	40.0	10.7	0.0229
11.3	41.0	9.9	0.0232
11.5	40.0	10.7	0.0220
12.6	39.0	11.3	0.0221
13.5	40.0	10.7	0.0235
13.8	39.0	10.2	0.0240
15.0	42.0	10.5	0.0252
16.2	41.0	10.2	0.0231
17.3	48.5	9.3	0.0237
17.9[(a)]	41.5[(a)]	11.0[(a)]	0.0155[(a)]
17.9	40.5	11.0	0.0173
19.3	41.0	10.5	0.0166
20.3	42.0	11.0	0.0134
21.6	40.5	10.7	0.0122
23.2	43.5	10.5	0.0130
(a) Averaged data from extended run (minus first 200 minutes)			

An increase in TMP should cause the line to become steeper. Decreasing TMP is expected to have the opposite effect. The line is based solely on the 10-hour averages during the extended run, and not the instantaneous data obtained during the actual dewatering. The data from the dewatering correspond well with the averages, although there is a drop between the first and second dewatering. This drop is attributed to the fact that Tank AZ-101 slurry in the second dewatering ran through the CUF 24 hours longer than the first. As a result, there may have been some particle attrition between dewaterings. This theory is supported by the PSD data (see Section 4.3), which indicate that the particle size decreased during the testing.

3.1.3.1 Dewatering of Wash 1 and Wash 2

After the second dewatering, the AZ-101 slurry was batch washed twice with a 1-liter batch of inhibited water (0.01 M NaOH), and dewatered at 11 ft/s axial velocity and 40 psid TMP. Because all dewaterings were conducted at the same axial velocity and TMP without backpulsing, the results should have been directly comparable with the first dewatering data. Figure 3.9 shows the flux during the dewatering as a function of solids concentration. Table 3.4 shows similar data in tabular form. The flux during the dewatering from wash 1 and wash 2 was significantly higher than the previous dewatering of the original supernatant, resulting in a steeper line. The increase in flux is attributed to the decreased viscosity of the fluid.

The effect of viscosity on the permeate flux was determined as follows. The viscosity of AZ-101 supernatant was previously measured to be approximately 2 cP at 65°C.[a] The viscosity, corrected to 25°C, was calculated to be 4.1 cP by assuming that the viscosity of the supernatant liquid changed proportionally to water as a function of temperature (for comparison, if all the sodium measured in the original Tank AZ-101 supernatant were assumed to be from sodium hydroxide, the corresponding

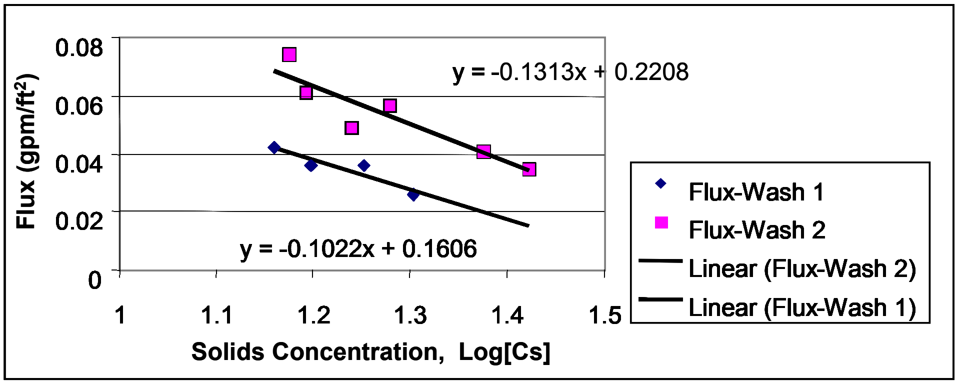


Figure 3.9. Dewatering During Wash 1 and Wash 2

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[a] Peterson, ME, RD Scheele, and JM Tingey. 1989. "Characterization of the First Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 101-AZ." Internal Letter Report, Pacific Northwest National Laboratory, Richland, WA.

Table 3.4. Permeate Flux of Washed AZ-101 at Various Solids Loadings

Wt% Undissolved Solids	TMP (psid)	Axial Velocity (ft/s)	Flux (gpm/ft^2)	Dewatering Step
14.5	44.0	10.2	0.0424	Wash 1
15.8	40.6	10.6	0.0360	Wash 1
17.9	41.5	10.7	0.0359	Wash 1
20.2	42.5	10.5	0.0256	Wash 1
15.0	Variable(a)	Variable(a)	0.0741	Wash 2
15.6	40.5	10.5	0.0609	Wash 2
17.3	39.0	10.2	0.0491	Wash 2
19.0	35.5	11.3	0.0567	Wash 2
23.7	42.0	10.2	0.0409	Wash 2
26.5	41.0	10.7	0.0344	Wash 2

(a) Flow and pressure varied during reading.

viscosity of such a solution would be 3.6 cP). The viscosity of the wash 1 and wash 2 supernatants was calculated to be 1.6 and 1.24 cP, respectively, by assuming that their viscosity changed, as a function of their measured sodium concentration, proportionally to that of sodium hydroxide. Figure 3.10 displays the permeate flux of the initial dewatering and the wash 1 and wash 2 dewatering calculated from the equations in Figures 3.8 and 3.9. Not unexpectedly, the data indicate that the permeate flux is proportional to $(\text{viscosity})^{-1}$. The linear fits shown have a forced zero intercept. These data were compared with a crossflow filtration model, with the results presented in Appendix E.

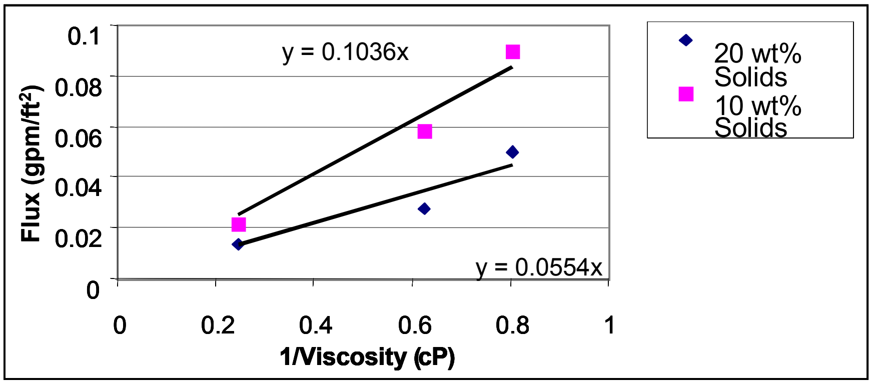

Figure 3.10. Effect of Viscosity on the Permeate Flux

3.1.3.2 Dewatering of the Leached Slurry and Subsequent Rinses

After the 8-hour leach, the slurry was dewatered. The slurry was then batch rinsed three times with 1200 g/batch of inhibited (0.01M NaOH) water and dewatered. All of the dewaterings were at 11 ft/s axial velocity and 40 psid TMP. The flux during each of these dewaterings is shown in Figure 3.11. Table 3.5 shows similar data in tabular form. It is interesting that after the leach, the flux displayed little or no decrease with increasing solids concentration. The flux of each rinse was higher than the previous rinse, which again is most likely due to decreasing viscosity of the permeate. In all cases, the permeate flow rate was fast and did not require any backpulsing.

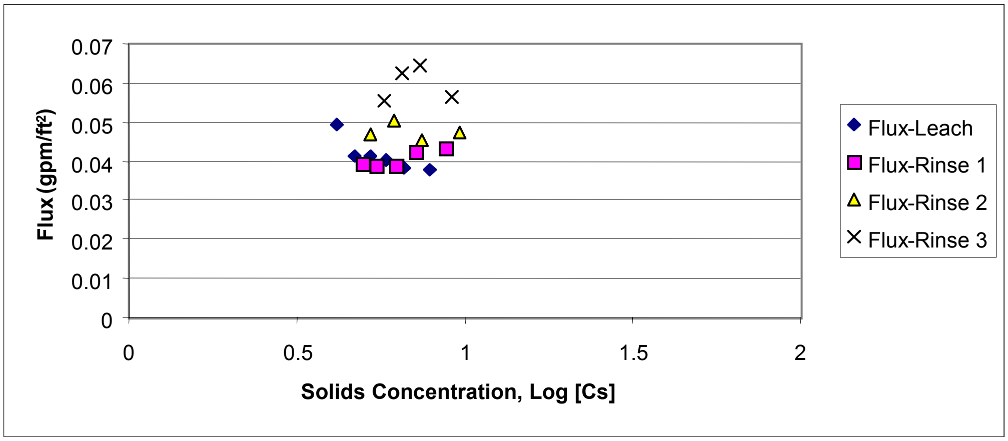

Figure 3.11. Dewatering of the Leached and Rinsed AZ-101 Slurry

Table 3.5. Permeate Flux of Leached and Rinsed AZ-101 at Various Solids Loadings

Wt% Undissolved Solids	TMP (psid)	Axial Velocity (ft/s)	Flux (gpm/ft^2)	Dewatering Step
4.2	44.0	10.2	0.0494	Leach
4.7	38.0	11.3	0.0413	Leach
5.3	39.0	11.0	0.0413	Leach
5.8	38.0	11.3	0.0401	Leach
6.6	40.0	10.5	0.0384	Leach
7.8	40.5	10.7	0.0379	Leach
5.0	40.0	10.7	0.0391	Rinse 1
5.5	40.0	10.2	0.0388	Rinse 1
6.2	40.0	10.2	0.0390	Rinse 1
7.2	41.5	10.7	0.0421	Rinse 1
8.8	43.0	10.5	0.0433	Rinse 1
5.3	43.5	11.0	0.0468	Rinse 2
6.1	45.0	11.0	0.0506	Rinse 2
7.4	41.0	11.3	0.0452	Rinse 2
9.7	44.0	10.7	0.0474	Rinse 2
5.8	42.5	11.6	0.0553	Rinse 3
6.5	44.4	11.2	0.0626	Rinse 3
7.3	42.0	10.9	0.0644	Rinse 3
9.1	41.0	11.0	0.0566	Rinse 3

3.1.3.3 Post-Test $SrCO_3$ Slurry and Clean Water Flux

After Tank AZ-101 testing was completed, the CUF was drained and thoroughly rinsed 11 times with a total of 7 liters of inhibited water (0.01 M NaOH), and a CWF test was performed. The results are shown in Figure 3.12. As expected, the CWF decreased compared with the measurement prior to testing. Conducting the $SrCO_3$ slurry test before the CWF testing improved the CWF rates. Consequently, we decided to determine if this phenomenon was repeatable after testing Tank AZ-101. The $SrCO_3$ slurry was run, and the CUF was again thoroughly rinsed, four times, with a total of 3 liters of inhibited water. The CWF post-$SrCO_3$ testing was also improved. Finally, the system was acid cleaned with 2 M HNO_3 and then neutralized. The CWF again showed some improvement, but was still lower than that measured before testing Tank AZ-101.

If judged by the CWF measurements alone, the filter appeared to be irreversibly fouled during the course of the AZ-101 testing. However, this fouling is not evident when judged by the $SrCO_3$ testing. As can be seen in Figure 3.13, the $SrCO_3$ slurry flux was approximately equivalent before and after testing. The

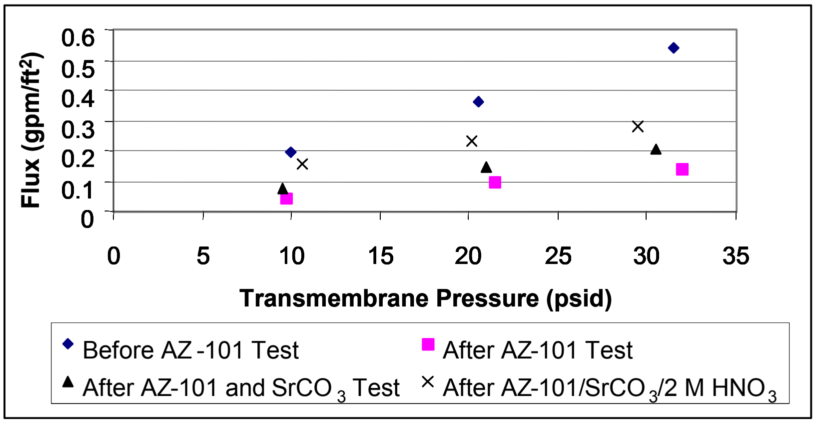

Figure 3.12. Clean Water Flux Testing

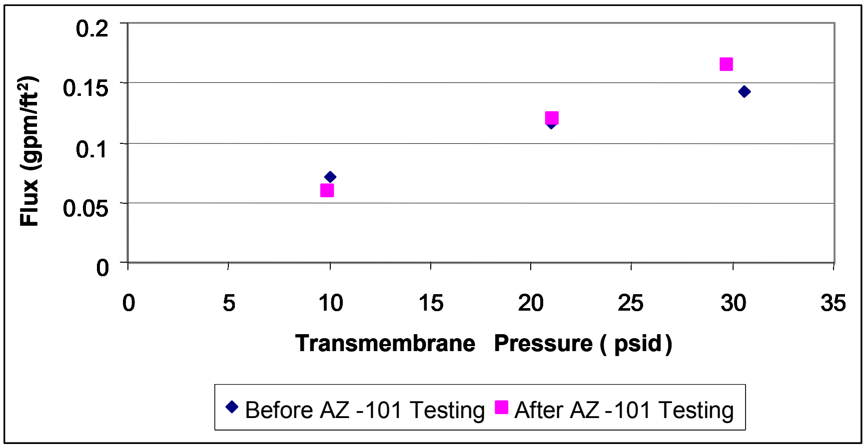

Figure 3.13. $SrCO_3$ Slurry Tests

AZ-101 testing was followed by $SrCO_3$ testing before acid cleaning the filter. The $SrCO_3$ slurry results better reflect the AZ-101 filtration data in that little time dependency of permeate flux or irreversible fouling was observed.

The $SrCO_3$ slurry testing is recommended for future testing, as it appears that the CWF measurement is, to a large extent (at least in the hot cell where it is difficult to fully clean the system), measuring the cleanliness of the CUF, rather than the filter alone. The $SrCO_3$ slurry seems to act as a filter aid by masking impurities in the CUF and providing filter resistance measurements consistent with test results observed.

3.2 Sludge Washing and Caustic Leaching Results

The chemical and radiochemical analyses obtained from the slurry washing and caustic leaching tests are presented in this section. Slurry samples were taken from the concentrated 17.9 wt% undissolved solids feed, following the two water washes, following the caustic leach, and at the conclusion of the tests (following the three final rinses). Liquid samples were taken of all permeates removed throughout the course of the wash/leach/rinse steps.

Nonradioactive component concentrations in the slurry samples are presented in Table 3.6 and reported on a dry total solids basis. The inductively coupled plasma-atomic emission spectroscopy (ICP-AES) data used in Table 3.6 represent an average of results from the NaOH and KOH fusions. All analytical results are provided in Appendix F.

Table 3.6 shows that the primary metals in the initial slurry were, from highest to lowest concentration, sodium, zirconium, aluminum, and iron. Aluminum and chromium were the principal metals removed during the caustic leaching, as well as some additional sodium. After sludge washing, caustic leaching, and rinsing, the concentrations of metals in the final slurry were, from highest to lowest, iron, aluminum, zirconium, and sodium. Table 3.6 also displays the ion chromatography (IC) results for the (water leached) slurry samples on a dry weight basis. Significant soluble anions present in the slurry were nitrite, nitrate, and sulfate.

Table 3.7 displays the nonradioactive components in the permeate samples on a volumetric basis. The primary dissolved metals in the initial permeate were sodium, aluminum, potassium, chromium, and phosphorus. Comparing concentrations in the original supernatant (AZ-A) to the rinse 3 permeate (AZ-M) indicates that the pretreatment removed a majority of the soluble analytes. One exception of (possible) consequence to the projected volume of HLW glass is aluminum, which is present in the rinse 3 permeate in a fairly significant concentration. Of the total aluminum in the post-rinse slurry sample (AZ-6), 13% is contributed by the liquid fraction, suggesting that further rinsing would reduce the total aluminum.

The total carbon (TC), total inorganic carbon (TIC), and total organic carbon (TOC) are also provided in Table 3.7. The concentrations of TC and TIC decrease due to dilution during processing. However, the concentrations of both increase significantly in the third rinse. Nothing unusual was noted during processing between the second and third rinse. The cause of this anomalous behavior is unknown and therefore somewhat suspect.

Table 3.6. Nonradioactive Component Concentrations in the Slurry

Analyte	17.9 wt% Solids Slurry (AZ-0)	Post-Wash Sample (AZ-2)	Post-Leach Sample (AZ-4)	Post-Rinse Sample (AZ-6)
ICP-AES Results	µg/g	µg/g	µg/g	µg/g
Ag	[457]	379	364	[631]
Al	95,000	186,000	79,850	100,075
Ba	382	726	726	1,510
Ca	[2,805]	4,800	4,515	8,098
Cd	3,785	7,680	7,485	14,500
Ce	[4,430]	[1,165]	[1,105]	[3,330]
Cr	1,555	2,235	1,570	2,428
Cu	[54]	[205]	[217]	583
Fe	52,750	106,000	104,600	202,500
K	[7,600]	[3,900]	[2,600]	[2,000]
La	1,560	2,965	2,940	5,808
Li	[115]	[200]	[108]	[130]
Mg	[410]	[730]	[710]	[1,200]
Mn	1,445	2,850	2,725	5,400
Mo	[160]	[90]	[66]	[66]
Na	130,000	45,500	164,000	53,600
Nd	1,185	2,265	2,225	4,350
Ni	2,760	5,390	5,880	9,850
P	1,715	[1,790]	2,175	4,505
Pb	[590]	1,085	[965]	1,785
Pd	[1,600]	[1,200]	[1,050]	[2,300]
Pr (ICP-MS)	236	450	507	883
Pt (ICP-MS)	<0.365	<0.365	<0.403	<0.325
Rh	[480]	[320]	[290]	[513]
Ru (ICP-MS)	416	813	900	1,540
Si	[4,855]	7,440	30,000	13,425
Sn	[1,600]	[1,950]	[2,100]	[3,025]
Sr	904	1,775	1,765	3,435
Ta (ICP-MS)	0.439	0.958	0.728	6.60
Ti	[54]	[96]	[91]	[178]
U (ICP-MS)	3,360	13,300	11,000	12,100
Y	[103]	[200]	[180]	[385]
Zn	[86]	[160]	[145]	[278]
Zr	14,300	24,300	21,900	65,700
IC Results				
Br	1,083	377	258	<160
Cl	<516	<117	2,646	684
C_2O_4	3,867	941	837	502
F	3,739	1,300	466	379
NO_2	112,930	36,740	24,835	7,062
NO_3	100,812	28,388	16,421	2,111
PO_4	1,753	641	746	<328
SO_4	45,378	10,128	6,695	2,352

Notes: 1. Overall error greater than 10-times detection limit is estimated to be within ±15%.
2. Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.

Table 3.7. Nonradioactive Component Concentrations in the Permeate Samples

Analyte	Original Supernatant (AZ-A)	Wash 1 Permeate (AZ-C)	Wash 2 Permeate (AZ-E)	Leach Permeate (AZ-G)	Rinse 1 Permeate (AZ-I)	Rinse 2 Permeate (AZ-K)	Rinse 3 Permeate (AZ-M)
	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
Al	5,325	2,350	1,210	14,600	7,900	3,990	2,060
As	[10]	--	--	--	--	--	--
B	69	75.9	73.2	61.5	78.3	54.7	71.9
Ca	[7]	[10]	--	--	--	--	--
Cd	[0.42]	--	--	[1.4]	--	--	--
Cr	624	217	102	135	70.0	36.0	19.1
Fe	[21]	[1.2]	[0.82]	[2.7]	[1.4]	[1.3]	[1.7]
K	4,040	1,680	866	[500]	[230]	[110]	--
Li	--	--	--	13.4	[7.7]	[4.1]	[2.3]
Mo	87	39.4	20.1	[11]	[5.4]	[2.8]	[1.5]
Na	102,500	42,300	20,800	60,600	33,700	16,000	8,640
P	494	193	83.4	81.4	37.9	[17]	[8.2]
Pb	[5]	[2.8]	--	[9.6]	[4.7]	--	--
Pd	[22]	--	--	--	--	--	--
Si	217	188	200	188	180	142	145
Sn	[50]	--	--	[49]	--	--	--
Sr	--	--	--	--	--	--	--
U	--	--	--	--	--	--	--
V	[2]	--	--	--	--	--	--
W	[55]	--	--	--	--	--	--
Zr	[2]	--	--	[1.6]	--	--	--
IC Results							
Br	1,010	430	250	<125	<125	<125	<125
Cl	<250	<125	<125	830	590	310	255
C_2O_4	1,000	1,710	910	600	430	<250	<250
F	1,670	1,410	770	430	260	<125	<125
NO_2	85,700	32,900	16,100	7,790	4,540	1,890	1,250
NO_3	71,700	29,300	13,400	6,560	3,990	1,930	1,295
PO_4	2,390	1,100	650	650	<250	<250	<250
SO_4	15,600	9,480	4,840	2,710	1,850	970	775
Other Results							
TC (a)	8,600	4,400	2,100	1,200	790	340	1,000
TIC (b)	8,470	4,010	2,020	1,160	730	310	950
TOC (c)	130	390	80	40	60	30	50

(a) measured by the furnace method
(b) measured by the hot persulfate method
(c) TOC is determined as TC minus TIC**.**

Notes:
1. Overall error greater than 10-times detection limit is estimated to be within ±15%.
2. Values in brackets [ ] are within 10-times detection limit with errors likely to exceed 15%.
3. ”--“ indicates measurement below detection.

The radioactive component concentrations are shown in Tables 3.8 and 3.9 for the slurry and permeate samples, respectively. The concentrations in the slurry are on a dry weight basis. Of the major radioactive isotopes, only ^{137}Cs and ^{99}Tc were significantly removed during the washing and leaching. As expected, the ^{90}Sr and TRU isotopes remained with the slurry. For the ^{241}Am alpha energy analysis (AEA) in the permeate samples, the concentration in the process blank (2.0 E-4 ⊠Ci/mL) was generally greater than that measured in the actual samples. However, in all cases (the process blank and the permeate samples), the concentrations measured were less than the specified liquid sample MRQ of 7.2 E-4 ⊠Ci/mL. As a result, these samples were not analyzed again.

Table 3.8. Radioactive Component Concentrations in the Slurry (Dry Weight Basis)

Analyte	**AZ-0 (17.9 wt% sample)**	**AZ-2 (post-wash)**	**AZ-4 (post-leach)**	**AZ-6-(post-rinse) Average**
	µCi/g	**µCi/g**	**µCi/g**	**µCi/g**
^{3}H	0.079154357	0.112454	0.1388764	0.2235142
^{14}C	0.005723866	0.003736	0.0049308	0.0049253
^{90}Sr	15800	30400	23600	61000
^{60}Co (GEA)	2.06	3.75	3.26	8.43
^{137}Cs (GEA)	2130	807	534	641
^{125}Sb (GEA)	8.89	18.2	14.2	38.6
^{154}Eu (GEA)	24.5	42.9	36.4	101
^{155}Eu (GEA)	28.7	58.1	50.9	120
^{241}Am (GEA)	47.7	56.6	51.1	198
^{241}Am (AEA)	41.4	77.9	66	165
$^{243/244}$Cm (AEA)	<2E-1	0.192	0.149	0.298
$^{239/240}$Pu (AEA)	2.44	4.13	3.35	9.58
^{99}Tc (ICP-MS)	0.509	0.153	0.149	0.0429
^{129}I (ICP-MS)	1.48 E-5	1.87 E-5	<1.04 E-5	<1.28 E-5
^{126}Sn (ICP-MS)	0.078 µg/g	0.144 µg/g	0.0946 µg/g	0.21 µg/g
^{237}Np (ICP-MS)	0.0278	0.0499	0.0438	0.135
^{233}U (ICP-MS)	1.21 E-3	4.97 E-3	4.11 E-3	4.53 E-3
^{234}U (ICP-MS)	1.54 E-3	5.95 E-3	5.08 E-3	5.38 E-3
^{235}U (ICP-MS)	6.27 E-5	2.44 E-4	2.05 E-4	2.25 E-4
^{236}U (ICP-MS)	1.35E-4	5.32 E-4	4.47 E-4	4.99 E-4
^{238}U (ICP-MS)	1.11 E-3	4.44 E-3	3.66 E-3	4.02 E-3
^{239}Pu (ICP-MS)	1.63	3.05	2.56	7.97
^{240}Pu (ICP-MS)	0.456	0.798	0.703	2.24
^{242}Pu (ICP-MS)	0.144 µg/g	0.110 µg/g	0.0263 µg/g	0.0537 µg/g

GEA: gamma energy analyses.
AEA: alpha energy.
ICP-MS: inductively coupled plasma-mass spectrometry.

Table 3.9. Radioactive Component Concentrations in the Permeate Samples

	Original Supernatant (AZ-A)	Wash 1 Permeate (AZ-C)	Wash 2 Permeate (AZ-E)	Leach Permeate (AZ-G)	Rinse 1 Permeate (AZ-I)	Rinse 2 Permeate (AZ-K)	Rinse 3 Permeate (AZ-M)
	(μCi/mL)	(μCi/mL)	(μCi/mL)	(μCi/mL)	(μCi/mL)	(μCi/mL)	(μCi/mL)
^{137}Cs (GEA)	1.38E+03	5.75E+02	3.12E+02	1.50E+02	8.84E+01	4.42E+01	2.08E+01
^{154}Eu (GEA)	<3E-2	<2E-2	<5E-3	<2E-2	<2E-3	<3E-3	<2E-3
^{155}Eu (GEA)	<8E-1	<5E-1	<8E-2	<3E-1	<5E-2	<7E-2	<2E-2
^{241}Am (GEA)	<3E0	<2E0	<8E-2	<9E-1	<7E-2	<9E-2	<2E-2
^{241}Am (AEA)	2.68E-05	1.74E-05	1.22E-05	1.89E-05	9.72E-05	3.50E-05	6.34E-04
^{99}Tc (ICP-MS)	4.10E-01	1.71E-01	7.32E-02	4.27E-02	1.90E-02	9.28E-03	4.82E-03
^{99}Tc (pertechnetate)	3.86E-01	1.62E-01	7.77E-02	3.90E-02	1.80E-02	8.84E-03	5.58E-03
^{90}Sr	1.19E+00	7.10E-01	2.73E-01	1.42E+00	9.56E-01	4.09E-01	3.65E-01

GEA: gamma energy analyses.
AEA: alpha energy.
ICP-MS: inductively coupled plasma-mass spectrometry.

The removal efficiencies both for the initial sludge washing and combined washing and caustic leaching of the nonradioactive components are shown in Table 3.10. For columns 2 and 3, the results are based on the measured amount removed in the permeate compared with the amount in the original slurry in all but one case. The exception is sodium where the mass balance is based on the amount remaining in the slurry after each processing step compared with the amount in the original slurry. This was necessary for sodium because the sodium added in the form of caustic in the wash, leach, and rinse solutions made it difficult to close the mass balance. The results for columns 4 and 5 are based on the measured amount remaining in the slurry. Comparing columns 3 and 4 provides a range of the amount removed from the wash, leach, and rinse, based on what was measured as removed (column 3) and what was measured as remaining (column 4).

For the two washing steps, 1 liter of inhibited water (0.01 M NaOH) solution was added to the slurry and then approximately an equivalent amount of permeate was removed through the filter. Results indicate that 85% of the sodium was removed from the slurry during the water washing steps. The majority of the soluble anions (fluoride, nitrite, nitrate, sulfate, and oxalate) were removed during the first two water washes. Other components with significant removal efficiencies during the water wash were chromium with 56% removal and phosphorus with ~40% removal.

The equivalent of 1.5 liters of 5 M NaOH was added for the caustic leach, resulting in an estimated 2.8 M NaOH concentration. The three subsequent rinses were each performed with 1.2 liters of 0.01 M NaOH, resulting in an estimated 1.45, 0.65, and 0.26 M NaOH solution, respectively. The overall amount removed in the washes, leach, and rinses is also shown in Table 3.10. While only 9% of the aluminum was removed during the washing, a total of 70% was removed by washing and caustic leaching. Caustic leaching also significantly improved the amount of chromium and phosphorus removed.

The residue column indicates how much of the initial mass of each component remained in the final slurry after sludge washing, caustic leaching, and rinsing. The final column is the mass recovery, which indicates how well the mass balance closed (i.e., how much of each component was accounted for).

Table 3.10. Selected Component Removal Efficiencies

Analyte	Removed in Wash (%)	Total Removed in Wash, Leach, and Rinse (%)	Total Removed in Wash, Leach, and Rinse (%) (Based on Slurry Residue)	Residue (%)	Recovery (%)
Al	9	70	75	25	99
B	23	67	98	2	72
Ba	0	0	6	94	107
Cd	0	0	8	92	104
Cr	56	93	64	36	132
Fe	0	0	8	92	104
La	0	0	11	89	101
Mn	0	0	11	89	101
Na	85[(a)]	91[(a)]	91	9	110
Nd	0	0	13	87	99
Ni	0	0	15	85	97
P	42	60	40	60	128
Si	14	40	35	65	117
Sr	0	0	9	91	103
Zr	0	0	<0	108	121
^{137}Cs	>100	>100	93	7	137
^{154}Eu	<0.22	<0.60	2	98	110
^{155}Eu	<4.6	<8.3	0	100	120
^{241}Am (AEA)	0	0	5	95	107
^{90}Sr	0	0	8	92	104
C_2O_4	>100	>100	97	3	183
F	>100	>100	98	2	159
NO_2	>100	>100	99	1	140
NO_3	>100	>100	100	0	134
SO_4	66	93	99	1	94

(a) Because of the significant sodium added during the leaching, Na numbers are calculated based on what was remaining in the sludge rather than what was removed.

In all cases the recovery is greater than the sum of the "total removed in the water wash and caustic leach" and the "fraction is solids residue," because the recovery takes sampling into account. For example, in the case of phosphorus, 60% of the starting mass was removed in the wash, leach and rinse. The amount measured in the final residue, was also 60% of the initial mass, for a total of 120%. The indicated 128% recovery is higher than 120%, because it also takes into account phosphorus mass removed during sampling. The recovery deviates from 100% because of the variability in the analysis, which, for ICP-AES, is estimated to be ±15%. Without replicate samples, it is not possible to isolate which samples may have contributed to the error.

Overall, the recoveries were very good. The recovery can be represented as:

$$\text{Recovery} = \frac{(\text{Analyte}_{\text{wash}} + \text{Analyte}_{\text{leach}} + \text{Analyte}_{\text{rinse}} + \text{Analyte}_{\text{sampling}} + \text{Analyte}_{\text{residue}}}{\text{Analyte}_{\text{initial.sludge}}} \tag{3.4}$$

The insoluble radioactive component concentrations provide a means of measuring the capability of the filter to separate the undissolved solids from the liquids. The isotope ^{241}Am is basically insoluble in caustic solutions, and its concentration was measured for all permeates and slurries; consequently, it was used to measure filter removal efficiency in terms of a decontamination factor (DF)(a) for each step of the process. The ^{241}Am water wash DFs were approximately 803,000 for the original slurry; the water wash DF was 2,160,000; the caustic leach DF was 935,000; and the final rinse DF was 40,300. These DFs are likely biased low, due to the problem with the process blank showing a greater concentration of ^{241}Am than the permeate samples (mentioned above). Nevertheless, these high DFs indicate good solid/liquid separations using the Mott 0.1-µm sintered metal filter.

(a) DF = concentration in the slurry/concentration in permeate.

4.0 Physical Properties, Rheology, Particle Size Distribution, and Energetics

This section describes physical, rheological, particle size, and energetics analyses conducted on samples of Tank AZ-101 slurry. These sub-samples were taken via pipette directly from the CUF slurry reservoir. Samples for physical property measurement were taken four times during the course of testing: 1) immediately after the tests with the high solids matrix, 2) after the second wash, 3) after the leach, and 4) after the final rinse. Rheological measurements were taken twice: 1) immediately after the high solids matrix and 2) after the final rinse. The rheology samples were immediately characterized by shear stress versus shear rate. Following rheological measurements, the material was returned to the CUF for continued testing. Particle size distribution measurements were taken three times: 1) before testing in the CUF, 2) immediately after the high solids matrix, and 3) after the final rinse. A sample for energetics analysis was taken after the final rinse.

4.1 Physical Properties Analysis

The Tank AZ-101 physical property samples were analyzed for density of the bulk slurries, centrifuged solids, and centrifuged supernatant. The density results are listed in Table 4.1. The weight percent and volume percent settled solids (on a wet basis), wt% and vol% centrifuged solids (on a wet basis), and wt% total solids (on a dry basis) were also measured for these samples (Table 4.2).

Table 4.1. Density Measurements for Samples of Tank AZ-101 Slurry

Slurry Sample	Bulk Density, g/mL			
	Sample size (g)	**Slurry**	**Centrifuged Solids**	**Supernatant**
AZ-1A, Taken after high solids matrix	8.828	1.338	1.613	1.189
AZ-3, Taken after wash 2	5.457	1.240	1.516	1.033
AZ-5, Taken after leach	9.690	1.211	1.561	1.121
AZ-7A, Taken after rinse 3	8.790	1.127	1.481	1.003

Table 4.2. Weight Percent and Volume Percent Solids Measurements for Samples of Tank AZ-101 Slurry

Slurry Sample	Vol% Wet Centrifuged Solids	Wt% Wet Centrifuged Solids	Wt% Total Solids	Wt% Undissolved Solids	Wt% Dissolved Solids in Supernatant
AZ-1A, Taken after high solids matrix	36.4	43.9	38.8	17.9	26.2
AZ-3, Taken after wash 2	43.2	52.8	27.3	27.0	1.35
AZ-5, Taken after leach	20.0	25.8	22.1	8.8	14.8
AZ-7A, Taken after rinse 3	20.5	27.0	13.7	10.9	3.43

The density of the centrifuged supernatant for the concentrated slurry was 1.189 g/mL. As would be expected, this value decreased to 1.033 following the two inhibited water rinses. The supernatant density increased to 1.121 g/mL following the caustic leach (~3 M NaOH). After the final inhibited water rinse, the supernatant density decreased to 1.003 g/mL.

A known mass of each slurry sample, M_{CT}, was placed in volume-graduated centrifuge cones. The samples were then centrifuged at approximately 1000 times the force of gravity for 1 hour. The total volume (V_{CT}) and volume of centrifuged solids (V_{CS}) were recorded. The vol% centrifuged solids was calculated ($V_{CS/}/V_{CT}$ x 100%). The centrifuged supernatant was then decanted into a graduated cylinder; its mass (M_{CL}) and volume were (V_{CL}) recorded; and the density was calculated ($D_{CL}=M_{CL}/V_{CL}$). The mass (M_{CS}) and volume (V_{CS}) of the centrifuged solids were then recorded, and the density was calculated ($D_{CS}=M_{CS}/V_{CS}$). The wt% centrifuged solids ($Wt\%_{CS}=M_{CS}/M_{CT}$ x 100%) and vol% centrifuged solids ($Vol\%_{CS}=V_{CS}/V_{CT}$ x 100%) were also calculated.

The centrifuged solids and supernatants were then dried at 50°C for 3 days, followed by 105°C for 10 more days. The mass of the dried centrifuged supernatant (M_{dcl}) and dried centrifuged solids (M_{DCS}) were then measured. Assuming that all mass lost during the drying process was water and not other volatile component, the wt% total solids in the bulk slurry was calculated (Wt% total solids = $[M_{DCS}+M_{DCL}]/M_{CT}$ x 100%).

The bulk density of the slurry and the density of the centrifuged solids both decreased over the course of the run. The latter result suggests that the washing, leaching, rinsing, and pumping had an effect on the packing characteristics of this sludge.

An additional calculation was performed to determine the wt% undissolved solids in the samples, excluding all interstitial liquid, which can also be viewed as the solids left if all the supernatant could be removed from the bulk slurry. The equation used for this calculation was:

$$\text{Wt\% undissolved solids} = \left(1 - \frac{1 - \dfrac{M_{dsc}}{M_{cs}}}{1 - \dfrac{M_{dcl}}{M_{cl}}} \right) \text{x} \frac{M_{cs}}{M_B} \text{x } 100\% \tag{4.1}$$

This calculation assumes 1) that the supernatant and the interstitial liquid have the same composition, and 2) that all mass loss during the drying of the centrifuged solids was water loss from interstitial liquid.

The physical property data trended as expected. The supernatant density and wt% solids (total, dissolved, and undissolved) were used in the mass balance for the CUF testing with excellent results, as the calculated and measured values matched well.

4.2 Rheological and Flow Properties

The rheological properties of the Tank AZ-101 slurries were determined with a Haake viscometer. Both samples exhibited yield pseudoplastic behavior and best fit the Casson model. This rheological behavior was expected, as yield pseuodplastic behavior was also observed for slurries from Tank AZ-102

(Brooks et al. 2000b). The concentrated 17.9 wt% undissolved solids material had an apparent viscosity of approximately 15 cP @ 600 s^{-1}. The viscosity of the 10.9 wt% washed and leached solids had an apparent viscosity of approximately 5 cP @ 600 s^{-1}.

This section covers tests performed with the Haake M5 measurement head in the A cell of the HLRF to determine the rheology of Tank AZ-101 waste. The primary tests were standard shear stress vs. shear rate curves.

4.2.1 Equipment Capabilities and Sensor Selection

Measurements were performed using a Haake M5 rheometer remoted for hot cell operations. For this work, the M5 was equipped with an MVI sensor. The fluid is loaded into the sensor cup, and the sensor inner spindle is turned within the fluid. The resulting fluid resistance to the flow causes a small movement in a torsion bar mounted between the motor and the drive shaft that is measured by an electronic transducer. This measurement head and sensor combination has the capacity of an optimal effective viscosity range of 10 to 10^5 cP, and can measure over a shear rate range of 0 to 1150 1/s. A 48.4 cP standard oil was used to validate the calibration of the machine. Refer to Appendix G for more background information.

4.2.2 Test Method

Two separate concentrations of the Tank AZ-101 slurry were tested during the CUF run. The first was the 17.9 wt% solids slurry and the other was the final washed solids. Because of material and time limitations, only one temperature was tested, 25°C.

For the test, the samples were loaded and then ramped up from 0 to 1000 1/s in 5 minutes, held at 1000 1/s for 5 minutes, and then ramped from 1000 to 0 1/s in 5 minutes. This ramp cycle was operated at least once for each sample. Therefore, each sample was tested through a minimum of two complete ramp cycles from 0 to 1000 1/s over a total time of 15 minutes. If the second run data closely overlaid the first run data, the testing for that sample was considered complete. If there was a noticeable variation in the data, the sample was ramped through this cycle again until two consecutive similar data sets were obtained. This repetition determines if rheological changes are made to the material while under the influence of shear. Shear history is often an important part of determining expected rheological behaviors. Once the previous sample was tested to the point of obtaining consistent data, it was removed and a new sample loaded for the next run parameter.

The purpose of this set of testing parameters was to identify the rheological behavior and shear sensitivity of the materials. The first ramp cycle shows newly loaded or fresh sample behavior, including breakdown of sample structure through hysteresis, if present. Hysteresis is when the ramp-down curve is different from the ramp-up curve. An immediate repeat allows little or no time for the sample to recover. The complete cycle repeat with the used sample shows the effects of a shear history with a short time of recovery for the sample.

4.2.3 Run Results

Each rheological test of the 17.9 wt% Tank AZ-101 slurry material yielded virtually identical results. A typical rheogram from this slurry is shown in Figure 4.1. All four runs of the 17.9 wt% slurry yielded classic pseudoplastic curves that best fit a Casson model, though they have high correlations to the Bingham plastic model as well. The Bingham plastic model is closely related to the Casson model. Both viscosity model equations are provided in Appendix G. Table 4.3 displays the relevant parameters fitting the Casson and Bingham model for each run.

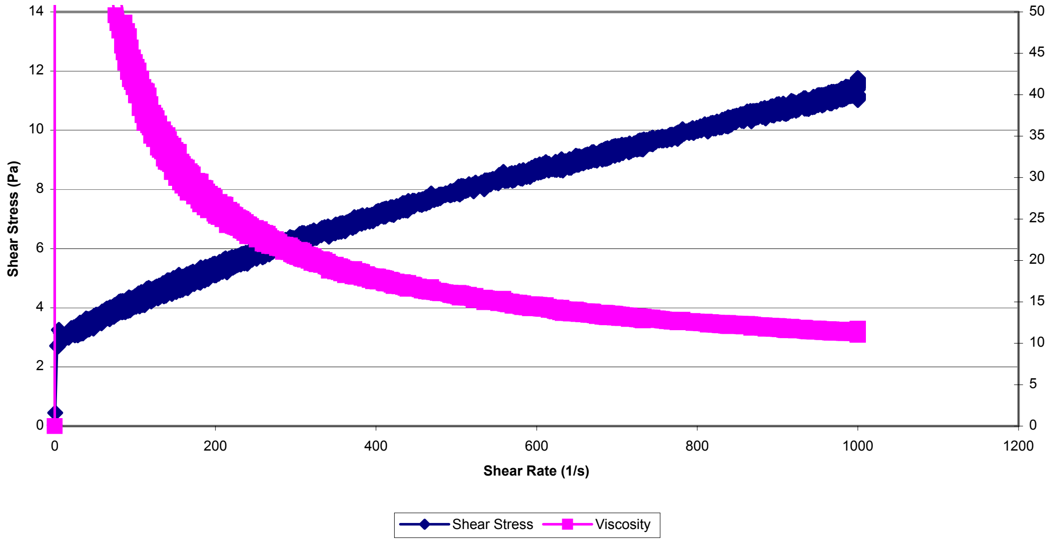

Figure 4.1. Rheogram of Untreated AZ-101 17.9 wt% Slurry (Sample AZ-1C)

Table 4.3. Model Fit Parameters for the 17.9 wt% AZ-101 Slurry (Sample AZ-1C)

	Casson			**Bingham**		
Sample	τ_o^B	η_p	R^2	τ_o^B	η_p	R^2
Run 1-1	1.982	0.0038	0.99	3.460	0.0082	0.98
Run 1-2	2.316	0.0035	1.00	3.817	0.0080	0.99
Run 2-1	2.406	0.0035	1.00	3.936	0.0083	0.99
Run 2-2	2.150	0.0040	0.99	3.736	0.0086	0.98

As can be seen in Figure 4.1, a small yield stress of 2 to 4 Pa is evident. This yield stress is very low and close to that seen when running water, and is probably due to the system's mechanical "start-up" resistance that can cause a false yield stress in low-viscosity fluids. The start-up resistance can be attributed to the energy input required to overcome the inertial forces of the sensor itself. It is likely that this at least partially contributed to the magnitude of the "yield stress" seen in these samples. The viscosity of the untreated 17.9 wt% slurry had an apparent viscosity of 15 cP at 600 s^{-1}.

Rheograms of the caustic leached and washed solids (from sample AZ-7C) were not as consistent as the concentrated matrix pre-wash sample. Several runs of each sample were needed to obtain repeatable results, and all the runs had spiked scatter zones. These are zones where large unrepeatable shear stress data spikes occur. Spiked scatter zones often occur in the initial runs of high solids slurries, but diminish or disappear during repeat analysis as chunks and agglomerations of the material are broken down. Such was not the case with these samples. Thus, we conclude that the spiked scatter zones were not caused by soft agglomerations.

An example of a rheogram from this sample is shown in Figure 4.2. There is a small yield stress of approximately 1 Pa, although it is believed that at least part of the yield stress is due to the instrument, as discussed above. In some of the runs at higher shear rates, it is suspected that Taylor vortices developed, as can be seen in Figure 4.2 at a shear rate of approximately 750 to 800 s^{-1}. Taylor vortices are a flow pattern interference that can develop in a cup-and-bob system above certain shear rates. They are the result of a secondary flow that occurs as the inner cylinder of the concentric-cylinder instrument rotates when analyzing a material at too high a shear rate. Typically, there is a clear demarcation of where Taylor vortices begin, but in this case the vortices developed over a range between 750 and 800 s^{-1}. Thus the data taken above ~750 s^{-1} are not expected to be accurate.

The viscosity of the caustic leached and washed slurry (10.9 wt% undissolved solids) was approximately 5 cP at 600 s^{-1}. However, because of the scatter in the viscosity data, model fits should be used rather than any individual data point. Hysteresis was seen in several of the runs, but again not in reproducible patterns. All of the runs were yield pseudoplastic in nature and fit Casson or Bingham plastic models best, with parameters shown in Table 4.4.

The measured viscosity of the sludge washed and leached sample (AZ-7C) was expected to be less than untreated Tank AZ-101 sample (AZ-1C), because AZ-7C had both a lower solids concentration and a less viscous permeate. The viscosities measured for both samples are within the range expected by the waste treatment plant and appear to be reasonable for processing.

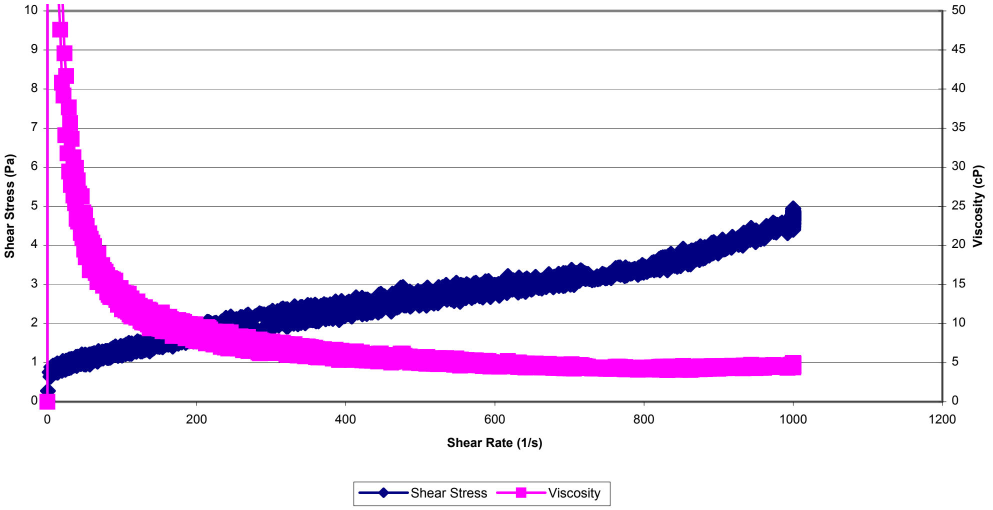

Figure 4.2. Rheogram of the Sludge Washed and Caustic Leached 10.9 wt% AZ-101 Slurry (Sample AZ-7C)

Table 4.4. Model Fit Parameters for the Caustic Leached and Washed AZ-101 Slurry (10.9 wt% Undissolved Solids from Sample AZ-7C)

Sample	Casson			Bingham		
	τ_o^B	η_p	R^2	τ_o^B	η_p	R^2
Run 1-2	0.658	0.0015	0.98	1.17	0.0031	0.97
Run 1-3	0.4636	0.0015	0.98	0.8705	0.0029	0.98
Run 1-4	0.3217	0.0017	0.97	0.6393	0.0031	0.97
Run 2-1	0.8730	0.0012	0.98	1.404	0.0029	0.98
Run 2-2	1.102	0.0026	0.84	1.102	0.00262	0.84
Run 3-1	0.6236	0.0016	0.98	1.123	0.0032	0.98

4.3 Particle Size Distribution

The PSDs of slurry samples from Tank AZ-101 are described and labeled as AZ-AR,(a) AZ-1B, and AZ-7B. The AZ-AR sample represents the as-received tank waste sample. The AZ-1B sample is as-received Tank AZ-101 material that was concentrated via crossflow filtration to approximately twice the wt% undissolved solids of the AZ-AR material. The AZ-1B sample was taken after 44 hours of testing in the CUF. The AZ-7B sample is crossflow-filtered material (i.e., AZ-1B) after wash/leach steps have been performed while in the filter. A Microtrac X-100 particle analyzer and an ultrafine particle analyzer (UPA) were used to measure the PSD of the tank samples.

4.3.1 Operating Conditions

The PSD of the samples was measured in the Microtrac X-100 at a flow rate of 40 mL/s. The flow rate was then increased to 60 mL/s, and the PSD was measured. The samples were then sonicated with 40W ultrasonic waves for 90 seconds at a flow rate of 60 mL/s, and the PSD was again measured. The sample was then sonicated a second time with 40W ultrasonic waves for 90 seconds at a flow rate of 60 mL/s, and the particle size was measured. The different flow rates and ultrasonic energy inputs were used to determine the shear sensitivity of the slurry. The purpose of the shear variations was not to compare to the shear experienced in the CUF but to investigate whether flocculation/de-agglomeration was occurring. Analyses were performed in triplicate on each sample under all flow/sonication conditions. The averages of these triplicate measurements are provided in Section 4.3.4.

No sonication or flow options were available for the UPA. Therefore, the sample was placed in the instrument, and the measurements were performed on the as-received, stationary material.

4.3.2 Suspending Medium

The suspending medium for the AZ-AR and AZ-1B analyses was a surrogate supernatant based on the analytical laboratory data obtained for the Tank AZ-101 supernatant liquid (Table 4.5). A 0.01 M NaOH solution was used as a suspending medium for the AZ-7B sample.

(a) AZ-AR is a sample of the "as-received" slurry from Tank AZ-101. The source bottle of this sample was AZ-101-PCB-2. The sample pedigree is described in Urie et al. (2002).

Table 4.5. Surrogate Supernatant Composition

Component	Concentration (M)
$NaNO_3$	3.38E-01
NaOH	1.46E+00
$Al(NO_3)_3 \cdot 9H_2O$	1.97E-01
Na_2SO_4	1.67E-01
$Na_2HPO_4 \cdot 7H_2O$	1.71E-02
NaCl	6.34E-03
$NaNO_2$	1.46E+00
$NaCO_3$	6.80E-01
$Na_2C_2O_4$	1.11E-02
NaF	1.05E-01

4.3.3 Calibration Checks

Instrument performance was checked against a range of National Institute of Science and Technology (NIST) traceable standards from Duke Scientific Corporation. These standards are polystyrene microspheres dispersed in a 1-mM KCl solution, and were run before the sample was analyzed. The number basis mean results were within 10% of the NIST traceable values.

4.3.4 Results

The PSDs from the Microtrac X-100 measurements are presented graphically in Figures 4.3 through 4.5. This set of figures compares the PSD of the as-received slurry (AZ-AR), the concentrated CUF slurry (AZ-1B), and the washed/leached CUF slurry (AZ-7B) at different rates of shear. The differential and cumulative volume distributions for the X-100 are given, with differential and cumulative area and population distributions provided in Appendix H.

On a volume basis (see Figures 4.3 and 4.4), the as-received material (AZ-AR) appears to consist of larger particles (i.e., 3- to 30-µm range) than AZ-1B and AZ-7B. As the shear increases on AZ-AR, no significant PSD changes are observed. The CUF concentrated material (AZ-1B) has a significant volume of smaller particles in the 0.2- to 2.0-µm range as compared with AZ-AR, most likely due to the high shear forces in the CUF. As the shear rate in the X-100 increases (specifically after sonication), particles in the 2- to 3-µm range appear to de-agglomerate into particles in the 0.2- to 1-µm range, which indicates that flocculation is most likely occurring in the original sample. The washed/leached CUF material (AZ-7B) at low shear (40 mL/s) appears to have a similar shape to the AZ-1B material, although it is shifted to the right. As the shear increases, the peak value shifts to the left and corresponds well with AZ-1B, which indicates that flocculation/de-agglomeration is occurring. However, a significant volume

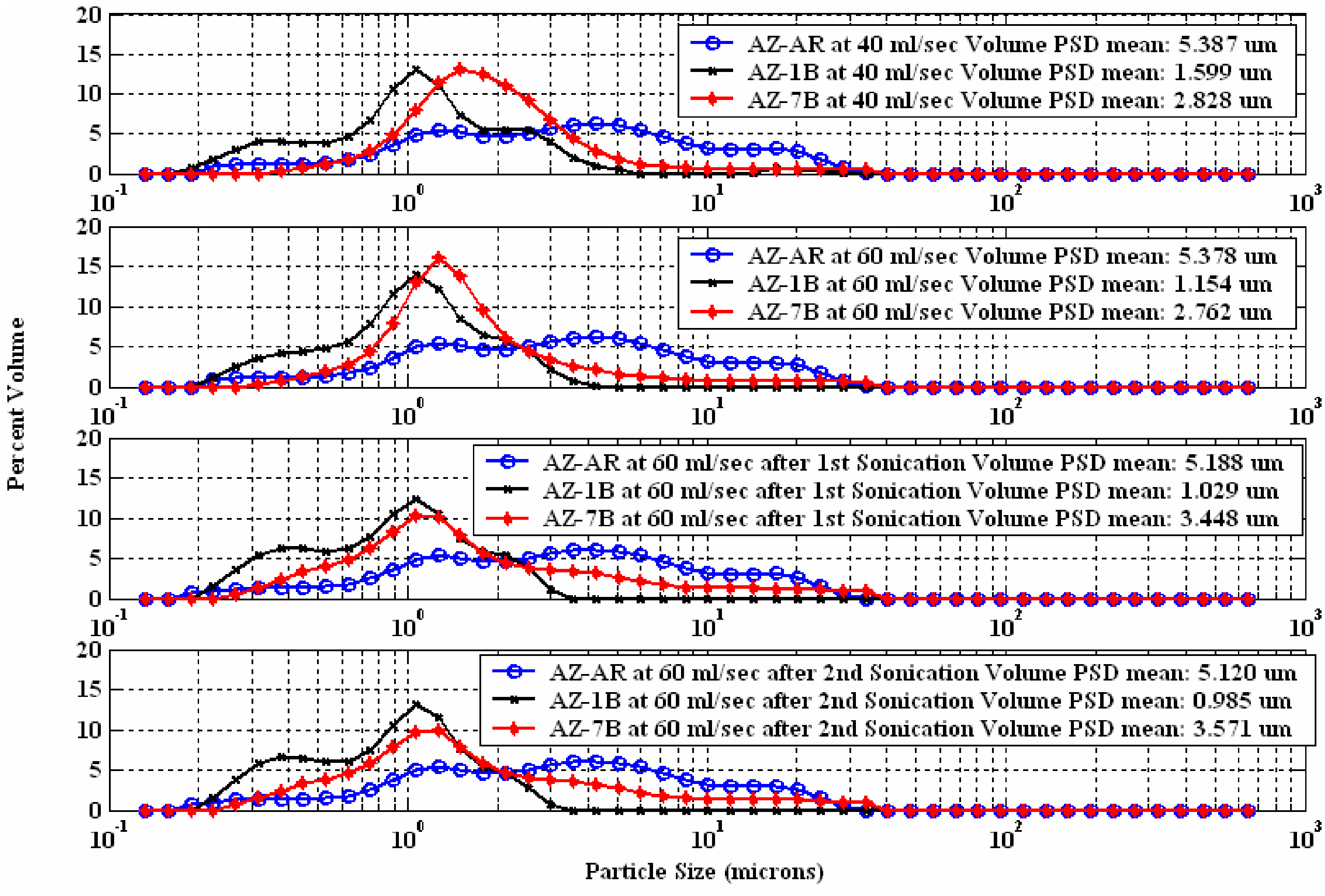

Figure 4.3. X-100 Differential Particle Size Distribution of AZ-AR, AZ-1B, and AZ-7B on a Volume Basis

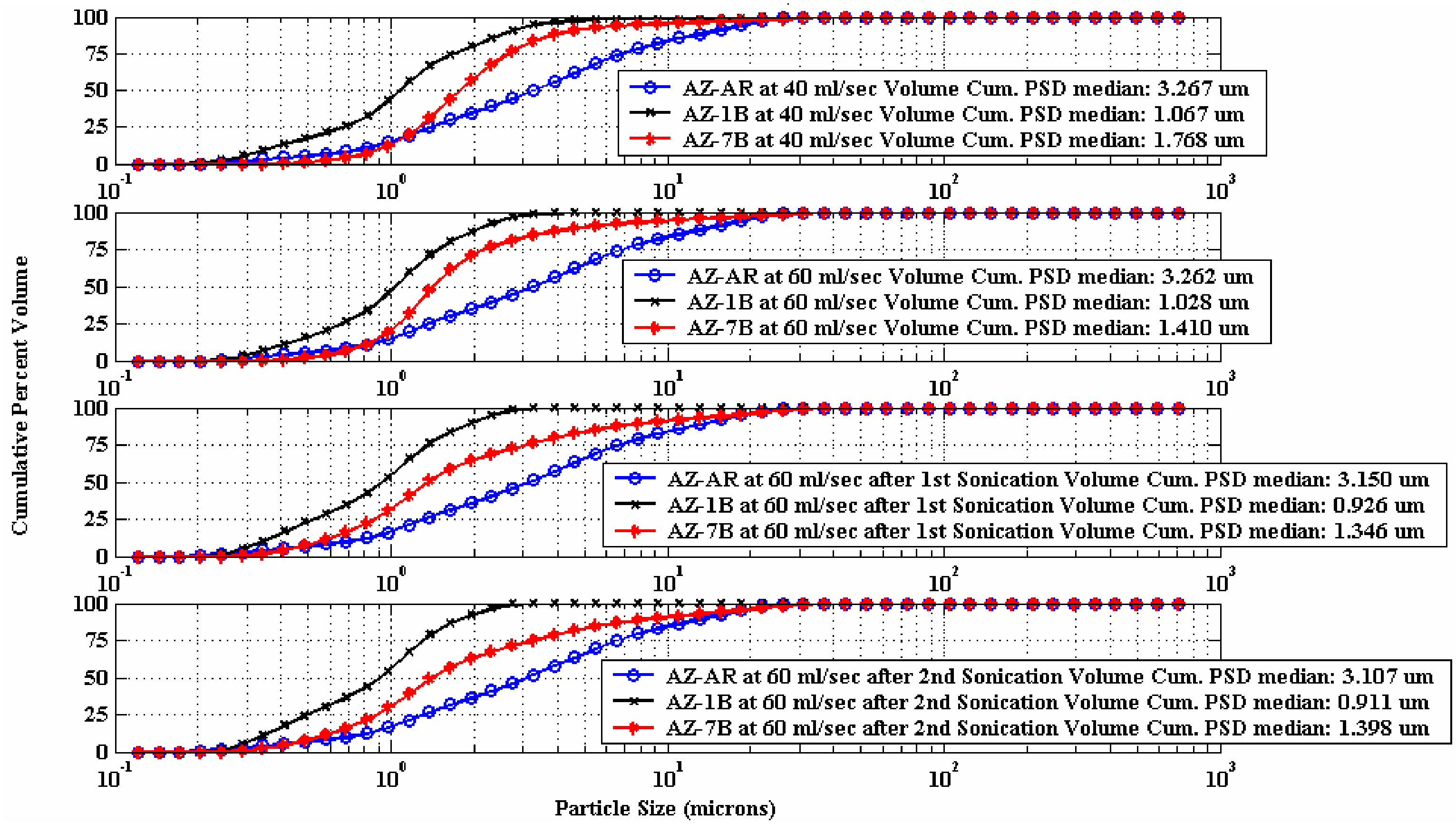

Figure 4.4. X-100 Cumulative Particle Size Distribution of AZ-AR, AZ-1B, and AZ-7B on a Volume Basis

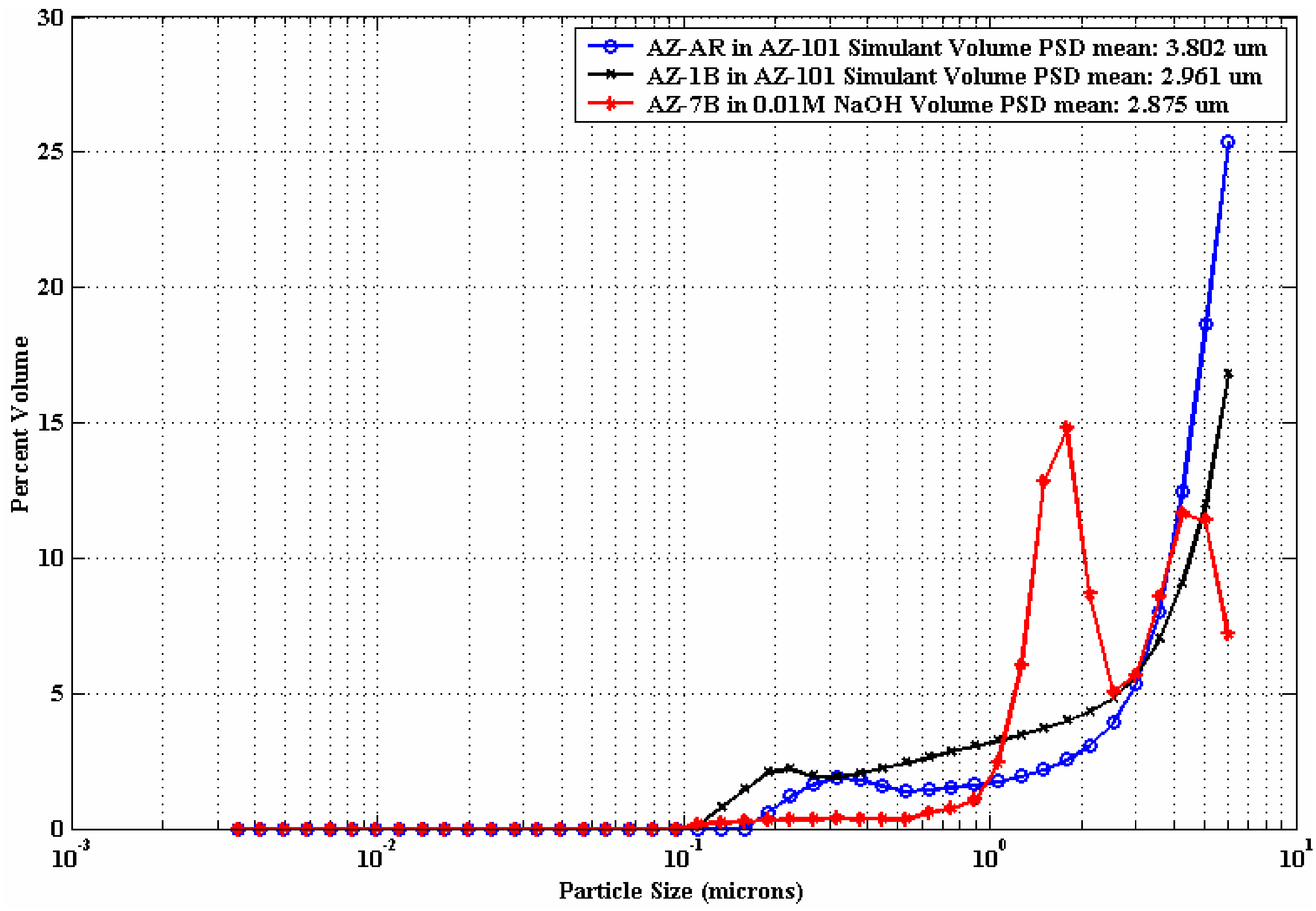

Figure 4.5. UPA Differential Particle Size Distribution of AZ-AR, AZ-1B, and AZ-7B on a Volume Basis

of larger particles in the 2- to 30-μm range is present after washing/leaching, which is most likely due to dissolution of smaller particles during the wash/leach steps, increasing the relative volume of larger particles.

The degree of flocculation observed in the AZ-7B sample appears significantly greater than the AZ-AR and AZ-1B samples. The AZ-7B sample is suspended in a 0.01 M NaOH solution, while the AZ-AR and AZ-1B samples are suspended in a tank supernatant slurry. Because of this difference, the zeta potential of each of these samples is likely to differ. If the zeta potential of the AZ-7B sample is closer to zero than the AZ-AR and AZ-1B samples, the degree of flocculation of the AZ-7B sample could be much higher than the AZ-AR and AZ-1B samples.

These samples were also analyzed in the UPA without flow or sonication, as shown in Figure 4.5. The particle size range that is common to both the X-100 and the UPA is 0.12 to 6.5 μm. The UPA data show no significant volume of particles outside this common range. Because no particles are observed below 0.1 μm with the UPA, the X-100 particle size distributions should be considered a complete representation of the PSD over a range of 0.003 to 704 μm.

4.4 Energetics of Tank AZ-101 Solids

A sample of the sludge washed and caustic leached slurry was analyzed for exothermic reactions by differential scanning calorimetry (DSC) to address safety concerns regarding potential reactivity of the waste stored in the waste treatment plant. A strategy introduced by Babad et al. (1995) to assess the reactivity hazards of stored organic-bearing HLW was used. In this strategy, waste energetics measured by DSC are used to identify potentially reactive wastes. If an exothermic reaction that produces >480 J/g dry waste is observed, then the waste requires further study.

4.4.1 Measurement Strategy

To provide data for assessing the thermal reactivity hazard of the washed Tank AZ-101 solids, the differential thermal analysis (DTA)-based approach (Bryan et al. 2002) was again used. In this strategy, the waste energetics and thermal behavior were measured between room temperature and 500°C using a simultaneous thermogravimetric analyzer (TGA)/DTA. In addition, the DTA enthalpy results were supplemented with similar DSC analyses. If the DTA observed an exothermic reaction releasing >480 J/g dry waste, we planned to measure the Tank AZ-101 waste energetics using the reactive system screening tool (RSST) after consulting with the Contractor. See Scheele et al. (1995), Wahl et al. (1996), and CCPS (1995) for descriptions of the thermoanalytical methods.

4.4.2 Experimental

The AZ-101 TGA/DTA analyses were performed in triplicate and the DSC analyses in duplicate. For analyses, the DTA and DSC were temperature- and heat-calibrated using melting point standards. To measure heat changes in the Tank AZ-101 sample, the DTA/TGA and the DSC were programmed to heat to 100°C at 5°C/min, hold at 100°C for 30 minutes to evaporate any free water, and then heat to 500°C at 5°C/min. Nitrogen or argon was used as the purge gas to eliminate oxygen and any of its reactions with organic compounds in the waste during the analysis. For calculating enthalpy changes, the vendor-supplied programs for the instruments were used.

4.4.3 Theoretical Heat of Reaction

The estimated maximum reaction enthalpies based on measured oxalate, succinate, and TOC[(a)] indicate potential heat production of less than 100 J/g dry sludge. Using Burger's (1995) estimated reaction enthalpies and assuming complete reaction between oxalate and nitrate or nitrite (without hydroxide participation in the reaction), the maximum enthalpy is -1.2 J/g dry sludge.[(b)] Likewise, succinate could produce a maximum of -1.2 J/g dry sludge.[(c)]

If we assume that the 0.013 g TOC/g dry sludge (measured by the furnace TOC method) is oxalate and that the oxalate reacts with the available nitrate and nitrite (there is insufficient oxidant to completely oxidize the available TOC as oxalate), the sludge could produce between -60 and -91 J/g dry sludge, depending on the degree of hydroxide participation. Using TOC values from the hot persulfate method yields about one-tenth the enthalpies. Based on these calculated estimates, the maximum possible enthalpy production is well below the 480 J/g dry waste criterion.

4.4.4 Results

The AZ-101 washed solids exhibited endothermic behavior as shown in Figure 4.6, which presents the average of the triplicate DTA, TGA, and DTG (see below) analyses for the AZ-101 washed solids between 100°C and 500°C. Figure 4.7 shows each of the triplicate DTA runs, and Figure 4.8 provides the average of the DSC analyses. Comparing the DTA and DSC results presented in Figures 4.6 and 4.8, respectively, shows the same thermal behavior. The curvature observed for the DTA curve is attributed to baseline drift, as the same drift was observed when analyzing an inert alumina.

The drying reaction is not presented in Figures 4.6 and 4.7 to facilitate analysis of the reactions. In addition to the TGA and DTA, Figure 4.6 provides the differential thermogravimetric analysis (DTG) curve, which is the derivative of the TGA and is another tool favored to help identify the reactions and the temperatures when reactions begin and end. As shown in Figure 4.6, the original samples contained an average of 98 wt% solids (2 wt% free water) based on the sample mass after heating at 100°C for 30 min. No exothermic reactions below 100°C were observed with either the DTA or DSC.

The TGA/DTG results indicate that two reactions occur after 100°C, while the DTA observed only one. In addition to the rapid mass loss observed between 200°C and 280°C, the TGA shows a continuous gradual mass loss between 100°C and 500°C. The DTA and DSC baseline drift and noise after the first reaction coupled with a very slow and low energy reaction likely explains the absence of a second observed reaction. The results of the TGA/DTA and DSC analyses are presented in Table 4.6; exothermic enthalpies are indicated by a negative sign and endothermic enthalpies are indicated by a positive sign.

(a) The tested material contained (on a dry basis) 1.3 wt% total organic carbon (TOC) as measured by the furnace method, 0.14 wt% TOC by the hot persulfate method, 500 ppm oxalate, and 100 ppm succinate. The total carbon from the measured organic species is about 170 ppm. Refer to Appendix F for a complete list of analytical results.

(b) If hydroxide participates in the reaction, oxalate's reactions with nitrate and nitrite could produce -1.7 J/g dry sludge.

(c) Burger did not provide reaction enthalpies for succinate with hydroxide and so are not estimated here.

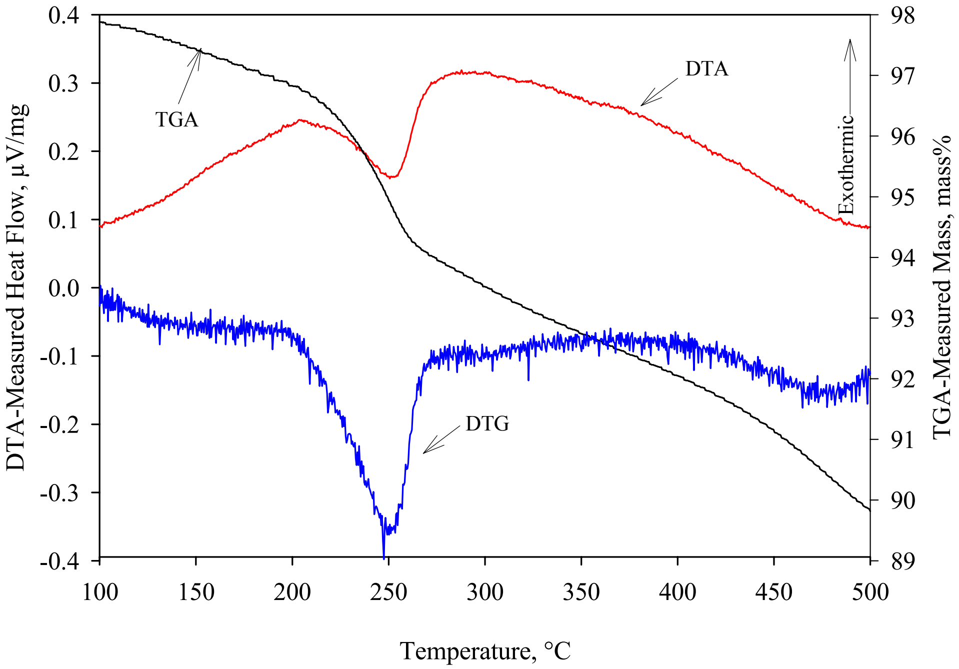

Figure 4.6. Average Thermal Behavior of AZ-101 Solids as Measured by DTA and TGA

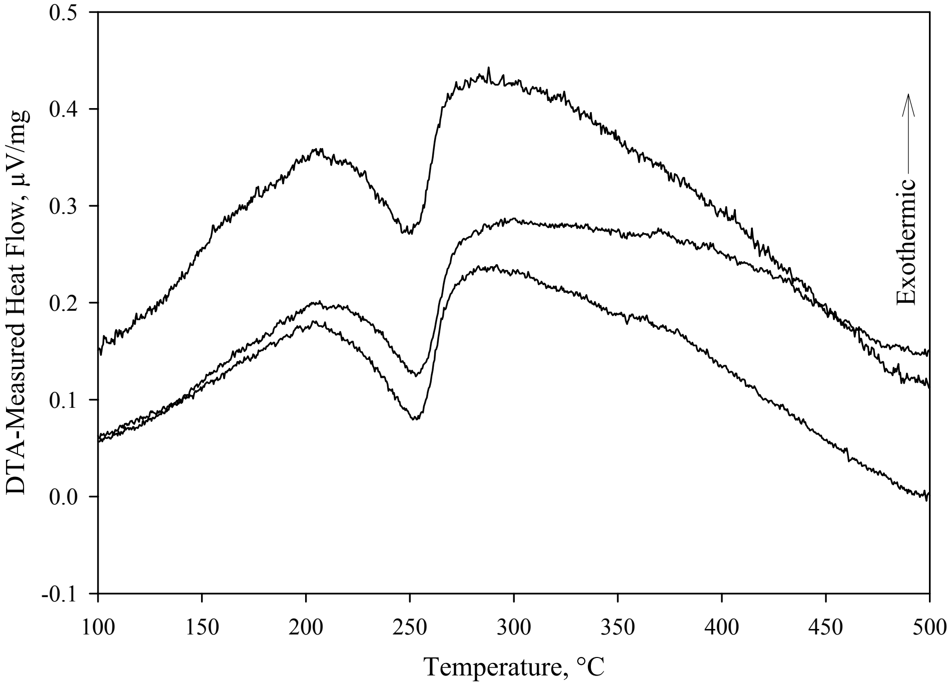

Figure 4.7. Thermal Behavior of Triplicate AZ-101 Solid Samples as Measured by DTA

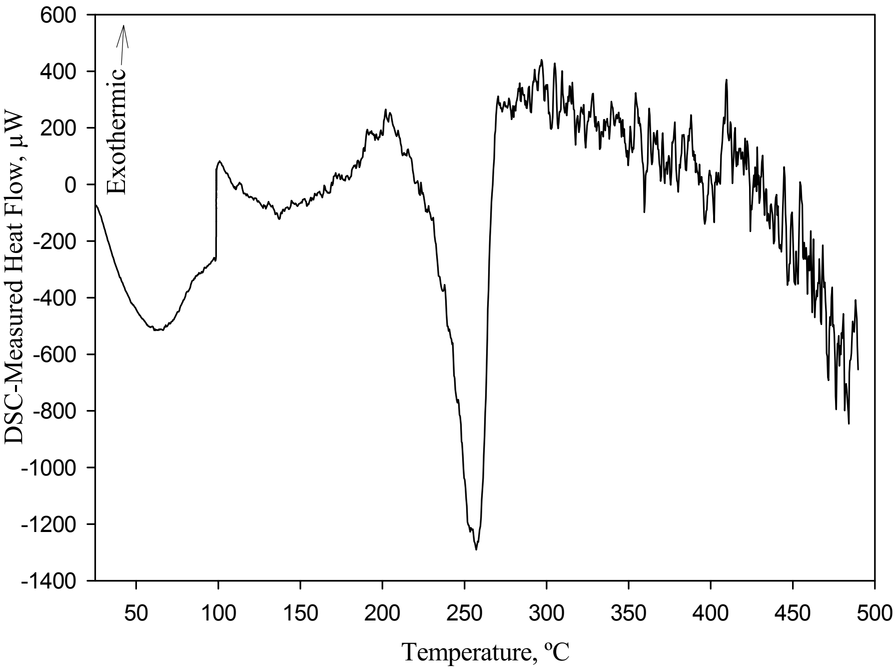

Figure 4.8. Average Thermal Behavior of AZ-101 Solids as Measured by DSC

Table 4.6. Thermal Behavior of AZ-101 as Measured by DTA/TGA and DSC

Sample	**Event Temperature Range (°C)**	**Mass Loss (wt%)**	**DTA-Measured Reaction Enthalpy (J/g)**	**DSC-Measured Reaction Enthalpy (J/g)**
Analysis #1	25 to 100	1.9	190[a]	54[a]
	200 to 280	3.0	35	55
	400 to 500	2.2	[b]	[b]
	Total (100°C to 500°C)	8.1	35	54
Analysis #2	25 to 100	2.2	49[a]	41[a]
	200 to 280	3.0	46	60
	400 to 500	2.3	[b]	[b]
	Total (100°C to 500°C)	8.0	46	60
Analysis #3	25 to 100	2.2	84[a]	[c]
	200 to 280	3.0	52	[c]
	400 to 500	2.2	[b]	[c]
	Total (100°C to 500°C)	8.1	51	[c]
Average	25 to 100	2.1	110[a]	48[a]
	200 to 280	3.0	47	68
	400 to 500	2.2	[b]	[b]
	Total (100°C to 500°C)	8.1	47	68

(a) Includes isothermal portion of analysis. DTA and DSC enthalpy measurements are dependent on heating rate with calibrations performed at a specific heat rate.

(b) A mass loss is observed indicating a reaction; however, no deviation in the DTA curve is observed to indicate true enthalpy change.

(c) DSC analysis performed only in duplicate.

The first observed reaction after 100°C occurs between 200°C and 280°C, based on the DTG and consumes +44 J/g waste with an average 3.0 wt% loss. The second DTG-indicated reaction occurs between 400°C and 500°C and causes a 2.2 wt% loss with no detectable enthalpy change. The average measured total enthalpy consumed between 100°C and 500°C is +47 J/g waste or +48 J/g dry waste.

These reactions are likely due to thermal decomposition of hydrates or hydrous oxides or carbonates, although most carbonates decompose at much higher temperatures. Analyzing the DTA/TGA off-gas by infrared spectroscopy or mass spectrometry would provide insights into the chemical nature of the observed reactions and would facilitate identification of significant reactions and help eliminate confounding baseline effects. It is recommended that an off-gas analysis system be added.

Figure 4.7 also shows that each replicate exhibits the same qualitative thermal behavior. Peak sizes are similar and occur at the same temperatures. The slopes in the baseline differ after 300°C likely due to differences in the heat capacities of the post-reaction residual material.

Because the Tank AZ-101 solids exhibited only endothermic behavior, the samples did not meet the Hanford threshold criterion (Babad et al. 1995) of a DSC-measured -480 J/g dry waste used to identify reactive wastes. The calculated maximum reaction enthalpies for measured oxalate, chelators, and TOC support the observed DTA- and DSC-measured reaction enthalpies. Based on the Babad et al. (1995) criterion of an enthalpy release of over -480 J/g dry waste for a waste to be designated as a potential reaction hazard and the absence of observable exothermic reactions, using the RSST to measure reaction enthalpies or thermal behavior is not recommended.

5.0 Conclusions

The results of the tests performed on sludge from Tank AZ-101 indicate that crossflow filtration provides excellent separation of solids and liquids. Washing and caustic leaching removed significant quantities of some analytes and radionuclides and reduced the mass of undissolved solids by approximately 56%. The analyses have also provided input to support waste treatment plant design and operation. In addition, the results verify the segregation of TRU from the liquids by means of filtration. No significant problems were encountered in the testing of this waste. The following conclusions reflect the testing and analyses performed on Tank AZ-101 sludge described in this document.

5.1 AZ-101 Crossflow Filtration

The Contractor design basis of 0.014 gpm/ft^2 can be met for a slurry at 7.6 wt% undissolved solids, as the average permeate flux ranged from 0.023 to 0.036 gpm/ft^2. Statistical modeling indicates that the dominant variable affecting the flux data for this solids loading is TMP. The permeate flux did not display a strong correlation with time, and changes in axial velocity also had little effect.

The Contractor design basis of 0.014 gpm/ft^2 can generally be met for a slurry of 17.9 wt% undissolved solids, as the average permeate flux ranged from 0.011 to 0.025 gpm/ft^2. Statistical modeling indicates that the dominant variable affecting the flux data for this solids loading is axial velocity. The permeate flux did not display a strong correlation with time, and changes in TMP also had little effect.

Ten-hour runs of the low and high solids slurries (without backpulsing) indicated very small permeate flux changes with time and that backpulsing provided only moderate benefit to flux for this waste.

Permeate flux of the as-received and washed slurries decayed linearly with the log of the undissolved solids concentration. The permeate flux of these slurries was also shown to be inversely proportional to viscosity. The permeate flux of the leached slurry did not exhibit significant decay over the solids concentration studied.

The AZ-101 solids did not significantly foul the filter membrane. The $SrCO_3$ slurry results reflect this result better than the CWF measurements. Consequently, it is recommended that $SrCO_3$ slurry tests be included in future testing, because the CWF measurement apparently measures the cleanliness of the CUF, not just the filter. The $SrCO_3$ slurry apparently acts as a filter aid by masking impurities in the CUF and providing flux measurements consistent with test trends observed.

The permeate decontamination factors for ^{241}Am (i.e., the ratio of concentrations in the slurry to the concentration in the permeate) were greater than 40,000 for the permeates collected, indicating excellent solid-liquid separations.

5.2 AZ-101 Wash and Caustic Leach Testing

Washing removed 85% of the sodium, 56% of the chromium, and approximately 40% of the phosphorus. Significant quantities of the soluble anions, including nitrate, nitrite, and sulfate were also removed. Of the major radioactive isotopes, only ^{137}Cs and ^{99}Tc were significantly removed during the leaching and washing.

Washing and caustic leaching together removed greater than 70% of the aluminum, 64% of the chromium, and 40% of the phosphorus. Caustic leaching also increased the removal of the water-soluble components, such as sulfate and sodium.

The primary metals in the initial slurry were, from highest to lowest concentration, sodium, aluminum, iron, and zirconium. After the sludge washing, caustic leaching, and rinsing, the concentrations of metals in the final slurry were iron, aluminum, zirconium, and sodium.

The total mass of undissolved solids was reduced by approximately 4% during the water washing and a total of 56% during the course of washing and leaching.

5.3 AZ-101 Rheological, Particle Size, and Energetic Properties

Rheology testing of the concentrated slurry and final slurry that had been sludge washed and caustic leached were found to have yield pseudoplastic behavior. The viscosities measured for both samples are within the range expected by the waste treatment plant and appear to be reasonable for processing.

There was a decrease in particle size from the initial as-received sludge to the concentrated slurry. The volume mean particle size under low flow conditions was 5.4 µm. After running in the CUF for approximately 38 hours, the volume mean particle size decreased to 1.6 µm. This decrease is attributed to particle attrition/de-agglomeration during pumping in the CUF. In contrast, the mean particle size increased to 2.8 µm after the sludge washing and caustic leaching treatment. This increase is attributed to some of the smaller particles dissolving during the pretreatment.

Energetics testing of the sludge washed and caustic leached slurry indicated no exotherms. Because Tank AZ-101 solids exhibited only endothermic behavior, the samples did not meet the Hanford threshold criterion of a DSC-measured -480 J/g dry waste used to identify reactive wastes.

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Appendix A

$SrCO_3$ Slurry Recipe

Appendix A

$SrCO_3$ Slurry Recipe

Use these components to prepare the $SrCO_3$ slurry for CUF filtration testing. This recipe will make a 1.5-liter batch.

Component	Concentration	FW	grams/1.5 L
NaOH	0.2	40	12
$NaNO_3$	1	84.99	127.49
$Na_2CO_3 \cdot 1\ H_2O$	0.5	124	93
$Sr(NO_3)_2$	0.35	211.63	111.11
Directions: Add 0.2 M NaOH, 1 M $NaNO_3$, and 0.5 M Na_2CO_3 to $Sr(NO_3)_2$, stir well and cook at 50°C for 4 hours, cool and let sit for 2 days. Then dilute to 2-liter mark. Now it is ready to test.			

Appendix B

Testing Mass Balance

Appendix B

This table provides the total mass and volume of slurry in CUF and the measured and calculated wt% insoluble solids during each process step.

Testing Mass Balance

Description	Total Mass, g	Total Volume, mL	Mass Added/Removed, g	Volume added/Removed, mL	**Measured** & Calculated wt% insoluble solids	Total Insoluble Solids in CUF, g
Start of test; Low solids matrix	4313	3488	0	0	7.6%	325.7
Permeate Sample AZ-A, AZ-B	4268	3450	-45	-38	7.6%	325.7
Dewater	1819	1391	-2448	-2059	17.9%	325.7
High Solids Matrix	1819	1391	0	0	17.9%	325.7
Slurry Samples AZ-0, AZ-1a, AZ-1b, AZ-1c	1776	1359	-44	-33	**17.9%**	317.9
Extended Run	1776	1359	0	0	17.9%	317.9
Added supernate/solids to CUF (estimate 165 mL supernatant)	2002	1540	226	181	16.6%	332.3
Further dewatering	1347	989	-656	-551	24.7%	332.3
Added Wash 1 water	2347	1989	1000	1000	14.2%	332.3
Permeate Samples AZ-C, AZ-D, AZ-Wash	2265	1913	-82	-75	14.7%	332.3
Wash 1	1262	995	-1003	-919	26.3%	332.3
Added Wash 2 water	2262	1995	1000	1000	14.7%	332.3
Permeate Sample AZ-E, AZ-F	2221	1956	-40	-39	15.0%	332.3
Removed Wash 2 water	1188	956	-1033	-1000	28.0%	332.3
Slurry samples AZ-2, AZ-3	1167	939	-20	-16	**27.0%**	314.6
Added Permeate back in because slurry too thick	1647	1403	480	464	19.1%	314.6
Lost ~20 mL of slurry	1622	1383	-25	-20	19.1%	309.9
Added 1000 mL of 3 M NaOH for Leach	2742	2383	1120	1000	11.3%	309.9
Added 500 mL of 9 M NaOH for Leach	3387	2883	645	500	9.1%	309.9
Leach at 85 C for 8 hours	3387	2883	0	0	4.1%	140.0
Water lost during leach	3362	2858	-25	-25	4.2%	140.0
Dewater after Leach (includes Permeate samples AZ-G, AZ-H, AZ-leach)	1599	1285	-1763	-1573	8.8%	140.0
Slurry samples AZ-4 and AZ-5	1569	1261	-30	-25	**8.8%**	137.4
Added Rinse Water for Rinse 1	2769	2461	1200	1200	5.0%	137.4
Removed Rinse 1 Water and Samples AZ-I, AZ-J, AZ-Rinse	1408	1211	-1361	-1250	9.8%	137.4
Added Rinse Water for Rinse 2	2608	2411	1200	1200	5.3%	137.4
Removed Rinse 2 water and samples AZ-K and AZ-L	1373	1176	-1235	-1235	10.0%	137.4
Added Rinse 3 water	2573	2376	1200	1200	5.3%	137.4
Removed Rinse 3 water and Samples AZ-m, AZ-N	1357	1160	-1216	-1216	10.1%	137.4
Removed AZ-6, AZ-7A, AZ-7B, AZ-7C, AZ-DSC	1215	1034	-142	-126	**10.9%**	132.0
Drained CUF	30	-18	-1185	-1052	10.9%	3.2
Added IW to rinse out CUF (3 stages)	1980	1932	1950	1950	0.2%	3.2
Drained CUF (3 stages)	56	8	-1924	-1924	NA	NA

Appendix C

Analytical Requirements

Appendix C

Analytical Requirements

Table C.1. Analytical Requirements for Solids and Liquids (Except Sample AZ-6)

Analyte	**Solids(a) Minimum Reportable Quantity**	**Liquid Minimum Reportable Quantity**	**Analysis Method**
	µCi/g	**µCi/mL**	
^{137}Cs	6.0E-02	9.0E+00	GEA
^{60}Co	1.2E-02	Not required	
^{125}Sb	6.0E+00	Not required	
^{154}Eu	6.0E-02	2.0E-03	
^{155}Eu	6.0E-02	9.0E-02	
^{241}Am	1.2E-03	7.2E-04	GEA, AEA
^{243}Cm, ^{244}Cm	6.0E-05	Not required	AEA
^{239}Pu	6.0E+00 µg/g	Not required	
^{126}Sn	6.0E-02	Not required	ICP-MS
^{129}I	3.0E+01 µg/g	Not required	
^{99}Tc	6.0E+00 µg/g	1.5E-03	
^{237}Np	1.8E+00 µg/g	Not required	
^{3}H	1.5E-02	Not required	Extraction/Beta Count
^{14}C	1.8E-03	Not required	Combustion Release/Beta Count
^{90}Sr	7.01E+01	1.5E-01	Separation/Beta Count
^{99}Tc	Not required	1.5E-03	Separation/Beta Count
Uranium isotopes	Not required(b)	Not required(b)	
Plutonium isotopes	Not required(b)	Not required(b)	
	µg/g	**µg/mL**	
Al	3.3E+02	7.5E+01	ICP-AES
Ag	9.0E+02	1.75E+01	
As	3.0E+00	Not required	
B	3.0E+00	Not required	
Ba	6.0E+02	7.8E+01	
Be	3.0E+00	Not required	ICP-AES
Ca	1.8E+02	1.5E+02	
Cd	1.1E+01	7.5E+00	
Ce	6.0E+00	Not required	
Co	3.0E+00	3.0E+01	
Cr	1.2E+02	1.5E+01	
Cs	3.0E-01	3.0E-01	
Cu	1.8E+01	1.7E+01	
Fe	1.4E+02	1.5E+02	

Table C.1. (contd)

Analyte	Slurry(a) Minimum Reportable Quantity	Liquid Minimum Reportable Quantity	Analysis Method
Hg	1.5E+00	Not required	Cold Vapor AA
K	1.5E+03	2.0E+2	ICP-AES
La	6.0E+01	3.5E+01	
Li	3.0E+01	Not required	
Mg	5.4E+02	1.5E+02	
Mn	3.0E+02	1.5E+02	
	µg/g	**µg/mL**	
Mo	6.0E+00	9.0E+01	ICP-AES
Nd	7.7E+01	Not required	
Na	1.5E+02	7.5E+01	
Ni	1.6E+02	3.0E+01	
Pb	6.0E+02	3.0E+02	
Pr	6.0E+00	Not required	ICP-MS
Pt	3.0E+00	Not required	
Rb	6.0E+00	Not required	
Sb	1.2E+01	Not required	ICP-AES
Se	3.0E+02	Not required	
Si	3.0E+03	1.7E+02	
Sr	3.0E+02	Not required	
Ta	6.0E+00	Not required	ICP-MS
Te	6.0E+00	Not required	ICP-AES
Th	6.0E+02	Not required	
Ti	1.5E+02	1.7E+01	
Tl	6.0E+02	Not required	
V	6.0E+02	Not required	
U	6.0E+02	6.0E+02	
W	6.0E+00	Not required	
Y	2.7E+02	Not required	
Zn	6.0E+00	1.65E+01	
Zr	6.0E+02	N/A	
TOC	6.0E+01	1.5E+03	TOC
TIC	3.0E+01	1.5E+02	TIC
Cl	2.3E+02	2.5E+1	IC
CO_3	3.0E+01	Not required	TIC
CN	3.0E+00	Not required	Total CN
NH_3	6.0E+01	1.0E+02	Ion Selective Electrode
F	7.5E+03	1.5E+02	IC
NO_3	4.5E+02	3.0E+03	
SO_4	1.2E+03	2.3E+03	
PO_4	6.0E+02	2.5E+03	

(a) Acid digestion and KOH fusion for slurry samples.

(b) Per Contractor assumption that the isotopic ratios are unchanged and that the pretreatment process does not affect isotopic ratios.

Table C.2. Analytical Requirements for Sample AZ-6

Analyte	Slurry Minimum Reportable Quantity(a) µg/g	Analysis Method/Driver
Al	3.3E+02	
Ba	6.0E+02	
Ca	1.8E+02	
Cd	1.1E+01	
Cr	1.2E+02	
Fe	1.4E+02	
K	1.5E+03	ICP-AES(b)
La	6.0E+01	(Pretreatment Specification)
Mg	5.4E+02	
Na	1.5E+02	
Ni	1.6E+02	
P	6.00E+02	
Pb	6.0E+02	
U	6.0E+02	
Ag	9.0E+02	
As	3.0E+00	
B	3.0E+00	
Be	3.0E+00	
Bi	--	
Ce	6.0E+00	
Co	3.0E+00	
Cu	1.8E+01	
Dy	--	
Eu	--	ICP-AES(b)
Li	3.0E+01	(Vitrification Request)
Mn	3.0E+02	
Mo	6.0E+00	
Nd	7.7E+01	
Sb	1.3E+01	
Se	3.0E_02	
Si	3.0E+03	
Sn	--	
Sr	3.0E+02	
Te	6.0E+00	
Th	6.0E+02	
Ti	1.5E+02	
T1	6.0E+02	

Table C.2. (contd)

Analyte	Slurry Minimum Reportable Quantity[(a)]	Analysis Method/Driver
V	6.0E+02	
W	6.0E+00	
Y	2.7E+02	
Zn	6.0E+00	
Zr	6.0E+02	
Cs	3.0E-01	
Rb	6.0E+00	ICP-MS (Pretreatment Specification)
Pd	--	
Pr	6.0E+00	ICP-MS (Vitrification Specification)
Pt	3.0E+00	
Rh	--	
Ru	--	
Ta	6.0E+00	
TOC	6.0E+01	Silver catalyze persulfate and furnace oxidation method
TIC	3.0E+01	Silver catalyze persulfate and furnace oxidation method
Cl	2.3E+02	
CO_3	3.0E+01	
Br	--	
F	7.5E+03	
NO_2	3.00E+03	IC Anions
NO_3	4.5E+02	
oxalate	1.80E+03	
PO_4	6.0E+02	
SO_4	1.2E+03	
Hg	1.5E+00	Cold Vapor AA
CN	3.0E+00	Colorimetric
NH_3	6.0E+01	ISE
Total and Free OH	7.50E+04	Titration
Organic Analytes[(c)]	**µg/g**	
Acetate	--	
Citrate	1.50E+03	
Formate	1.50E+03	

Table C.2. (contd)

Analyte	Slurry Minimum Reportable Quantity(a)	Analysis Method/Driver
Gluconate	1.50E+03	IC (Organic Anions)
Gylcolate	1.50E+03	
D2EHPA	1.50E+03	
EDTA	1.50E+03	
HEDTA	1.50E+03	Derivatization/GC-MS
IDA	1.50E+03	
NTA	1.50E+03	
Radionuclides	**µCi/g**	
^{99}Tc	6.0E+00 µg/g	
^{237}Np	1.8E+00 µg/g	
^{239}Pu	3.00E-02	ICP-MS (Pretreatment Specification)
^{240}Pu	1.00E-02	
^{241}Pu/^{241}Am	5.10E-02	
^{129}I	3.0E+01 µg/g	
^{233}U	--	
^{234}U	--	ICP-MS (Vitrification Request)
^{235}U	--	
^{236}U	--	
^{238}U	--	
^{242}Pu	--	
^{99}Tc	Not required	Separations/Liquid Beta Scintillation without sample oxidation to determine pertechnetate
^{63}Ni	--	Beta Scintillation
^{90}Sr	7.01E+01	
^{241}Pu	--	Separations/Liquid Scintillation
^{3}H	1.5E-02	
^{14}C	1.8E-03	Distillation and Liquid Scintillation
^{151}Sm	--	Separation/Beta Scintillation
^{79}Se	--	
^{236}Pu	--	
^{238}Pu	1.00E-02	

Table C.2. (contd)

Analyte	Slurry Minimum Reportable Quantity$^{(a)}$	Analysis Method/Driver
^{239}Pu	6.0E+00 μg/g	
$^{239/240}$Pu	3.00E-02	Separations/AEA
^{241}Am	1.2E-03	
^{242}Am	--	
^{242}Cm	1.50E-01	
^{242}Pu	--	
$^{243/244}$Cm	6.0E-05	
^{51}Cr	--	
^{59}Fe	--	
^{60}Co	1.2E-02	
^{88}Y	--	
^{95}Nb	--	
^{103}Ru	--	
^{106}Ru	--	
^{113}Sn	--	
^{125}Sb	6.0E+00	Extended Counting Time GEA
^{126}Sn	6.0E-02	
^{126}Sn/Sb	--	
^{134}Cs	--	
^{137}Cs	6.0E-02	
^{144}Ce	--	
^{152}Eu	--	
^{154}Eu	6.0E-02	
^{155}Eu	6.0E-02	
^{232}Th	--	
Total Alpha	2.30E-01	Alpha Counting
Sum of Alpha	To be determined	Summation of ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{242}Cm $^{243+244}$Cm

Table C.2. (contd)

Analyte	**Slurry Minimum Reportable Quantity**[(a)]	**Analysis Method/Driver**
Physical Property	**Expected Range**	
Wt% Oven Dried Solids	0.1 to 100 wt%	Gravimetry
Separate Organic Phase	N/A	Visual Observation
Density	0.9 to 1.7 gm/mL	Gravimetry
Wt% Oxides	0.1 to 100 wt%	Gravimetry

(a) Those analytes without a specified MRQ are to be determined as a best effort by the laboratory. The detection limit for each analyte should be reported along with the analytical results. Matrix spikes and laboratory control standards are not required for these analytes, but should be reported when available.

(b) Report any additional ICP-AES analytes on an opportunistic basis.

(c) If organic analytes listed are not found in the initial sludge, this analysis will be omitted.

Appendix D

Raw Filtration Data

Appendix D

Raw Filtration Data

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
11/1/2001	1.0	3:08	22.2	3.8	20	19.8	40	17.2
	1.0	3:18	23.5	3.86	20	20	20	33.2
	1.0	3:28	25.7	3.76	21	21	10	22
Backpulsed								
Test stopped due to break on volumetric cylinder.								
Backpulsed again								
11/2/2001	1.0	7:34	25.5	3.62	34	34	30	28.9
Stopped. Flow too low. Will do another acid clean. Acid cleaned system rinsed. Neutralized.								
	1.0	10:31	28.9	3.84	10	10	40	10.84
	1.0	10:40	23.9	3.78	10	10	10	19.88
	1.0	10:50	22.1	3.72	10	10	10	29.12
Backpulsed								
	1.0	10:56	24.9	3.75	20	21.6	30	9.03
	1.0	11:06	22.2	3.78	20	21.6	10	20.15
	1.0	11:16	27.5	3.78	20	21.5	10	20.43
Backpulsed								
	1.0	11:22	24.6	3.7	30	31.5	10	10.4
	1.0	11:31	19.1	3.73	30	30.7	10	19.38
	1.0	11:41	25.7	3.73	30.5	31.7	10	19.69
Ran system with 1 M NaOH ~1 hour.								
	1.0	2:09	23.7	3.76	10	10.8	10	5.3
	1.0	2:18	21.1	3.77	10	11	10	22.2
	1.0	2:28	24.5	3.76	10	11	10	24.69
Backpulsed								
	1.0	2:32	26.9	3.7	31	32	30	13.25
	1.0	2:42	25.5	3.73	31	31	10	11.71
	1.0	2:52	26.3	3.7	30.5	31	10	12.56
Backpulsed								
	1.0	2:56	24.9	3.75	20	20.1	10	4.28
	1.0	3:06	22.9	3.75	20	20	10	20.34
	1.0	3:16	26.4	3.75	20	20.5	10	20.85
11/7/2001	1.1	9:43	23.5	3.87	31	30	30	15.4
	1.1	9:53	24.5	3.82	33	31	30	15.4
	1.1	10:03	22.9	3.71	31	30.1	30	18
Backpulsed								
11/7/2001	1.1	10:11	22.2	3.84	21	20	30	20.2
	1.1	10:21	22.8	3.72	21	20	10	7.2
	1.1	10:31	21.1	3.75	22	20	10	7.8

Date	Test Number	Time	Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
			Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
Backpulsed								
	1.1	10:36	21	3.7	11	10	30	34.4
	1.1	10:46	21.8	3.78	10	10	30	36
	1.1	10:56	22.6	3.7	10	10	20	24
Backpulsed								
	1.2	10:28	21.2	3.5	35	32	30	5.8
	1.2	10:38	22.7	3.9	31	30	40	7
	1.2	10:48	24	3.7	32	31	30	4.6
Backpulsed								
	1.2	10:51	23.9	3.81	21	20	40	9
	1.2	11:01	24	3.75	21	20	40	9.2
	1.2	11:11	24.1	3.85	21	20	40	9.2
Backpulsed								
	1.1	11:15	23.9	3.76	10.5	10	30	11.6
	1.1	11:25	23.6	3.73	11	11	30	11.6
	1.1	11:35	23.4	3.83	10	10	30	13
11/12/2001	1.3	8:45	24.7	3	46	44	20	30.6
	1.3	8:58	23.6	3.7	40	38	30	69.4
	1.3	9:06	25.1	2.72	60	53	30	60.8
	1.3	9:16	23.6	3.53	42	40	20	56.2
	1.3	9:25	25	3.23	45	42	20	52
	1.3	9:35	23.8	3.61	48	46	20	51
	1.3	9:45	24.5	3.6	42	38	20	55.2
Backpulsed								
	1.4	9:57	24.8	2.7	52	50	20	32
	1.4	10:00	24.9	4.3	40	38	20	48.8
	1.4	10:07	24.9	3.7	44	43	20	47.8
	1.4	10:17	25.7	3.9	45	43	15	35.8
	1.4	10:29	24.7	3.3	45	42	20	56.4
	1.4	10:37	25.8	3.6	43	41	20	54.6
	1.4	10:47	24.4	4	43	40	20	60
	1.4	10:57	25	3.3	48	45	20	52.8
Backpulsed								
	1.5	11:05	23.9	3.3	47	45	20	37.2
	1.5	11:20	26	3.6	43	41	20	48.2
	1.5	11:26	24.5	3.8	42	40	15	39.8
	1.5	11:37	24.5	3.7	47	45	20	48.8
	1.5	11:46	24.7	3.3	43	41	20	55.6
	1.5	11:56	24.1	4	42	40	15	44.8
	1.5	12:06	25.3	3.9	42	40	20	55.8
Backpulsed								
	1.6	12:17	23.7	2.9	32	31	10	25.2
	1.6	12:28	23.9			40	10	24
	1.6	12:30	22.1	2.4	35	33	13	37.3
	1.6	12:36	21.5	2.8	35	33	14	40.8
	1.6	12:46	22.2	3	32	30	18	58.4
	1.6	13:00	21.5	2.5	32	30	8	24.6
	1.6	13:08	22	3	30	27	13	45
	1.6	13:15	22.3	3	30	30	15	53.6

Date	Test Number	Time	Slurry Temperature (°C)	Slurry Loop Flow Rate (gpm)	Filter Inlet Pressure (psig)	Filter Outlet Volume (psig)	Permeate Flow Rate	
							Volume (mL)	Time (s)
Backpulsed								
	1.7	13:30	23.9	4.7	30	28	16	43
	1.7	13:40	25.2	4.8	30	26	15	49.6
	1.7	13:50	26.2	4.5	35	31	20	60.6
	1.7	14:02	25.2	4.6	30	29	15	51
	1.7	14:11	26.5	4.4	30	30	15	48.8
	1.7	14:20	24.8	4.5	34	30	15	57
	1.7	14:31	25.6	4.6	33	30	15	67.6
Backpulsed								
	1.8	14:45	Started					
	1.8	14:50	27.1	4.5	60	56	10	18.6
	1.8	14:58	26.3	4.5	50	48	15	33.56
	1.8	15:05	25.9	4.6	48	46	20	49
	1.8	15:16	27.6	4.2	54	52	20	46.2
	1.8	15:26	24.9	4.3	55	52	16	39.6
	1.8	15:36	36.9	4.3	55	52	30	71.8
	1.8	15:46	25	4.4	55	51	20	53
Backpulsed								
	1.9	15:58	25.1	3.1	53	51	30	58.2
	1.9	16:07	22.4	2.7	55	53	15	37.8
	1.9	16:15	22.9	3	53	50	10	30.6
	1.9	16:26	23.7	3.3	50	48	10	32.8
	1.9	16:36	23.1	3.2	55	53	10	28.3
	1.9	16:46	24.8	3.1	53	50	10	29.2
	1.9	16:56	23.1	3.1	53	50	10	28.5
Completed one backpulse.								
	1.10	17:09	23.5	3.6	43	41.8	10	22.2
	1.10	17:19	23.2	3.7	44	42.8	10	23.9
	1.10	17:29	25.3	3.9	41	38.9	10	25.7
	1.10	17:39	22.7	3.8	41	38.4	10	29
	1.10	17:49	22	3.8	42	40	10	29
	1.10	17:59	24.1	3.8	42	40	10	26.3
	1.10	18:09	22.5	3.7	44	42	10	32.4
Backpulsed								
	1.11	18:21	23	2.45	40	39.8	10	24.3
	1.11	18:31	21.2	2.4	41	41	10	32
	1.11	18:41	22.1	2.4	40	38.9	10	32.6
	1.11	18:51	21.4	2.2	44	43	10	40.8
	1.11	19:01	21.5	2.4	45	43	10	36.3
	1.11	19:11	22.4	2.5	42	40	10	41.1
	1.11	19:21	21.2	2.6	39	38	10	46.4
Backpulsed								
	1.12	19:32	23.9	5.1	45	39	10	21.5
	1.12	19:42	34.6	5.1	45	38	10	29.3
	1.12	19:52	23.9	5.3	42	36	10	32.6
	1.12	20:02	24.2	5.05	47	43	10	28.8
	1.12	20:12	23.8	5.1	50	45	10	27.8
	1.12	20:22	24.8	5.1	44	40	10	31.2
	1.12	20:32	24.4	5.1	42	38	10	34.4

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
Backpulsed								
	1.13	20:45	20.5	3.8	22	19.5	10	40.7
	1.13	20:55	21.3	3.8	21	19	10	
	1.13	21:00	22.3	3.8	21	19	10	45.5
	1.13	21:05	23	3.7	22	20	10	
	1.13	21:10	23.8	3.8	21	19	5	27.6
	1.13	21:15	24.3	3.7	24	22	10	40.3
	1.13	21:25	20	3.8	23	21	5	26.2
	1.13	21:35	20.6	3.7	23	21	5	27.4
	1.13	21:45	22.4	3.6	24	22	10	46
Backpulsed								
	1.14	21:59	23.3	3.5	68	64	20	34.5
	1.14	22:09	22.6	3.7	64	60	10	22.4
	1.14	22:19	24.3	3.7	62	58	10	22.7
	1.14	22:29	23.5	3.6	64	62	10	23.1
	1.14	22:39	24.7	3.5	63	61	10	23.8
	1.14	22:49	24.1	3.5	63	61	10	23.2
	1.14	22:59	24.1	3.8	60	58	10	25.8
Backpulsed								
	1.15	23:09	23.1	3.8	41	38	10	28
	1.15	23:19	21.8	3.8	42	40	10	27.65
	1.15	23:29	24	3.7	43	41	10	25.2
	1.15	23:39	23.4	3.7	43	41	10	25.6
	1.15	23:49	23	3.8	43	41	10	29.1
	1.15	23:59	23.4	3.7	44	42	10	26.7
11/13/2001	1.15	0:09	23.8	4	40	38	10	30.03
No backpulse								
	1.16a	0:19	23.3	3.77	41.5	39.2	10	27.06
	1.16a	0:29	22.9	3.9	40	37.6	10	31.22
	1.16a	0:39	24.3	3.34	45	42	10	27.19
	1.16a	0:49	22.7	3.66	42	39.9	10	32.21
	1.16a	0:59	24.1	3.59	42	40	10	29.15
	1.16a	1:09	22.7	3.99	43	41	10	28.72
	1.16a	1:19	23.9	4.12	41.5	38.2	10	30.72
	1.16a	1:29	22.8	3.94	40	37	10	31.94
	1.16a	1:39	23.8	3.77	41	39.8	10	30.37
	1.16a	1:49	23.2	4.08	42	38.9	10	29.59
	1.16a	1:59	23.3	3.97	41	39.6	10	31
	1.16a	2:09	23.5	3.92	40.5	38.9	10	30.78
	1.16a	2:19	23.2	3.99	40.5	40.2	10	29.75
	1.16a	2:29	24.4	3.88	42	38	10	29.81
	1.16a	2:41	23.4	3.89	43	40.5	10	31.63
	1.16a	2:49	24.4	4	42	40.1	10	31.72
	1.16a	2:59	22.8	3.97	40.5	40	10	33
	1.16a	3:09	24.3	3.86	41	39.7	10	31.44
	1.16a	3:19	22.6	3.92	41	38.6	10	31.63
	1.16a	3:29	23.8	21:21	40	37	10	32.91
	1.16a	3:39	22.7	4.01	41	38	10	30.97
	1.16a	3:49	23.5	3.94	40.5	38.1	10	29.72

Date	Test Number	Time	Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
			Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
	1.16a	3:59	22.8	3.8	40.5	37.9	10	33.56
	1.16a	4:09	23.1	4.1	41	38	10	32
	1.16a	4:19	23.4	3.83	40	38.9	10	32.34
	1.16a	4:29	22.9	3.99	42	38.4	10	31.91
	1.16a	4:39	23.7	3.88	41	38.1	10	34.03
	1.16a	4:49	22.6	3.93	42	39.1	10	33.41
	1.16a	4:59	24	3.895	40	37.2	10	31.85
	1.16a	5:08	22.4	3.81	41	39.2	10	31.9
	1.16a	5:19	24	3.86	41	40.2	10	32.25
	1.16a	5:29	22.5	3.73	41	39.5	10	31.31
	1.16a	5:39	23.6	3.81	40.5	38.4	10	32.16
	1.16a	5:49	22.6	3.79	41	39.1	10	33.94
	1.16a	5:59	23.3	3.74	40	38.7	10	31.4
	1.16a	6:09	22.8	3.62	40	38.6	10	33.87
	1.16a	6:19	23	3.8	41	40.2	10	33.53
	1.16a	6:29	23.3	3.79	40	38.8	10	33.65
	1.16a	6:39	22.6	3.76	40	39.2	10	32.44
	1.16a	6:49	23.8	3.76	41	39.3	10	32.75
	1.16a	6:59	22.5	3.6	42	37.2	10	33.3
	1.16a	7:09	23.8	3.92	42	39.9	10	36.03
	1.16a	7:19	22.3	3.8	40	37.2	10	35.22
	1.16a	7:29	23.8	3.71	41	38.2	10	32.07
	1.16a	7:39	22.1	3.65	41	39	10	31.09
	1.16a	7:49	23.3	3.96	40	38.6	10	34.44
	1.16a	7:59	22.4	3.67	40	39.1	10	33.19
	1.16a	8:10	23.4	3.6	43	41	10	30.22
	1.16a	8:30	22.3	3.6	40	38	10	35.9
	1.16a	8:40	22.6	3.9	39	37	10	39.91
Dewater								
	1.16b	8:48	22	3.6	42	40	10	34.06
	1.16b	8:58	23.4	3.7	38	36	10	36.41
	1.16b	9:08	21.8	3.8	41	39	10	38.28
	1.16b	24:14 Minutes #1 Filled 400 mL.						
	1.16b	9:15	23	3.7	42	40	10	31.09
	1.16b	9:25	22.5	3.6	43	41	10	32.03
	1.16b	9:34	22.5	3.8	43	41	10	35.56
	1.16b	21:56 Minutes #2 Filled 400 mL.						
	1.16b	9:40	24					
	1.16b	9:47	22.5	3.8	41	39	10	37
	1.16b	9:52		3.7	41	39		
	1.16b	9:55	22.6	3.7	41	39	10	37.75
	1.16b		23.6	3.5	42	40		
	1.16b	10:02	24	3.4	42	40	10	35.85
	1.16b	10:04	23.8	3.6	40	38	10	
	1.16b	10:06	23.1	3.7	41	39	10	38.75
	1.16b	10:12	21.9	3.6	44	42	6:11 ~100 mL	
	1.16b	10:19	23.2	3.9	40	38	10	38.4
	1.16b			3.9	40	38	18:35 ~300 mL	
	1.16b	10:28	23.2	3.7	41	39	10	36.13

Date	Test Number	Time	Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
			Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
	1.16b	24:09 ~400 mL #4 full						
	1.16b	10:32	21.9	3.5	40	38	10	36.78
	1.16b	10:38	22.9	3.6	41	39	6:10 ~100 mL	
	1.16b	10:42	23.9	3.6	43	41	10	33.03
	1.16b	10:45	24.5	3.6	44	42	12:14 ~200 mL	
	1.16b	10:50	22.8	3.5	42	40	10	37.16
24:23 ~400 mL #5 full								
End of dewatering - stirrer turned off - pump on.								
Backpulse once before starting test matrix 1.7.								
	1.17	11:21	22.7	3.7	43	40	10	30.43
	1.17	11:31	24	3.7	41	39	10	38.16
	1.17	11:43	23.3	3.9	44	42	10	38.07
	1.17	11:53	24.8	3.8	41	38	10	38.51
	1.17	12:02	22.6	3.8	41	39	10	39.6
	1.17	12:13	26	3.9	42	39.6	10	36
	1.17	12:22	24	3.9	42	39	10	38.56
Backpulsed								
	1.18	12:30	24	3.7	42	39	10	27.3
	1.18	12:40	25.9	3.8	42	38	10	35.8
	1.18	12:50	23.9	3.6	42	38	10	38.2
	1.18	13:00	26.2	3.6	43	40	10	37
	1.18	13:11	23.9	3.7	42	40	10	37.8
	1.18	13:20	25.4	3.7	42	40	10	37.1
	1.18	13:31	23.7	3.8	42	40	10	40
Backpulsed								
	1.19	13:38	24	3.8	40	38	10	31.34
	1.19	13:48	26	3.9	43	41	10	37.2
	1.19	13:58	24	3.8	40	38	10	38.68
	1.19	14:08	26.1	3.7	41	39	10	35.47
	1.19	14:18	23.6	3.8	42	40	10	38.18
	1.19	14:28	25.3	3.9	43	41	10	36.16
	1.19	14:38	23.6	3.6	46	44	10	38.89
Backpulsed								
	1.20	14:47	24.2	3.2	31	30	10	36.44
	1.20	14:57	21.6	3	31	30	10	52.75
	1.20	15:07	22.6	3	30	29	10	55.09
	1.20	15:20	22.2	2.9	30	28	10	58.6
	1.20	15:30	22.7	3	34	32	10	51.97
	1.20	15:37	23.7	3	33	31	10	52.22
	1.20	15:47	23.3	3.2	35	33	10	52.28
Backpulsed								
	1.21	16:00	25.2	4.4	34	30	10	27.78
	1.21	16:10	23.9	4.6	34	30	10	32.4
	1.21	16:20	25.9	4.7	32	28	10	32.6
	1.21	16:30	34.2	4.3	30	27	10	35.4
	1.21	16:40	25.5	4.5	30	27	10	34.4
	1.21	16:49	25.4	4.59	31	28	10	33.2
	1.21	17:00	24.6	4.5	30	27	10	36.2

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
Backpulsed								
	1.22	17:12	26.5	4.48	51	49	10	25.4
	1.22	17:22	24.7	4.43	51	48	10	34
	1.22	17:35	27.6	4.41	53	50	10	30.2
	1.22	17:44	23.9	3.25	49	45	10	39.2
	1.22	17:48	24.4	4.58	52	49	10	33.6
	1.22	17:55	27.1	4.7	51	56	10	33.6
	1.22	18:06	23.9	4.7	48	43	10	38
	1.22	18:13	23.7	4.57	49	44	10	38.4
Backpulsed								
		18:22	Started					
	1.23	18:23	24	3	49	46.5	10	35
	1.23	18:33	22.3	3.2	50	47	10	57.2
	1.23	18:43	24.3	3.1	48	44	10	58.6
	1.23	18:52	23.5	3	48	44	10	59.4
	1.23	19:03	23.6	3.2	49	46	10	57.2
	1.23	19:15	23.6	3.1	48	46	10	57
	1.23	19:25	24.7	3.4	50	47	10	60
Backpulsed								
		19:37	Started					
	1.24	19:38	24.8	3.84	41	38	10	29.8
	1.24	19:48	24.5	3.7	42	40	10	36.4
	1.24	19:58	27	3.75	37	40	10	39.8
	1.24	20:08	24	3.84	38	41	10	40.8
	1.24	20:18	25.8	3.7	38	41	10	40.4
	1.24	20:28	25.1	3.8	39	42	10	40.4
	1.24	20:37	24.9	3.9	41	36	10	44.2
Backpulsed								
	1.25	20:54	23.5	2.3	41	40	10	42
	1.25	21:08	24.5	2.6	42	40	5	37.2
	1.25	21:16	25.1	2.55	45	43	5	35
	1.25	21:24	22.6	2.43	45	43	5	40
	1.25	21:34	24	2.42	44	43	5	39.6
	1.25	21:43	24.4	2.5	41	40	5	40
	1.25	21:54	22.9	2.5	44	43	5	42
Backpulsed								
	1.26	22:05	25.4	4.9	45	40	10	25.4
	1.26	22:14	23.4	4.6	48	44	10	31.6
	1.26	22:24	25.2	5.19	40	35	10	34
	1.26	22:34	23.9	5.08	41	36	10	32.5
	1.26	22:44	25.3	5.2	41	36	10	32.5
	1.26	22:54	25.7	5.15	40	35.1	10	30.8
	1.26	23:04	24.8	5.25	40	35	10	31
Backpulsed								
	1.27	23:15	21	3.82	22	20.7	5	36.4
	1.27	23:26	23.3	4.07	25	22.8	10	38
	1.27	23:38	22.2	3.92	22	20.1	10	47.5
	1.27	23:48	24.4	3.9	22	20.7	10	44
	1.27	23:55	24.3	3.95	22	20.7	10	43

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
11/14/2001	1.27	0:05	23.7	3.77	20	19.2	10	48.2
	1.27	0:15	25.2	3.92	22	20.6	10	44
Backpulsed								
	1.28	0:33	30.5	3.5	62	61.4	10	34
	1.28	0:43	25.9	3.76	60	58.3	10	45.07
	1.28	0:53	22	3.67	60	58.5	10	50.13
	1.28	1:03	24.6	3.89	60.5	59.2	10	45.12
	1.28	1:13	21.8	3.5	61	60.4	10	49.28
	1.28	1:23	24	3.83	60	57.7	10	47.44
	1.28	1:33	22.3	3.5	60	59.6	10	49.78
Backpulsed								
	1.29	1:48	23.5	3.65	40	38.9	10	35.31
	1.29	1:58	18.5	3.7	42	39.5	10	48.59
	1.29	2:08	20.3	3.69	42	40.6	10	47.72
	1.29	2:18	18.3	3.64	42	39.9	10	51.63
	1.29	2:27	19.3	3.66	41	39.8	10	51.57
	1.29	2:37	19.9	3.84	43	39	10	48.94
	1.29	2:47	19.7	3.87	44	41.9	10	46.12
	1.29	2:57	22.8	3.87	42	40.8	10	47.65
CUF in idle @ ~5:00 am after sample pulls.								
Backpulsed								
	1.29a	5:57	19.7	3.54	41	40.9	10	41.03
	1.29a	6:07	20.3	3.89	42	40.1	10	51.87
	1.29a	6:47	22.5	3.91	40	37.8	10	61.9
	1.29a	7:17	20.9	3.72	42	40.6	10	49.21
	1.29a	7:32	20	3.7	40.5	38	10	61.6
	1.29a	7:58	20.1	3.92	41	36.9	10	63.94
	1.29a	8:07	22.2	3.94	42	40.5	10	54.16
	1.29a	8:17	22.5	3.84	44	42	10	48.81
	1.29a	8:27	22.2	3.7	41	39	10	54.38
	1.29a	8:37	25.1	3.88	42	40	10	52.19
	1.29a	8:47	21.7	3.8	42	40	10	56.1
	1.29a	8:57	24.5	3.86	41	39	10	52.4
	1.29a	9:07	22.5	3.9	42	40	10	56.5
	1.29a	9:17	22.8	3.9	43	41	10	51.63
	1.29a	9:27	25.5	3.8	42	40	10	49
	1.29a	9:37	22.1	3.75	43	41	10	54.68
	1.29a	9:47	25	3.77	43	41	10	54.68
	1.29a	9:57	23.9	3.67	41	39.2	10	54.72
	1.29a	10:07	24.5	3.81	45	42	10	50.34
	1.29a	10:17	24.2	3.8	45	43	10	48.44
	1.29a	10:27	22.5	3.65	40	38	10	58.94
	1.29a	10:37	25.5	3.75	43	41	10	51.87
	1.29a	10:47	22.2	3.8	44	41	10	55.13
	1.29a	10:57	24.2	3.9	43	39	10	53.6
	1.29a	11:08	22.5	3.7	41	38	10	53.28
	1.29a	11:18	23.8	3.7	42	40	10	53.69
	1.29a	11:28	25.1	3.7	43	40	10	52.22
	1.29a	11:38	22.5	3.76	44	41	10	54.22

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
	1.29a	11:47	25.2	3.8	45	42	10	50.32
	1.29a	11:58	22.2	3.8	45	42	10	58.25
	1.29a	12:08	23.6	3.7	43	40	10	51.78
	1.29a	12:25	22.5	3.8	42	40	10	56.5
	1.29a	12:38	25.8	3.9	43	40	10	49.94
	1.29a	12:54	23.8	3.9	43	41	10	53.62
	1.29a	13:05	23.4	3.8	41	39	10	54.09
	1.29a	13:18	24	3.9	44	41	10	54.4
	1.29a	13:27	25	3.8	40	38	10	53.03
	1.29a	13:37	22.9	3.8	42	39	10	57.69
	1.29a	13:47	25.8	3.7	41	38	5	25.37
	1.29a	13:57	22.3	3.7	43	40	10	56.34
	1.29a	14:07	24.5	3.7	43	40	10	52.87
	1.29a	14:17	23.6	3.8	43	40	10	55.47
	1.29a	14:26	23.9	3.8	43	40	10	55.46
	1.29a	14:37	25.8	3.8	43	40	10	52.85
	1.29a	14:48	22.9	3.8	43	40	10	55.79
	1.29a	14:58	25.9	3.7	43	40	10	51.06
	1.29a	15:12	23.5	3.8	43	40	10	54.1
	1.29a	15:20	25.7	3.9	44	41	10	50.78
	1.29a	15:30	22.3	3.7	40	38	10	58.53
	1.29a	15:45	26	3.9	45	42	10	49.62
	1.29a	15:57	Level measurement					
	1.29a	16:01	24	3.9	44	41	10	54.28
Stopped run – missed data point solids from AZ-101. Theo 20% to the CUF. We got a temporary plug. Extended run completed before adding residue solids.								
		16:58	Started dewatering test 1.29 B Level = 5.0 in. = ~1625 mL.					
	1.29b	17:00	23.2	3.2	50	47	10	35.8
		17:05	Removed 100 mL total.					
	1.29b	17:08	21.8	3.8	39	42	5	25.6
		17:14	Removed 200 mL total.					
	1.29b	17:17	24.9	3.6	39	43	5	24.4
		17:22	Removed 300 mL total into AZ-101 Super #6.					
			Level = 4 5/8 in. = 1550 mL.					
	1.29b	17:34	Started dewatering into AZ-101 Super #7.					
	1.29b	17:35	23.7	3.8	44	40	10	62.4
	1.29b	17:44	27	3.7	42	39	9	56.4
		17:45	Removed 100 mL into AZ-101 Super #7.					
		17:49	Level 4 3/8 in. = 1400 mL.					
		17:54	25.6	3.6	46	41	5	30.6
	1.29b	17:56	Removed 200 mL into AZ-101 Super #7.					
	1.29b	18:00	Stopped dewatering due to temporary plug.					
			Have ~225 mL in AZ-101 Super #7.					
			Level = 3 3/8 in. = 1000 mL.					Inconsistent with previous reading.
			Removed 3 in. accuracy of level indicate or poor at these low levels.					
			Level = 5 3/4 in.					
	1.29.1	18:34	Started Dewatering Test 1.29.1.					
	1.29.1	18:37	24.3	3.5	45	43	10	19.4

			Slurry	Slurry Loop	Filter Inlet	Filter Outlet	Permeate Flow Rate	
Date	Test Number	Time	Temperature (°C)	Flow Rate (gpm)	Pressure (psig)	Volume (psig)	Volume (mL)	Time (s)
	1.29.1	18:41	Removed 100 mL into permeate #1.					
		18:44	Removed 200 mL into permeate #1					
		18:45	26	3.66	42	39.2	10	21.8
		18:49	Removed 300 mL into permeate #1.					
	1.29.1	18:54	24.5	3.7	43	40	10	22.8
		18:56	Removed 500 mL into permeate #1.					
		18:57	Level 3 7/8 in.					
	1.29.1	19:03	27.2	3.6	44	41	10	29.6
		19:06	Removed 700 mL into permeate #1 - stopped test to take a level - took 2nd measurement with flow at 1.3 gpm.					
			Level = 3 in.					
		20:00	Added 1 liter of inhibited water to CUF.					
			Took level measurement = 6 in. = 2000 mL.					
	1.29.3	20:32	Start Test (2nd Wash)					
	1.29.3	20:33	20.2	Various	Various	Various	20	25
		20:35	20.2	3.6	42	39	5	7.6
		20:37	Removed 200 mL into permeate #1.					
		20:41	Removed 300 mL into permeate #1.					
	1.29.3	20:42	20.8	3.5	40	38	15	27.8
		20:44	Removed 400 mL into permeate #1.					
		20:47	Removed 500 mL into permeate #1. Stopped dewatering to switch bottles.					
	1.29.3	20:58	Started dewatering into permeate #2.					
	1.29.3	20:58	23	3.9	37	34	15	22.6
	1.29.3	21:00	Removed 100 mL into permeate #2.					
	1.29.3	21:03	Removed 200 mL into permeate #2.					
	1.29.3	21:06	Removed 300 mL into permeate #2.					
	1.29.3	21:07	21.2	3.7	44	40	10	22
	1.29.3	21:10	Removed 400 mL into permeate #2.					
	1.29.3	21:13	22.8	3.7	43	39	10	25
	1.29.3	21:14	Stopped dewatering.				Total in permeate #2 ~500 mL.	
	1.29.3	21:20	Turned down pump and pressure.					
	1.29.3	11:18	Caustic leach completed.					
11/15/2001	1.29.4	11:48	Agitator off temp = 24°C.					
	1.29.4	11:57	23.2	3.5	45	43	15	25.81
	1.29.4	12:00						
	1.29.4	12:03	23.9	3.7		40.3		
	1.29.4	12:06	22.6	3.6	41			
	1.29.4	12:07	21.3	3.9	39	37	15	32.57
	1.29.4	12:15						
	1.29.4	12:17	20.9	3.8	40	38	15	32.91
	1.29.4	12:21	21.6	3.74	41	39		
	1.29.4							
	1.29.4							Bottle removed
	1.29.4	12:28	AZ - Leach bottle filled.				1:50 records to fill ~55 mL.	
	1.29.4		Level Measurement, 5 3/4 in..					

Date	Test Number	Time	Slurry Temperature (°C)	Slurry Loop Flow Rate (gpm)	Filter Inlet Pressure (psig)	Filter Outlet Volume (psig)	Permeate Flow Rate Volume (mL)	Time (s)
	1.29.4	12:36	22.6	3.9	39	37	15	32.35
	1.29.4	12:43	Removed 200 mL.					
	1.29.4	12:46	23.1	3.6	41	39	15	33.28
	1.29.4							
	1.29.4		24.4	3.7		37		
	1.29.4			3.7		37		
	1.29.4	12:56	25.3	3.7	41	40	15	31.69
	1.29.4							
			Take Level Measurement					
		13:35	1200 mL inhibited water added.					
		13:42						
		14:14	22.6	3.7	41	39	15	33.1
								Time
								Time
		14:24	24	3.5	41	39	15	32.09
	1.29.5							
						Time	18:14 Minutes:sec Removed 500 mL.	
	1.29.5	14:34	23.3	3.5	41	39	15	32.6
	1.29.5	14:40	Put system on recycle in order to switch sample bottles.					
	1.29.5	14:41	Permeate going to sample "AZ-Rinse".					
	1.29.5	14:43	Removed 50 mL into AZ-rinse system back to recycle.					
		14:45	Permeate going to AZ-permeate #2.					
			Reset clock counter to use undercounting up.					
	1.29.5	14:48	24.7	3.7	43	40	15	29
		14:52						
		14:54						
	1.29.5	14:55	25.3	3.6	44	42	15	27.69
		15:01	CUF on Recycle Rinse #1 completed.			Total volume removed = 500 +50 +700 = 1250 mL.		
	1.29.5a	15:30	24	3.8	45	42	15	26.6
	1.29.5a	15:40	25.8	3.8	46	44	15	23.41
						Bottle # 1 removed.		
	1.29.5a	15:52	Bottle#2 t=0					
		15:53	23.5	3.9	42	40	15	27.97
	1.29.5a		24.6	3.7	45	43	15	25.81
							Bottle #2	
	1.30	16:26	Started dewater of 3rd rinse.					
	1.30	16:58	Reached first 100 mL in bottle.					

	1.30	17:00	20.5	4	43	42	10	16.6
	1.30	17:00	Reached 200 mL in permeate 1.					
	1.30	17:04	Reached 400 mL in permeate 1.					
	1.30	17:06	22.6	3.86	45	43.7	20	27.6
	1.30	17:08	Reached 500 mL in permeate 1.					
	1.30	17:12	Reached 700 mL in permeate 1.					
	1.30	17:28	22.8	3.75	43	41	15	20
	1.30	17:28	Started filling permeate 2.					
	1.30	17:30	Reached 100 mL in permeate 2.					
	1.30	17:32	Level = 4 3/4 in.					
	1.30	17:33	Reached 200 mL in permeate 2.					
	1.30	17:35	Reached 300 mL in permeate 2.					
	1.30	17:37	24.9	3.8	42	40	20	28.6
	1.30	17:38	Reached 400 mL in permeate 2.					
	1.30	17:40	Turned off flow to permeate 2.					
	1.30	17:46	Took level 4 1/8 in. (low pressure).					
		17:50	Took second level at standard condition 3 7/8 in.					
Began emptying CUF								
	1.31.1	10:58	22.8	3.8	11	9.7	15	27.4
	1.31.1	11:08	23	3.73	11	9.7	15	27.66
	1.31.1	11:18	20.4	3.7	10	9.4	15	31.72
Backpulsed								
	1.31.1	11:25	21.9	3.8	22	21	30	20
	1.31.1	11:35	23.8	3.83	22	20.7	30	25.25
	1.31.1	11:45	22.2	3.81	22	20.8	30	27.03
Backpulsed								
	1.31.1	11:51	23.3	3.83	32	30	30	16.07
	1.31.1	12:01	25.1	3.82	32	30.1	30	17.66
	1.31.1	12:11	23.5	3.7	33	31	30	18.22
Backpulsed 12:13 pump shut down								
			Drained CUF After CWF Test.					
			Added $SrCO_3$ to CUF.					
11/16/2001	1.33	13:23	21.9	3.75	10	8.9	15	20
	1.33	13:33	23.6	3.86	10.5	9.2	15	20.8
	1.33	13:43	22.3	3.7	10.5	9.3	15	21.8
Backpulsed								
	1.33	13:50	23	3.8	21.5	20	30	20
	1.33	14:00	24.6	3.73	22	20	30	20.2
	1.33	14:10	22.8	3.73	22	20	30	21.2
Backpulsed								
	1.33	14:13	23.7	3.77	31	29	30	13.6
	1.33	14:23	25.3	3.65	31	29.4	40	18.4
	1.33	14:33	23.6	3.67	30.5	28.9	40	20.2
Backpulsed								
	Drained $SrCO_3$ - Had slight gray hue to it.							
		14:50	Rinse 1 - 0.5 L I.W.					
		15:10	Rinse 2 - 0.5 L I.W.					
			Rinse 3 - 1.0 L I.W.					
		15:45	Added 1 liter to let sit over the weekend					
11/19/2001		9:15	Ran system and conducted backpulse (rinse 4)					
		9:44	Backpulse					
		9:48	20.2	3.7	12	9.9	20	13
		9:58	24.9	3.7	11	9	30	30.8

		10:08	22.3	3.7	10.5	8.6	20	23.6
		10:10	Backpulse					
		10:11	23.2	3.9	22	19	40	19.6
		10:21	24.8	3.8	22.5	19.5	30	16.6
		10:31	23.1	3.8	22.5	19.5	30	17.2
		10:33	Backpulse					
		10:34	23.7	3.7	32	29.1	40	13.8
		10:44	25.5	3.8	32.5	29.5	20	7.8
		10:54	24	3.8	32	29	40	16
			Shut system down and drained.					
			Acid cleaned.					
			Neutralized acid rinse.					
Backpulsed								
		13:51	26	3.8	12	9.1	30	11.4
		14:01	22.2	3.7	12	9.2	30	12
		14:11	23.2	3.7	12	9.3	30	16.2
Backpulsed								
		14:14	24.2	3.9	22	19.4	30	9.4
		14:24	237	3.9	22	19.4	30	10
		14:34	23.6	3.8	21.5	18.9	30	10.8
Backpulsed								
		14:37	24.8	3.9	32.5	29.1	30	7.6
		14:47	24.4	3.8	31	28	30	8.4
		14:57	24.4	3.8	31	28	30	8.8
			Secured CUF					

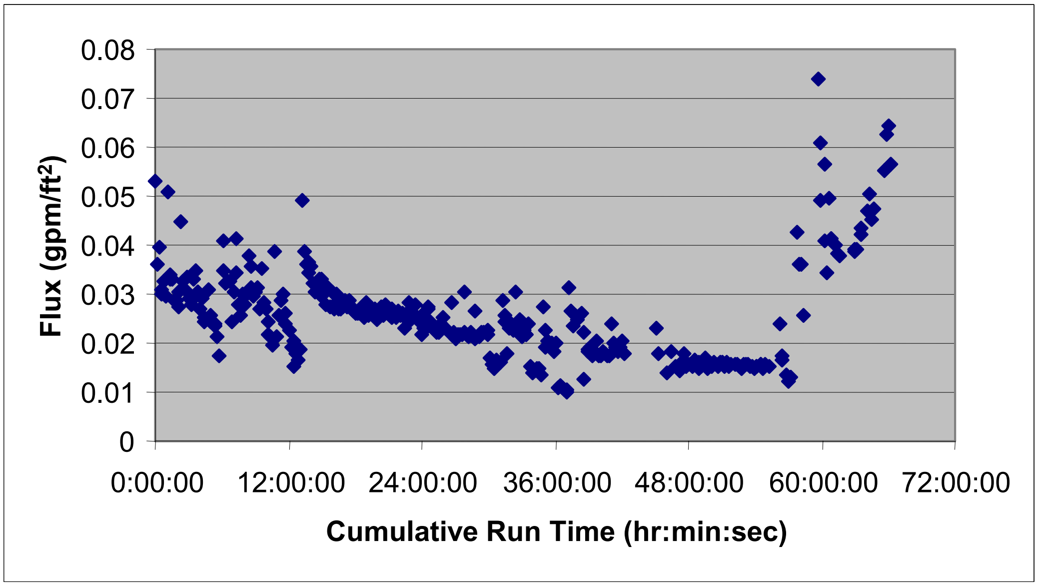

Figure D.1. Summary of AZ-101 Slurry Permeate Flux

Table D.1. Approximate Event Times for Figure D.1

Event	Approximate Cumulative Run Time (hr:min:sec)
Low Solids Matrix	0:00:00-15:24:00
Extended Run	14:24:00-23:55:00
Dewater	24:03:00-26:05:00
High Solids Matrix	26:36:00-42:12:00
Extended Run	45:12:00-55:16:00
Dewater	56:15:00-57:09:00
Wash 1	57:52:00-58:18:00
Wash 2	59:48:00-60:28:00
Leach (excluding 8-hour leach time)	60:37:00-61:36:00
Rinse 1	62:54:00-63:35:00
Rinse 2	64:10:00-64:45:00
Rinse 3	65:40:00-66:17:00

Appendix E

Modeling

Appendix E

Modeling

A cursory review of the literature was made in an attempt to identify a theoretical or semi-theoretical model with the potential to adequately explain the data. The review was restricted to articles since 1995, as models identified in a previous review (Geeting and Reynolds 1996) were generally lacking. For example, gel-layer modeling (Porter 1972) is derived from a single mass balance equation with two unknowns—the permeate flux and the particle concentration distribution over the membrane. As a result, assumptions must be made to evaluate the permeate flux. Zydney's lift model (Zydney and Colton 1986) is based on the gel-layer model and was introduced because the gel-layer model generally under-predicted the permeate flux. However, it too is based on a single mass balance equation with two unknowns.

One model was selected for review, as a comprehensive review of models was beyond the scope of this work. The model selected was a model introduced by Song and Elimelech (1995), based on the hydrodynamics and thermodynamics of particle suspensions. In this model, the flow field and drag force are described by basic theories in hydrodynamics, while many bulk properties of particle suspensions are governed by thermodynamic principles. The model uses both a mass balance and an energy balance to describe the concentration polarization in crossflow filtration. Hard spherical particles were assumed in the derivation of the theory, as well as complete rejection of the particles by the membrane. In addition, the model is developed using a rectangular channel.

The following is a description of the model. Although an attempt was made to provide an independent summary, in some cases, phrases are abstracted directly from the source document.

Cake Formation

The hydrodynamics in a filter cake layer is different from that in a gel layer where the particle concentration is below maximum packing. Song and Elimelech developed a model for both cases, that is, when a filter cake is present and when it is not. The criterion for cake formation is given as follows:

$$N_f := 4 \cdot \pi \cdot a_p^3 \cdot \frac{\Delta P_p}{3 \cdot k \cdot T}$$

where,

a_p = particle radius

ΔP_p = pressure drop across the concentration polarization layer (for an approximation substitute applied hydraulic pressure, ΔP)

k = Boltzmann constant, 1.3803×10^{-23} J K^{-1}

T = absolute Temperature.

N_f is dimensionless and is introduced as the filtration number; it can be considered as the ratio of the energy needed to bring a particle from the membrane surface to the bulk suspension to the thermal energy of the particle. When N_f is greater than 15, a cake layer is expected to form on the membrane surface for a monodisperse suspension of rigid spherical particles. In our case, $N_f >> 15$, and is clearly in the range for which cake formation is expected.

The basic steps in the development of the model are to determine the distribution of retained particles in the polarization layer. The concentration in the cake layer is assumed to correspond to the maximum packing of the retained particles. The thickness of the cake layer is determined by applying Stokes' law combined with Happel's cell model, which accounts for the effect of neighboring particles. The conservation of particle-flux along the filter channel and a derived relationship between permeate velocity and cake thickness lead to a crossflow filtration equation, with the average permeate velocity determined as follows:

$$V := \left[\left(\frac{3}{2}\right)^{\frac{2}{3}} D^{\frac{2}{3}} \cdot \gamma^{\frac{1}{3}} \cdot \left(1 + N_c\right)^{\frac{1}{3}} \cdot L^{\frac{-1}{3}}\right] \cdot \left[\frac{\Delta P_p}{\left(A_{smaxstar} \cdot k \cdot T \cdot C_o\right)}\right]^{\frac{1}{3}}$$

where,

V = average permeate velocity (flux)

D = particle diffusion coefficient; $D = kT/6\pi\mu a_p$

μ = fluid viscosity

γ = fluid shear rate

L = length of filter channel

N_c = cake forming factor

$$N_c := \frac{\left(\Delta P_c \cdot A_{smaxstar}\right)}{\Delta P_p \cdot A_{smax}}$$

where subscripts ΔP_c and ΔP_p are the pressure drops across the cake layer and polarization layer, respectively.

A_{smax} and $A_{smaxstar}$ are correction functions for Stokes' law based on Happel's cell model evaluated at θ corresponding to the maximum packing of the retained particles, and at the onset of cake formation, respectively.

$$A_s := \frac{\left(1 + \frac{2\cdot\theta^5}{3}\right)}{\left(1 - \frac{3}{2}\cdot\theta + \frac{3}{2}\cdot\theta^5 - \theta^6\right)}$$

where, θ is defined by $\theta=(1-\varepsilon)^{1/3}$ and ε is the cake or particle layer porosity.

The crossflow filtration equation, as well as the similar equation corresponding applied when N_f is small (not shown), establishes the filtration curve shown in Figure E.1. The first region corresponds to the filter membrane being the dominant resistance to filtration, and the permeate flux increases proportionally with pressure. The second region corresponds to the polarization layer being the dominant resistance. In this region, the permeate flux initially increases as the one-third power of pressure, but the rate of increase declines in the final stage due to a canceling effect from a pressure dependent variable. In the final region, the filter cake is the dominant resistance, and the permeate flux increases as the one-third power of the pressure.

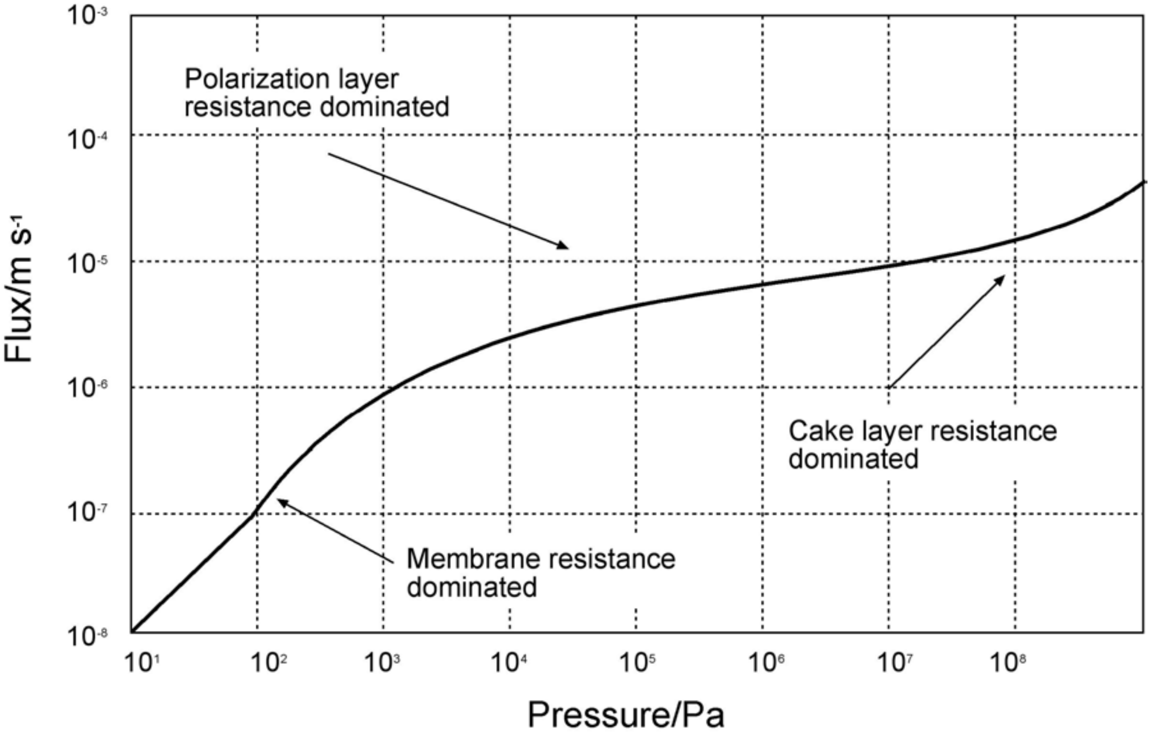

Figure E.1. Complete Filtration Curve (adapted from Song and Elimelech 1995)

The model indicates that for AZ-101 sludge, at both the low and high solids matrices, we were operating in the region where cake layer resistance should dominate.

Influence of Input Parameters on Predicted Permeate Velocity

The model predicts the permeate flux to be proportional to input parameters as follows:

Permeate flux proportional to:	Parameter
	$(\text{transmembrane pressure})^{1/3}$
	$(\text{axial velocity})^{1/3}$
	$(\text{particle diameter})^{1/3}$
	$(\text{viscosity})^{-2/3}$
	$(\text{volume fraction solids})^{-1/3}$

Table E.1 compares the model parameter exponents with exponents calculated from the empirical data from AZ-101 sludge. For this comparison, an exponent was calculated for each parameter without considering whether this was best mathematical fit. Comparing the empirical data to the model indicates the data to be very difficult for one model to capture. For example, note that the 7.6 wt% solids slurry depended on TMP, while the 17.9 wt% slurry did not. The opposite is true for axial velocity. The model predicts the correct tendency for all of the parameters. That is, if the model predicts an increase or decrease in permeate flux with a parameter, the opposite dependency was not observed. Clearly, modeling the TMP and axial velocity are difficult when such changes are observed.

A comparison of the permeate flux predicted with the actual data at the center of the matrix is provided in Table E.2. The model predicted permeate flux was approximately one-half the measured flux. Of the

Table E.1. Comparison of Model and Empirical Parameter Exponents

Parameter	Model Exponent	7.6 wt % solids Exponent	17.9 wt% solids Exponent	Dewatering Exponent
Transmembrane Pressure	$(\text{TMP})^{1/3}$	$(\text{TMP})^{1/2}$	$(\text{TMP})^{0}$	ND
Axial Velocity	$(\text{Vel})^{1/3}$	$(\text{Vel})^{0}$	$(\text{Vel})^{1}$	ND
Volume fraction Solids	$(\text{Solids})^{-1/3}$	ND	ND	$(\text{Solids})^{-0.6}$
Viscosity	$(\text{Viscosity})^{-2/3}$	ND	ND	$(\text{Viscosity})^{-1}$
Particle Size	$(\text{PSD})^{1/3}$	ND	ND	ND
ND = Not Determined				

Table E.2. Comparison of Predicted and Actual Permeate Flux

Slurry	Flux Predicted by Model[(a)] (gpm/ft^2)	Actual Flux (gpm/ft^2)
7.6 wt% slurry	0.013	0.031
17.9 wt% slurry	0.0095	0.021
(a) Model parameters were as follows: particle radius 1.01E-6 m; viscosity 4.1 cP; axial velocity 11 ft/s; transmembrane pressure 40 psid; volume fraction solids at 7.6 wt% solids = 2.5%; volume fraction solids at 17.9 wt% solids=6.3%.		

model parameters, the only one not measured at the time of testing was the viscosity. The viscosity was measured on AZ-101 supernatant to be ~2 cP at 65°C.(1) This was corrected to 4.1 cP at 25°C assuming that the viscosity of the supernatant behaved proportionally to water as a function of temperature. The input viscosity must be reduced to approximately 1.2 cP to get the predicted flux from the model to match the actual. At this reduced viscosity, the predicted flux for the 7.6 and 17.9 wt% slurries are 0.030 and 0.022 gpm/ft^2, respectively. This indicates that with the model properly normalized, it predicts the actual flux change with solids loading well over the range indicated. If another parameter were used for the normalization, one could expect a fairly good predictive capability for changes in viscosity. The model predicts a -2/3 exponential dependency with viscosity, while the empirical data suggest an exponent of -1. Over the range of interest, for example, 1 to 5 cP, if the model were normalized (i.e., adjusted to fit the data) at 1 cP, its predicted flux would be approximately 70% high at 5 cP, assuming that the empirical data held over the range.

References

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Appendix F

Analytical Results

Project / WP#:	**42365/ W60513**
ASR#:	**6284.01**
Client:	**J. Geeting**
Total Samples:	**4 (slurry)**

RPL#:	**02-0829**	**02-0832**
Client ID:	**AZ-0**	**AZ-6**
Sample Preparation: **PNL-ALO-116 (nominally 0.2g/100mL)**		
Analytes of Interest: **Specified in Table 1 and Table 2 of ASR**		

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

Analyst: **D.R. Sanders**

Analysis Date (File): **01-31-02 (A0763)**

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)**

MW Urie 4/5/02
Reviewed by

Renee Russell 4/5/02
Concur

Four slurry samples (RPL# 02-0829 through 02-0832) were submitted under Analytical Service Request (ASR) 6284.01. The samples were dried per the ASR instructions, then prepared for analysis by sodium peroxide fusion per PNL-ALO-116. The samples were digested using nominally 0.2 g of dried sample and diluting to a final volume of 100 mL. The results from the sodium peroxide fusion preparation are used primarily to obtain a good K and Ni results and to compare the results obtained from the potassium hydroxide fusion preparation.

The inductively coupled plasma atomic emission spectrometer (ICPAES) analytes of interest (AOI) were specified on the ASR for each of the samples; ASR Table 2 applies to sample AZ-6 (02-0832) and ASR Table 1 applies to the remaining samples. All other analytes that were not requested are reported, but have not been fully evaluated for QC performance. A summary of the ICPAES analysis of the samples, including QC performance, is given in the attached ICPAES Data Report (2 pages). The ICPAES results are reported in µg/g dried solids and have been adjusted for all preparative and analysis processing factors.

Two processing blanks, a laboratory control sample (SRM 2710 – Montana Soil), and a duplicate of sample AZ-6 were prepared and analyzed with the samples. The LCS was prepared by using 0.19 g SRM 2710 and diluting to a final volume of 100 mL. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the laboratory's QA plan; no additional QC requirements were specified by the ASR. Quality control standards results met tolerance requirements for the specific AOIs except as noted below.

Process Blanks:

Low concentrations of Ag, Al, Ca, Ce, Cr, Fe, Li, Ni, Si, Sr, and U were detected in the process blanks. However, the blank concentrations for these AOIs were within the tolerance limit of ≤EQL.

Duplicate RPD (Relative Percent Difference):

Sample AZ-6 (02-0832) was prepared in duplicate. All AOIs measured above the EQL were within the tolerance limit of <20% RPD, except Ag and Ce. Silver had a RPD of 27% and Ce had a RPD of 47%, indicating that either these analytes were not homogeneous throughout the solids sample or there was poor dissolution of these analytes by the fusion method.

Laboratory Control Standard (LCS):

For the AOIs present in the LCS material (SRM 2710 – Montana Soil), the recoveries were within tolerance of 80% to 120% except Ni. The spike concentration of Ni was less than 20% of the sample concentration and the recovery results are considered meaningless. For Ni, the serial dilution results are used to evaluate potential matrix interferences.

Matrix Spiked Sample:

No matrix spike sample was prepared.

Post-Spiked Samples (Spike A Elements):

All post-spiked AOIs were recovered within tolerance of 75% to 125% except Ag, Al, Cd, Fe, Mn, Ni, and Sr. The low Ag recovery indicates a significant matrix or dissolution problem; most likely the concentration of chloride was either too high or too low to maintain the Ag in solution. The spike concentration of the other analytes was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the serial dilution results are used to evaluate potential matrix interferences.

Post-Spiked Samples (Spike B Elements):

All post-spiked AOIs were recovered within tolerance of 75% to 125% except Ce and La. The Ce recovery of <0% indicates a significant matrix issue and the reported results may be biased low. For La, the post spike analysis uses a general spiking solution intended to be usable on the majority of samples analyzed by ICPAES. However, for the sample analyzed, the spike concentration for La was less than 20% of the sample concentration and the recovery results are considered meaningless. For La, the serial dilution results are used to evaluate potential matrix interferences.

Five fold serial dilution:

All AOIs measured above the EQL were within the tolerance limit of ±10% after correcting for dilution.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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).
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

	Multiplier=	981.7	981.7	1015.7	1079.9	885.3	956.9	992.1
	RPL/LAB #=	**02-00832-B1-Zr @2**	**02-00832-B2-Zr @2**	**02-00829-Zr @2**	**02-00830-Zr @2**	**02-00831-Zr @2**	**02-00832-Zr @2**	**02-00832-Zr-DUP @2**
Det. Limit	*Client ID=*	*process blank 1*	*process blank 2*	*AZ-0*	*AZ-2*	*AZ-4*	*AZ-6*	*AZ-6-Dup*
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	[28]	724	381	405	930	706
0.060	Al	[190]	[150]	101,000	192,000	70,100	103,000	102,000
0.250	As	--	--	--	--	--	--	--
0.050	B	--	--	1,070	[56]	5,230	[96]	[72]
0.010	Ba	--	--	419	798	883	1,490	1,540
0.010	Be	--	--	--	[16]	[14]	[24]	[24]
0.100	Bi (a)	--	--	--	[120]	[98]	--	--
0.250	Ca	[1,800]	[1,700]	3,810	5,940	5,930	8,230	9,150
0.015	Cd	--	--	3,940	7,870	8,510	13,900	14,000
0.200	Ce	--	[560]	8,320	[1,400]	[1,400]	5,800	3,600
0.050	Co	--	--	--	[67]	[71]	[120]	[120]
0.020	Cr	[23]	[32]	1,680	2,450	1,500	2,620	2,490
0.025	Cu	--	--	[54]	[210]	254	591	582
0.050	Dy (a)	--	--	[58]	--	--	[66]	[63]
0.100	Eu (a)	--	--	--	--	--	--	--
0.025	Fe	[140]	[230]	55,300	108,000	118,000	193,000	200,000
2.000	K	--	--	[7,600]	[3,900]	[2,600]	[2,000]	--
0.050	La	--	--	1,680	3,170	3,480	5,190	5,570
0.030	Li	--	[30]	[140]	[230]	[130]	[150]	[150]
0.100	Mg	--	--	--	[710]	[760]	[770]	[950]
0.050	Mn	--	--	1,470	2,810	3,060	5,160	5,350
0.050	Mo	--	--	[170]	[94]	[61]	[65]	[66]
0.150	Na	na	na	na	na	na	na	na
0.100	Nd (a)	--	--	1,240	2,410	2,600	4,010	4,460
0.030	Ni	[30]	[35]	2,760	5,390	5,880	9,850	10,200
0.100	P (a)	--	--	2,230	3,260	2,860	4,560	4,880
0.100	Pb	--	--	[650]	1,150	1,060	1,780	1,880
0.750	Pd (b)	--	--	[2,400]	[1,100]	[1,100]	[2,600]	[2,200]
0.300	Rh (b)	--	--	[480]	--	[300]	[570]	[500]
1.100	Ru (b)	--	--	--	--	--	[1,500]	[1,500]
0.500	Sb (a)	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--
0.500	Si	[740]	--	[4,000]	7,850	47,800	12,800	13,300
1.500	Sn (a)	--	--	[1,600]	[2,300]	[2,100]	[3,600]	[3,600]
0.015	Sr	[51]	[42]	974	1,920	2,120	3,220	3,360
1.500	Te	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--
0.025	Ti	--	--	[53]	[95]	[100]	[160]	[160]
0.500	Tl	--	--	--	--	--	--	--
2.000	U	--	[2,500]	22,400	[8,800]	[9,300]	23,000	[19,000]
0.050	V	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--
0.050	Y	--	--	[110]	[210]	[230]	[360]	[390]
0.050	Zn	--	--	[94]	[160]	[160]	[270]	[280]
0.050	Zr	na	na	na	na	na	na	na

(a) AOI for AZ-6 only (b) Not an AOI

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 1/31/02

Criteria>	<20%	80% - 120%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-00832 & 02-00832-D (@2)	LCS/BS (@2)	LCS/BS (@10)	MS (none)	02-00832 + Post Spike A	02-00832 + Post Spike B	02-00832 @2/@10 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Ag	27				39		
Al	0.7	98			nr		-2.3
As					96		
B					97		
Ba	3.6	92			93		3.5
Be					93		
Bi (a)					96		
Ca	11	93			101		
Cd	0.6				nr		-0.9
Ce	47					-24	
Co					99		
Cr	5.4				75		-4.7
Cu	1.5	84			91		
Dy (a)						99	
Eu (a)						102	
Fe	3.7	97			nr		7.0
K					99		
La	7.1					nr	6.8
Li					98		
Mg		100			102		
Mn	3.6	101			nr		4.3
Mo					98		
Na	na	na			na		na
Nd (a)	11					124	
Ni	3.4	nr			nr		4.5
P (a)	6.7				103		
Pb	5.4	108			115		
Pd (b)						44	
Rh (b)						95	
Ru (b)						99	
Sb (a)					103		
Se					105		
Si	4.0	over range	102		107		
Sn (a)						85	
Sr	4.3	91			nr		3.8
Te						108	
Th						96	
Ti		90			91		
Tl					98		
U	20					83	
V					93		
W							
Y					95		
Zn		98			100		
Zr	na	na			na		na

(a) AOI for AZ-6 only (b) Not an AOI

Shaded results exceed acceptance criteria

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

n/a = not applicable; KOH flux and Ni crucible used for preparing samples.

Battelle PNNL/RSE/Inorganic Analysis ... ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	**42365/ W60513**
ASR#:	**6284**
Client:	**J. Geeting**
Total Samples:	**4 (slurry)**

RPL#:	**02-0829**	**02-0832**
Client ID:	**AZ-0**	**AZ-6**
Sample Preparation: **PNL-ALO-115 (nominally 0.2g/100mL)**		
Analytes of Interest: **Specified in Table 1 and Table 2 of ASR**		

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

Analyst: **D.R. Sanders**

Analysis Date (File): **01-09-02 (A0751)**

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)**

MW Thu 04/05/02
Reviewed by

Reneé Russell 4/4/02
Concur

Four slurry samples (RPL# 02-0829 through 02-0832) were submitted under Analytical Service Request (ASR) 6284. The samples were dried per the ASR instructions, then prepared for analysis by potassium hydroxide fusion per PNL-ALO-115. The samples were digested using nominally 0.2 g of dried sample and diluting to a final volume of 100 mL.

The inductively coupled plasma atomic emission spectrometer (ICPAES) analytes of interest (AOI) were specified on the ASR for each of the samples; ASR Table 2 applies to sample AZ-6 (02-0832) and ASR Table 1 applies to the remaining samples. All other analytes that were not requested are reported, but have not been fully evaluated for QC performance. A summary of the ICPAES analysis of the samples, including QC performance, is given in the attached ICPAES Data Report (2 pages). The ICPAES results are reported in μg/g dried solids and have been adjusted for all preparative and analysis processing factors.

Two processing blanks, a laboratory control sample (SRM 2710 – Montana Soil), and a duplicate of sample AZ-6 were prepared and analyzed with the samples. The LCS was prepared by using 0.2 g SRM 2710 and diluting to a final volume of 100 mL. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the laboratory's QA plan; no additional QC requirements were specified by the ASR. Quality control standards results met tolerance requirements for the specific AOIs except as noted below.

Process Blanks:

Aluminum, Cr, Fe, Mn, Na, Pb, and U were detected in the process blanks. However, the blank concentrations for these AOIs were within the tolerance limit of ≤EQL or ≤5% of the concentration in the sample, except Na and Cr. The Cr blank concentration ranged from 11% to 18% of the sample concentrations. The Na blank concentration slightly exceeded the 5% tolerance limit for sample AZ-2 (02-0830).

Duplicate RPD (Relative Percent Difference):

Sample AZ-6 (02-0832) was prepared in duplicate. All AOIs measured above the EQL were within the tolerance limit of <20% RPD, except Ag. Silver had a RPD of 33%, indicating either Ag was not homogeneous throughout the solids sample or there was poor dissolution of Ag by the fusion method.

Laboratory Control Standard (LCS):

For the AOIs present in the LCS material (SRM 2710 – Montana Soil), the recoveries were within tolerance of 80% to 120%; except Cu and Si. The low Si recovery (73%) appears to be from incomplete dissolution of the silicon in the LCS. The low Cu recovery (75%) is most likely due to actual concentration of the Cu measured (i.e., concentration was <EQL and has high variability).

Matrix Spiked Sample:

No matrix spike sample was prepared.

Post-Spiked Samples (Spike A Elements):

All post-spiked AOIs were recovered within tolerance of 75% to 125% except Al, Cd, Fe, Mn, and Zr. The post spike analysis uses a general spiking solution intended to be usable on the majority of samples analyzed by ICPAES. However, for the sample analyzed, the spike concentration for Al, Cd, Fe, Mn, and Zr was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the serial dilution results are used to evaluate potential matrix interferences.

Post-Spiked Samples (Spike A Elements):

All post-spiked AOIs were recovered within tolerance of 75% to 125% except La. The post spike analysis uses a general spiking solution intended to be usable on the majority of samples analyzed by ICPAES. However, for the sample analyzed, the spike concentration for La was less than 20% of the sample concentration and the recovery results are considered meaningless. For La, the serial dilution results are used to evaluate potential matrix interferences

Five fold serial dilution:

All AOIs measured above the EQL were within the tolerance limit of ±10% after correcting for dilution.

Other QC Samples:

The K in the High End Calibration Range Check Standard was outside the tolerance limit of $\leq$5% at 8.9%; all other AOIs were within the tolerance limit.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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).
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis.... ICP~~ASE~~ AES Report
KO 4/10/02

	Multiplier=	954.1	954.1	1041.7	943.0	875.3	1011.1	918.7	4593.5
	RPL/LAB #=	02-00832-B1-Ni @2	02-00832-B2-Ni @2	02-00829-Ni @2	02-00830-Ni @2	02-00831-Ni @2	02-00832-DUP-Ni @2	02-00832-Ni @2	02-00832-Ni @10
	Client ID=	process blank	process blank	AZ-0	AZ-2	AZ-4	AZ-6-Dup	AZ-6	
Det. Limit	Run Date=	1/9/2002	1/9/2002	1/9/2002	1/9/2002	1/9/2002	1/9/2002	1/9/2002	1/9/2002
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	[190]	376	322	532	381	[420]
0.060	Al	[150]	[320]	89,000	180,000	89,600	96,000	99,300	
0.250	As	--	--	--	--	--	--	--	
0.050	B	--	--	1,060	[130]	2,180	[140]	[55]	
0.010	Ba	--	--	344	654	569	1,450	1,560	
0.010	Be	--	--	--	[15]	[13]	[27]	[28]	
0.100	Bi (a)	--	--	--	--	--	[150]	[150]	
0.250	Ca	--	--	[1,800]	3,660	3,100	7,340	7,670	
0.015	Cd	--	--	3,630	7,490	6,460	14,400	15,700	
0.200	Ce	--	--	[540]	[930]	[810]	[1,900]	2,020	
0.050	Co	--	--	--	[81]	[66]	[130]	[140]	
0.020	Cr	--	258	1,430	2,020	1,640	2,310	2,290	
0.025	Cu	--	--	--	[200]	[180]	542	618	
0.050	Dy (a)	--	--	--	--	--	--	--	
0.100	Eu (a)	--	--	--	--	--	--	--	
0.025	Fe	[47]	1,230	50,200	104,000	91,200	199,000	over range	218,000
2.000	K	na	na	na	na	na	na	na	
0.050	La	--	--	1,440	2,760	2,400	6,020	6,450	
0.030	Li	--	--	[90]	[170]	[86]	[110]	[110]	
0.100	Mg	--	--	[410]	[750]	[660]	1,480	1,600	
0.050	Mn	[52]	[92]	1,420	2,890	2,390	5,410	5,680	
0.050	Mo	--	--	[150]	[86]	[71]	[68]	[65]	
0.150	Na	2,410	2,300	130,000	45,500	164,000	60,200	53,600	
0.100	Nd (a)	[120]	[120]	1,130	2,120	1,850	4,320	4,610	
0.030	Ni	na	na	na	na	na	na	na	
0.100	P (a)	--	--	1,200	[320]	1,490	4,670	3,910	
0.100	Pb	[120]	[110]	[530]	1,020	[870]	1,680	1,800	
0.750	Pd (b)	--	--	[800]	[1,300]	[1,000]	[2,100]	[2,300]	
0.300	Rh (b)	--	--	--	[320]	[280]	[490]	[490]	
1.100	Ru (b)	--	--	--	--	--	[1,600]	[1,800]	
0.500	Sb (a)	--	--	--	--	--	--	--	
0.250	Se	--	--	--	--	--	--	--	
0.500	Si	--	--	5,710	7,030	12,200	13,500	14,100	
1.500	Sn (a)	--	--	--	[1,600]	--	[2,100]	[2,800]	
0.015	Sr	--	--	833	1,630	1,410	3,470	3,690	
1.500	Te	--	--	--	--	--	--	--	
1.000	Th	--	--	--	--	--	--	--	
0.025	Ti	--	--	[54]	[97]	[81]	[210]	[180]	
0.500	Tl	--	--	--	--	--	--	--	
2.000	U	[2,000]	[1,900]	[4,500]	[7,200]	[6,000]	[12,000]	[13,000]	
0.050	V	--	--	--	--	--	--	--	
2.000	W	--	--	--	--	--	--	--	
0.050	Y	--	--	[95]	[190]	[130]	[380]	[410]	
0.050	Zn	--	--	[77]	[160]	[130]	[270]	[290]	
0.050	Zr	--	--	14,300	24,300	21,900	64,400	65,700	

(a) AOI for AZ-6 only (b) Not an AOI

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 01/09/2002

Criteria>	<20%	<20%	80% - 120%	75%-125%	75%-125%	< +/-10%
QC ID=	02-00832 & 02-00832-D (@2)	02-00832 & 02-00832-D (@10)	02-00832 LCS (@2)	02-00832 + Post Spike A (@2)	02-00832 + Post Spike B (@2)	02-00832 @2/@10 Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Diff
Ag	33			90		
Al	3.5		89	nr		-1.9
As				101		
B				97		
Ba	6.8		81	111		1.1
Be				97		
Bi (a)				94		
Ca	4.3		91	98		
Cd	8.7			nr		-1.7
Ce					96	
Co				98		
Cr	0.6			107		0.1
Cu	13		75	98		
Dy (a)					94	
Eu (a)					93	
Fe	over range	2.9	90	nr		
K	na		na	na		na
La	6.9				nr	0.4
Li				94		
Mg	7.4		96	102		
Mn	4.9		97	nr		0.3
Mo				96		
Na	12		81	100		2.9
Nd (a)	6.6		nr		101	5.2
Ni	na		na	na		na
P (a)	18		99	108		
Pb	6.9		92	103		
Pd (b)					90	
Rh (b)					94	
Ru (b)					85	
Sb (a)				102		
Se				106		
Si	3.9		73	108		
Sn (a)					64	
Sr	6.1		86	nr		-0.4
Te					101	
Th					93	
Ti			82	92		
Tl				92		
U					97	
V				94		
W						
Y				95		
Zn			93	100		
Zr	1.9			nr		1.1

(a) AOI for AZ-6 only (b) Not an AOI

Shaded results exceed acceptance criteria

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

n/a = not applicable; KOH flux and Ni crucible used for preparing samples.

Battelle PNNL/RSE/Inorganic Analysis … ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#:	**42365/ W60513**
ASR#:	**6284**
Client:	**J. Geeting**
Total Samples:	**7**

RPL#:	02-0822	02-0828
Client ID:	**AZ-A**	**AZ-M**
Sample Preparation: **PNL-ALO-128 (1mL/25mL)**		
Analytes of Interest: **Specified in Table 1 of ASR**		

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

Analyst: **D.R. Sanders**

Analysis Date (File): **01-08-02 (A0750)**

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)**

04-05-02
Reviewed by

Renee Russell 4/4/02
Concur

4/4/02

Seven liquid samples (RPL# 02-0822 through 02-0828) submitted under Analytical Service Request (ASR) 6284 were prepared by acid digestion per PNL-ALO-128. The samples were digested using 1 mL of sample and diluting to a final volume of 25 mL.

The inductively coupled plasma atomic emission spectrometer (ICPAES) analytes of interest (AOI) were specified on the ASR (Table 1). All other analytes that were not requested are reported, but have not been fully evaluated for QC performance. A summary of the ICPAES analysis of the samples, including QC performance, is given in the attached ICPAES Data Report (3 pages). The ICPAES results are reported in µg/mL and have been adjusted for all preparative and analysis processing factors.

A processing blank, blank spike (BS), matrix-spike (MS), and duplicate (sample AZ-M) were prepared and analyzed with the samples. The BS and MS were prepared using approximately 2.5 mL of multi-element spiking solution "INT-QC-MCVA-1B" per 25 mL of final digestate volume. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan or the additional QC requirement specified in Table 5 of the ASR. Quality control standards results met tolerance requirements for the specific AOIs except as noted below.

Process Blanks:

Concentrations of the AOIs measured in the process blank were all within the tolerance limit of ≤EQL or ≤5% of the concentration in the sample, except Si and B. The level of Si in the process blank represents from 60% to 95% of the concentration of Si measured in the samples. The level of B in the process blank was essentially equivalent to that measured in the samples.

Duplicate RPD (Relative Percent Difference):

The RPDs for all AOIs measured above the EQL were within the tolerance limit of <20%.

Blank Spike:

Blank spike recoveries for the AOIs measured above the EQL were within tolerance of 80% to 120%. It should be noted that only about 75% of the AOIs were included in the blank spike.

Matrix Spiked Sample:

The matrix spike recoveries for the AOIs measured above the EQL were within tolerance of 75% to 125% except Al and Na. Sodium and Al recovery were not calculated since the Na and Al spike concentrations were less than 20% of the sample concentration.

Post-Spiked Samples (Spike A Elements):

Post spiking was performed on sample AZ-M (02-0828). All post spiked AOIs in the sample tested were recovered within the tolerance limit of 75% to 125%.

Post-Spiked Samples (Spike B Elements):

Post spiking was performed on sample AZ-M (02-0828). All post spiked AOIs in the sample tested were recovered within the tolerance limit of 75% to 125%.

Five fold serial dilution:

Serial dilution was performed on sample AZ-A (02-0822). All AOIs measured above the EQL were within tolerance limit of ±10% after correcting for dilution, except K. At 12% percent difference (%Diff), K slightly exceeded the tolerance limit; most likely due to the high sample concentration and the failure of the High End Calibration Range Check Standard.

Other QC Samples:

The K in the High End Calibration Range Check Standard was outside the tolerance limit of $\leq$5% at 7.5%. All other analytes of interest were within the tolerance limit.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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).
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

co4/11/02

Battelle PNNL/RSE/Inorganic Analysis.... ICP~~ASE~~ Report
AES

	Run Date=	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002
	Multiplier=	25.2	25.8	129.0	25.5	127.4	25.0	124.9	25.8
	RPL/LAB #=	02-00822-B	02-00822	02-00822 @5	02-00822-DUP	02-00822-DUP @5	02-00823	02-00823 @5	02-00824
Det. Limit	Client ID=	process blank	AZ-A		AZ-A-DUP		AZ-C		AZ-E
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--		--		--		--
0.060	Al	[9.5]	5,350		5,300		2,350		1,210
0.250	As	--	[9.8]		[9.7]		--		--
0.050	B	79.4	68.9		68.7		75.9		73.2
0.010	Ba	[0.29]	--		--		--		[0.75]
0.010	Be	--	--		--		--		--
0.250	Ca	--	--		[7.2]		[10]		--
0.015	Cd	--	[0.42]		[0.42]		--		--
0.200	Ce	--	--		--		--		--
0.050	Co	--	--		--		--		--
0.020	Cr	--	627		621		217		102
0.025	Cu	--	--		--		--		--
0.025	Fe	[0.95]	41.2		[1.5]		[1.2]		[0.82]
2.000	K	--	4,060		4,020		1,680		866
0.050	La	--	--		--		--		--
0.030	Li	--	--		--		--		--
0.100	Mg	--	--		--		--		--
0.050	Mn	--	--		--		--		--
0.050	Mo	--	87.0		86.6		39.4		20.1
0.100	Nd	--	--		--		--		--
0.150	Na	122	over range	103,000	over range	102,000	over range	42,300	20,800
0.030	Ni	[1.4]	--		--		--		--
0.100	Pb	--	[5.3]		[5.5]		[2.8]		--
0.500	Sb	--	--		--		--		--
0.250	Se	--	--		--		--		--
0.500	Si	134	223		211		188		200
0.015	Sr	--	--		--		--		--
1.500	Te	--	--		--		--		--
1.000	Th	--	--		--		--		--
0.025	Ti	--	--		--		--		--
0.500	Tl	--	--		--		--		--
2.000	U	--	--		--		--		--
0.050	V	--	[1.6]		[1.6]		--		--
2.000	W	--	[55]		[55]		--		--
0.050	Y	--	--		--		--		--
0.050	Zn	--	--		--		--		[4.2]
0.050	Zr	--	[2.2]		[2.2]		--		--
Other Analytes									
0.100	Bi	--	--		--		--		--
0.050	Dy	--	--		--		--		--
0.100	Eu	--	--		--		--		--
0.100	P	--	495		493		193		83.4
0.750	Pd	--	[22]		[22]		--		--
0.300	Rh	--	--		--		--		--
1.100	Ru	--	--		--		--		--
1.500	Sn	--	[50]		[50]		--		--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%. 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier (top of each column).

	Run Date=	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002	1/8/2002
	Multiplier=	25.3	126.4	26.1	130.4	25.8	26.0
	RPL/LAB #=	02-00825	02-00825 @5	02-00826	02-00826 @5	02-00827	02-00828
Det. Limit	Client ID=	AZ-G		AZ-I		AZ-K	AZ-M
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--		--		--	--
0.060	Al	14,600		7,900		3,990	2,060
0.250	As	--		--		--	--
0.050	B	61.5		78.3		54.7	71.9
0.010	Ba	--		--		--	[0.27]
0.010	Be	[0.26]		--		--	--
0.250	Ca	--		--		--	--
0.015	Cd	[1.4]		--		--	--
0.200	Ce	--		--		--	--
0.050	Co	--		--		--	--
0.020	Cr	135		70.0		36.0	19.1
0.025	Cu	--		--		--	--
0.025	Fe	[2.7]		[1.4]		[1.3]	[1.7]
2.000	K	[500]		[230]		[110]	--
0.050	La	--		--		--	--
0.030	Li	13.4		[7.7]		[4.1]	[2.3]
0.100	Mg	--		--		--	--
0.050	Mn	--		--		--	--
0.050	Mo	[11]		[5.4]		[2.8]	[1.5]
0.100	Nd	--		--		--	--
0.150	Na	over range	60,600	over range	33,700	16,000	8,640
0.030	Ni	--		--		--	--
0.100	Pb	[9.6]		[4.7]		--	--
0.500	Sb	--		--		--	--
0.250	Se	--		--		--	--
0.500	Si	188		180		142	145
0.015	Sr	--		--		--	--
1.500	Te	--		--		--	--
1.000	Th	--		--		--	--
0.025	Ti	--		--		--	--
0.500	Tl	--		--		--	--
2.000	U	--		--		--	--
0.050	V	--		--		--	--
2.000	W	--		--		--	--
0.050	Y	--		--		--	--
0.050	Zn	[3.0]		[1.5]		--	[1.4]
0.050	Zr	[1.6]		--		--	--
Other Analytes							
0.100	Bi	--		--		--	--
0.050	Dy	--		--		--	--
0.100	Eu	--		--		--	--
0.100	P	81.4		37.9		[17]	[8.2]
0.750	Pd	--		--		--	--
0.300	Rh	--		--		--	--
1.100	Ru	--		--		--	--
1.500	Sn	[49]		--		--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%. 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

ko4/10/02

QC Performance 01/08/2002

Criteria>	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%
QC ID=	02-00822 & 02-00822-D	02-00822 & 02-00822-D (@5)	LCS/BS	02-00828 & 02-00828-MS	02-00828 + Post Spike A	02-0xxxx + Post Spike B	02-00822 @1/@5 Serial Dil	02-00822 @5/@25 Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff
Ag			98	93	94			
Al	1.1		99	nr	105		3.5	
As				101	104			
B	0.3		71	43	103		3.3	
Ba			96	92	100			
Be					99			
Ca			101	97	103			
Cd			103	99	103			
Ce						98		
Co			103	98	104			
Cr	0.8		101	96	103		4.4	
Cu			101	95	101			
Fe			101	96	103		4.0	
K	1.0		104	102	107		12	
La						99		
Li			104	99	109			
Mg			104	99	107			
Mn					106			
Mo	0.5				105		3.7	
Nd						99		
Na	over range	0.8	99	nr	111		over range	5.1
Ni			102	99	105			
Pb			107	108	108			
Sb					102			
Se					105			
Si	5.6				112		1.6	
Sr			99	97	103			
Te						103		
Th						100		
Ti					97			
Tl					103			
U						98		
V			96	92	97			
W								
Y			98	95	99			
Zn			104	96	106			
Zr					102			
Other Analytes								
Bi			98	94	100			
Dy						101		
Eu						101		
P	0.4		102	98	104		-0.6	
Pd						92		
Rh						95		
Ru								
Sn						93		

Shaded results exceed acceptance criteria

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

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

PO Box 999, Richland, Washington 99352

Client:	J. Geeting	Charge Code/Project:	W60513 / 42365
ASR Number:	6284	Sample Receipt Date:	11/21/01
Sample Number:	02-0822 to 02-0832	Preparation Date:	01/11/02 (slurries)
Analyst:	MJ Steele	Analysis Date:	01/11-12/02 (slurries) 05/01-03/02 (liquids)

REVISION 1: REPLACE ICPAES WITH IC IN PARAGRAPH 2

Preparation Procedure: ALO-103 for 02-0829 to 02-0832
Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Seven AZ-101 liquid samples (02-0822 through 02-0828) and four AZ-101 slurry samples (02-0829 through 02-032) were received under Analytical Service Request (ASR) 6284. The four slurry samples were subjected to a water leach per PNL-ALO-103 by leaching approximately 1 g slurry in 15 mL of distilled, deionized water. The leaching process was performed in the Shielded Analytical Laboratory hot cells and aliquots of the leachates, along with the aliquots of the seven liquid samples, were transferred to the IC analysis workstation for anion analysis. The samples required additional laboratory dilutions from 10x to 2000x in order to ensure that the anions were measured within the calibration range and that the IC column was not overloaded during the analysis. The stated estimated quantitation levels (EQL) are based on the lowest calibration standard adjusted for the dilutions used for reporting the results.

Anions of interest and minimum reportable quantities (MRQ) were specified in the ASR (i.e., ASR 6284 Table 1 and Table 2). Anions other than those identified in the ASR are included in the results for information only, since these anions have not been fully evaluated for QC performance. A summary of the IC analysis results for the samples, as well as a summary of the QC performance, is given in Table 1 and Table 2.

Note: 1) The result for slurry AZ-6 is reported on both an as-received basis and a dry-weight basis. The dry-weight basis results have been generated by using a weight% solids result of 13.34% for slurry AZ-6. 2) A few slurry results are not the same as those provided in a preliminary report, and the preliminary report should be discarded.

Liquid Sample Analysis Q.C. Comments:

Duplicate: No duplicate was provided. However, a replicate of AZ-M was prepared in the laboratory and analyzed as a duplicate. For all anions measured above the EQL, the duplicate relative percent difference (RPD) meets the acceptance criteria of <20% of the laboratory's QA plan.

Laboratory Control Sample/Blank Spike - LCS 020419 and 020426: A blank spike LCS was prepared at the IC workstation by diluting the high verification check standard (HCV 020411) by 3x. Both LCSs demonstrated recoveries well within the 80% to 120% acceptance criteria.

Matrix Spike (CCV 020411 @2x): The matrix spike (MS) was prepared using the mid-range calibration check standard and sample AZ-M. All anions recovered within the 75% to 125% acceptance criteria.

Process/Dilution Blank: No anions were measured above the EQL in the distilled, deionized water used to dilute the liquid samples for analysis.

Slurry Sample Analysis Q.C. Comments:
The results for continuing calibration check standard slightly exceeded the QC acceptance criteria (i.e., 112% recovery versus a limit of 110%) for the initial analysis of the leached slurries (01/11/02). The leached slurry samples were reanalyzed (02/19/02) to confirm the reported results. For information and comparison, both the initial results from the leached samples and the 'rerun' results for the leached samples are provided in the Table 2.

Duplicate: No duplicate was provided. However, sample AZ-6 was leached and analyzed in duplicate. The RPDs are poor and many do not meet the acceptance criteria of <20% of the laboratory's QA plan. However, it should be noted that the RPD failures were for anions that were measured at only 2 to 10 times the EQL.

"HOT CELL" Laboratory Control Sample/Blank Spike – (BS HCV 010912): A hot cell blank spike was prepared by diluting the high calibration verification. This hot cell blank spike demonstrated recoveries well within the 80% to 120% acceptance criteria defined by the laboratory's QA Plan. However, the recoveries were generally a little lower than those from the blank spike prepared and analyzed at the IC workstation.

"IC Workstation" Laboratory Control Sample/Blank Spike – (LCS 020111 and 020219]): A blank spike LCS was prepared at the IC workstation for each analysis run on the slurry leaches by diluting the high verification check standard (HCV 010912) by 3x. The LCS demonstrated recoveries well within the 80% to 120% acceptance criteria.

Matrix Spike (HCV 010912 at approximately 2.4x): The matrix spike (MS) was prepared adding a known quantity of the high calibration verification standard to sample AZ-6 and subjecting this mixture to the same leaching process/handling as the samples. Nitrite could not be recovered because the concentration of the spike was significantly less that 20% of the sample concentration. Phosphate and sulfate demonstrated very low recoveries. Due to the poor recovers of the MS, a post spike was prepared and analyzed.

Post Spike (HCV 010912 @2x): The post spike (PS) was prepared using the high calibration check standard and the leachate from the AZ-6 sample. The PS recoveries were within the 75% to 125% recovery acceptance criteria for the anions of interest for both analysis runs.

Process/Dilution Blank: The distilled, deionized water used to leach and dilute the samples was analyzed for all reported analytes. Except for a very trace quantity of sulfate, no anions were measured above the EQL. The process blank meets the QA plan's acceptance criteria, since the sulfate concentration is significantly less than 5% of any sample concentration.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: ________ Date 5-21-02
Review/Approval: Marilyn J Steele Date 5-21-02
Excel Archive Information: ASR 6284 Liquids.xls and ASR 6284 Solids.xls

Table 1. Liquid Samples

RPL Number	Sample ID	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	**0.13**	**0.13**	**0.25**	**0.13**	**0.25**	**0.25**	**0.25**	**0.25**
Dilution Blank	Dilution Blank	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	MRQ	**150**	**25**	**n/a**	**n/a**	**3,000**	**2,500**	**2,300**	**n/a**
	EQL	**250**	**250**	**1,000**	**250**	**1,000**	**500**	**500**	**500**
02-00822	AZ-A	1,670	< 250	85,700	1,010	71,700	2,390	15,600	1,000
	EQL	**125**	**125**	**500**	**125**	**250**	**250**	**250**	**250**
02-00823	AZ-C	1,410	< 125	32,900	430	29,300	1,100	9,480	1,710
	EQL	**125**	**125**	**250**	**125**	**250**	**250**	**250**	**250**
02-00824	AZ-E	770	< 125	16,100	250	13,400	650	4,840	910
02-00825	AZ-G	430	830	7,790	< 125	6,560	650	2,710	600
02-00826	AZ-I	260	590	4,540	< 125	3,990	< 250	1,850	430
02-00827	AZ-K	< 125	310	1,890	< 125	1,930	< 250	970	< 250
02-00828	AZ-M	< 125	250	1,250	< 125	1,280	< 250	770	< 250
02-00828 Dup	AZ-M Dup	< 125	260	1,250	< 125	1,310	< 250	780	< 250
	RPD	**(b)**	**4%**	**0%**	**(b)**	**2%**	**(b)**	**1%**	**(b)**
Liquid Batch QC Sample Performance									
LCS 020419	Lab Control %Rec	104%	100%	101%	101%	95%	103%	102%	106%
LCS 020426	Lab Control %Rec	106%	98%	102%	102%	96%	104%	102%	107%
02-00828 MS	Matrix Spike %Rec	107%	101%	101%	104%	94%	104%	101%	108%

EQL: estimate quantitation limit
MRQ: minimum reportable quantity
RPD: relative percent difference

(a) The fluoride results should be considered the upper bound concentration for the fluoride; fluoride not resolved from acetate/fomate.

(b) Not applicable; sample and/or duplicate concentration <EQL.

Table 2: Slurry Samples

RPL Number	**Sample ID**	**F µg/ml**	**Cl µg/ml**	**NO_2 µg/ml**	**Br µg/ml**	**NO_3 µg/ml**	**PO_4 µg/ml**	**SO_4 µg/ml**	**C_2O_4 µg/ml**	
	EQL	**0.13**	**0.13**	**0.25**	**0.13**	**0.25**	**0.25**	**0.25**	**0.25**	**ALO-103 Leach Factor**
02-00832 DB	Dilution Blank at IC	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	0.30	< 0.25	
02-00832 PB	Process Blank at IC	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	0.35	< 0.25	
RPL Number	**Sample ID**	**µg/g**	**µg/g**	**µg/g**	**µg/g**	**µg/g**	**µg/g**	**µg/g**	**µg/g**	
	02-00829/02-00831 MRQ	**7,500**	**230**	**n/a**	**n/a**	**450**	**600**	**1,200**	**n/a**	
	EQL	**2.2**	**2.2**	**4.4**	**2.2**	**4.4**	**4.4**	**4.4**	**4.4**	
02-00832 PB	Process Blank as Sample	< 2.2	< 2.2	< 4.4	< 2.2	< 4.4	< 4.4	6.2	< 4.4	17.59 (d)
	EQL	**200**	**200**	**4,000**	**200**	**4,000**	**400**	**4,000**	**400**	
02-00829	AZ-0	1,400	< 200	44,000	370	39,000	960	17,500	1,810	15.86
RR 02-00829	Rerun AZ-0	1,500	< 200	43,600	470	39,200	< 400	17,700	1,190	15.86
	EQL	**32**	**32**	**580**	**32**	**580**	**63**	**63**	**63**	
02-00830	AZ-2	360	< 32	10,300	110	7,880	170	2,730	450	23.03
RR 02-00830	Rerun AZ-2	350	< 32	9,760	97	7,620	180	2,800	< 63	23.03
	EQL	**22**	**22**	**45**	**22**	**45**	**45**	**45**	**45**	
02-00831	AZ-4	120	580	5,350	61	3,530	170	1,490	190	18.12
RR 02-00831	Rerun AZ-4	86	590	5,630	52	3,730	160	1,470	180	18.12
	02-00832 MRQ	**7,500**	**230**	**3,000**	**n/a**	**450**	**600**	**1,200**	**1,800**	
	EQL	**22**	**22**	**45**	**22**	**45**	**45**	**45**	**45**	
02-00832	AZ-6	44	110	1,110	< 22	330	<45	410	84	15.40
RR 02-00832	Rerun AZ-6	< 22	96	920	< 22	240	<45	320	<45	15.40
02-00832 Dup	AZ-6 Dup	73	76	910	< 22	300	<45	260	76	15.55
RR 02-00832 Dup	Rerun AZ-6 Dup	68	94	940	< 22	290	<45	300	71	15.55
	RPD	**51%**	**36%**	**20%**	**(b)**	**7%**	**(b)**	**45%**	**11%**	
	Rerun RPD	**(b)**	**3%**	**3%**	**(b)**	**20%**	**(b)**	**5%**	**(b)**	
AZ-6 (02-00832) adjusted for 13.34 wt% solids										
	AZ-6 (dry basis)	330	820	8,320	< 170	2,440	< 340	3,080	630	15.40
	Rerun AZ-6 (dry basis)	< 170	720	6,890	< 170	1,790	< 340	2,370	< 340	15.40
	AZ-6 Dup (dry basis)	550	570	6,780	< 170	2,280	< 340	1,940	570	15.55
	Rerun AZ-6 Dup (dry basis)	510	700	7,080	< 170	2,200	< 340	2,250	530	15.55
Slurry Batch QC Sample Performance										
BS (HCV010912)	Hot Cell LCS %Rec	95%	94%	93%	94%	90%	95%	95%	98%	
LCS 020211	Lab LCS %Rec	94%	93%	98%	97%	93%	98%	97%	101%	
RR LCS 020219	Lab LCS %Rec	94%	97%	99%	98%	93%	95%	94%	100%	
02-00832 MS	Matrix Spike %Rec	114%	80%	(c)	129%	116%	63%	66%	101%	
02-00832 PS	Post Spike %Rec	94%	95%	97%	98%	92%	94%	92%	97%	
RR 02-00832 PS	Post Spike %Rec	97%	97%	99%	98%	94%	95%	94%	100%	

EQL: estimate quantitation limit MRQ: minimum reportable quantity RPD: relative percent difference

(a) The fluoride results should be considered the upper bound concentration for the fluoride; fluoride not resolved from acetate/fomate.

(b) Not applicable; sample and/or duplicate concentration <EQL.

(c) Not recoverable; MS diluted 10x and 105x; all anions except phosphate either <EQL or <<20% of sample concentration.

(d) Nominal process blank (i.e., distilled, deionized water) leach factor is the average of sample and duplicate leach factors. Used to provide process blank results in µg/g units, for comparison with samples.

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC/TIC Report – Furnace Oxidation Method

PO Box 999, Richland, Washington 99352

Project Number:	42365
Charge Code:	W60513
ASR Number:	6284
Client:	J. Geeting
Total Samples:	7 (liquids)

	First in Series	Last in Series
RPL Numbers	02-0822	02-0828
Client IDs	AZ-A	AZ-M

Analysis Procedure	PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"
Prep Procedure	None
Analyst	M. Steele
Analysis Date	06/19/2002 (TC) and 07/22/2002 (TOC)
Cal/Verify Standards	TOC: CMS-53219, TC: CMS-161359
MS/LCS Standards	TOC: CMS-161713, TC: CMS-161732
Excel Data File	ASR 6280 6284 6378 F 700 1000.xls
M&TE Numbers	Carbon System (WD13071)
	Balance (360-06-01-023)
All Analysis Records	Project File

Prepared By 7-30-02 **Date**

Reviewed By 7-30-02 **Date**

Carbon Results

RPL Number	Sample ID	TIC Results µgC/mL	TOC MDL µgC/mL	TOC Results µgC/mL	TOC RPD	TC MDL µgC/mL	TC Results µgC/mL	TC RPD
	MRQ	**150**		**1,500**				
02-00822	AZ-A	4,000	60	4,600		14	8,600	
02-00823	AZ-C	800	130	3,600		11	4,400	
02-00824	AZ-E	n/d	60	2,100		11	2,100	
02-00825	AZ-G	830	60	370		6	1,200	
02-00825 Dup	AZ-G Dup		30	330	13%			
02-00826	AZ-I	550	60	270		6	820	
02-00826 Dup	AZ-I Dup					6	760	9%
02-00827	AZ-K	80	40	260		6	340	
02-00828	AZ-M	540	30	460		6	1,000	
02-00824 MS	Recovery			114%				
02-00826 MS	Recovery						105%	
LCS/BS	Recovery			96%			102%	

TOC: total organic carbon TC: total carbon TIC: total inorganic carbon (by difference)
MDL: method detection limit RPD: relative percent difference MRQ: minimum reportable quantity
n/d: not calculated (TC = or > TOC)

Sample Analysis/Results Discussion

The TOC/TIC analyses of the seven liquid samples submitted under ASR 6284 were to be performed by both the hot persulfate and furnace oxidation methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate oxidation method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 700 °C for 10 minutes. The total carbon (TC) is determined on another aliquot of the sample by combusting at 1000 °C for 10 minutes. The total inorganic carbon (TIC) is obtained by difference.

The table above shows the results, rounded to two or three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

Quality Control Discussion

The calibration and QC standards for TC and TOC analysis are solid pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt (calcium carbonate for TC and α-D-glucose for TOC). The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC for the method involves calibration blanks, sample duplicates (laboratory), laboratory control sample, and matrix spike. The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs"; the performance of the QC samples is compared to this Plan. The ASR establishes the minimum reportable quantity (MRQ).

The calibration of the coulometer analysis system is checked by analyzing calibration check standards at the beginning, middle, and end of each day's run. The samples were analyzed for TOC as a batch and for TC as a batch. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the TOC was 99% and for TC was 97%.

Analysis Run 06/19/2002: TC QC

Laboratory Control Sample/Blank Spike: A TC LCS/BS (inorganic standard) was analyzed with the samples. At 102% TC, the LCS/BS recovery is well within acceptance criterion of 80% to 120%.

Duplicate: The TC measurement precision is demonstrated by the RPD between duplicate analyses. No duplicate sample was provided by the client; therefore, a laboratory duplicate were prepared from sample 'AZ-I'. At 9%, the TC RPD for the laboratory duplicate meets the acceptance criterion of <20%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the MS. A MS was prepared from sample 'AZ-I' by adding a known quantity of an inorganic standard. The TC MS recovery of 105% is well within the acceptance criterion of 75% to 125% recovery.

Analysis Run 07/22/2002: TOC QC

Laboratory Control Sample/Blank Spike: A TOC LCS/BS (organic standard) was analyzed with samples. At 96%, the LCS/BS recovery is well within acceptance criterion of 80% to 120%.

Duplicates: A laboratory duplicate was prepared from sample 'AZ-G'; the TOC RPD meets the QA Plan's acceptance criterion of <20%.

Matrix Spike: A MS were prepared from sample 'AZ-E' by adding a known quantity of an organic standard. The TOC MS recovery of 114% is slightly elevated, but within the acceptance criteria of 75% to 125%.

Deviations from Procedure

None.

Comparison: Furnace Oxidation and Hot Persulfate Oxidation Results

RPL Number	Sample ID	TIC-F(a) Results µgC/mL	TIC-HP Results µgC/mL	TOC-F Results µgC/mL	TOC-HP Results µgC/mL	TC-F Results µgC/mL	TC-HP(b) Results µgC/mL
02-00822	AZ-A	4,000	8,470	4,600	490	8,600	8,960
02-00823	AZ-C	800	4,010	3,600	440	4,400	4,450
02-00824	AZ-E	N/d	2,020	2,100	230	2,100	2,250
02-00825	AZ-G	830	1,160	370	130	1,200	1,290
02-00826	AZ-I	550	730	270	65	820	795
02-00827	AZ-K	80	310	260	< 50	340	310
02-00828	AZ-M	540	950	460	< 50	1,000	950

HP= Hot Persulfate Method F= Furnace Combustion Method n/d= not detected (TC≤TOC)

a) TIC Furn is determined by difference (TC-F minus TOC-F)
b) TC HP is determined by sum (TIC-HP plus TOC-HP)

The TC results between the two methods compare extremely well. However, there is significantly less correlation between the TIC and TOC results. At the 700 °C temperature used for analysis of TOC by the furnace method, some metal carbonates, such as iron, magnesium, and nickel, may fully oxidize and bias the TOC results high (thus a low TIC is calculated). Some organic compounds are difficult to oxidize by the hot persulfate method, leading to a potential low bias in the TOC results; this effect can also be seen in the furnace method (but is generally not as severe). Typically the furnace method produces the highest and most accurate TC results, and the hot persulfate method the most accurate TIC results; thus the best estimate for TOC is 'TC-F minus TIC-HP'.

General Comments

1) The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
3) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results <5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated.
4) For both the TC and TOC, the analysis MDL is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's t values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in µgC/ml or µgC/g) are calculated by using the analysis MDL adjusted for the sample volume or mass.

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Urie/Geeting	Temp.	Balance M&TE: 360-06-01-023
Project :		1000 Degree C	
Work Pkg:	CMC	Run time	
Analyzed:	June 19, 2002	10 Minutes	TIC Cal Std: JT Baker CMS#161359 11.99% Carbon <<[G]
ASR:	6378 6284		LCS/BS/MS TIC Std: Mallinckrodt CMS#161732 11.99% Carbon <<[C]

		Raw TC (ug C)	
Blanks:	Calibration blank (start of batch)	40.6	
	Calibration blank (start of batch)	41.3	
	Calibration blank (end of batch)	41.9	

41.3	<<< Blank Average (ug C)
0.6	<<< Blank Std Dev (ug C)
3	<<< ***# of Blanks analyzed***
2.8	<<< ***Method Det. Limit (ug C)*** [M]

		Total Carbon (TC)			
		[A] Raw TC (ug)	[B] Blk (ug)	[D] Std Vol (ml)	TIC % Rec
Standards:	Calibration Standard (start of batch)	2291	41	0.0196	95.7
	Calibration Standard (start of batch)	2700	41	0.0218	101.7
	Calibration Standard (middle of batch)	3345	41	0.0293	94.0
	Calibration Standard (end of batch)	1841	41	0.0156	96.2
QC	Blank Spike/LCS	1351	41	0.0111	101.5

96.9	<<< [L] Average TC % Rec

Formulas:

Standard TC % Recovery = ((A-B)/((G/100)*D))*E^{-6}*100

QC % Recovery = (((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)

Sample TC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

MS TC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).

For any TC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

Sample Results		[I] Raw	[J]	[K] Sam	TC	TC
RPL Number	Sample ID	TC (ug C)	Blk (ug C)	Vol. (ml)	(ug C/ml)	RPD (%)
ASR 6378						
02-01832	AP-104-AR-G	1616	41	0.20	8,123	
02-01832	AP-104-AR-G Dup	1628	41	0.20	8,185	1
02-01832	AR-104-AR-G Trip	1619	41	0.20	8,139	
02-01832 MS	AR-101-AR-G MS	2995	41	0.20	see below	
ASR 6284						
02-00822	AZ-A	1702	41	0.20	8,567	
02-00823	AZ-C	1107	41	0.25	4,398	
02-00824	AZ-E	548	41	0.25	2,091	
02-00825	AZ-G	617	41	0.50	1,188	
02-00827	AZ-K	204	41	0.50	336	
02-00828	AZ-M	525	41	0.50	998	
02-00826	AZ-I	440	41	0.50	823	
02-00826	AZ-I Dup	407	41	0.50	755	9
02-00826 MS	AZ-I	1111	41	0.50	see below	

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	Sample ID	(ug C)	(ug C)	(ug C/ml)	Vol. (ml)	(ug C)	wt (g)	(ug C)	% Recovery
02-00826 MS	Total Carbon Recovery (TC)	1111	41	823	0.50	411	0.0055	659	**105.0**
02-01832 MS	Total Carbon Recovery (TC)	2995	41	8123	0.20	1625	0.0126	1511	**94.2**

Reviewer/date: 7-30-02

PNNL Radiochemical Processing Group: TOC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Geeting	Temp.	Analyzer M&TE: WD13071 -- 701 Balance M&TE: 360-06-01-023
Project :		700 Degree C	
Work Pkg:	CMC	Run time	Cal Std: alpha-D-glucose Aldrich CMS#53219 40.00% Carbon <<[G]
Analyzed:	July 22, 2002	10 Minutes	BS MS Std: alpha-D-glucose Sigma CMS#161713 40.00% Carbon <<[C]
ASR:	6284		

		TOC (ug C)	
Blanks:	Calibration blank (start of batch)	22.2	
	Calibration blank (start of batch)	23.6	
	Calibration blank (end of batch)	18.1	

21.3	**<<< Blank Average (ug C)**
2.9	**<<< Blank Std Dev (ug C)**
3	***<<< # of Blanks analyzed***
13	***<<< Method Det. Limit (ug C)* [M]**

		TOC				
		[A] Raw TOC (ug)	**[B] Blk (ug)**	**[D] Std Vol (ml)**	**TOC % Rec**	
Standards:	Calibration Standard (start of batch)	1760	21	0.00450	96.6	
	Calibration Standard (start of batch)	960	21	0.00229	102.5	
	Calibration Standard (end of batch)	921	21	0.00229	98.2	
QC	Blank Spike/LCS	827	21	0.00212	95.9	

99.1 **<<< [L] Average TOC % Rec**

Formulas:	Standard TOC % Recovery = $((A-B)/((C/100)*D))*E^{-6}*100$
	QC % Recovery = $(((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)$
	Sample TOC (ug C/ml or ug C/g) = (I-J)/(K*L/100)
	MS TOC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U
Comments:	Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.
	The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).
	For any TC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].
	If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

Sample Results		[I] Raw	[J]	[K] Sam	TOC	
RPL Number	**Sample ID**	**TOC (ug C)**	**Blk (ug C)**	**Vol. (ml)**	**(ug C/ml)**	**RPD (%)**
ASR 6284						
02-00822	AZ-A	927	21	0.20	4,570 ✓	
02-00823	AZ-C	375	21	0.10	3,569	
02-00824	AZ-E	436	21	0.20	2,092	
02-00824 MS	AZ-E MS	829	21	0.20	see below	
02-00825	AZ-G	95	21	0.20	373	
02-00825 Dup	AZ-G Dup	151	21	0.40	327 ✓	13
02-00826	AZ-I	76	21	0.20	274	
02-00827	AZ-K	97	21	0.30	255	
02-00828	AZ-M	205	21	0.40	463 ✓	

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	**Client Sample ID**	**(ug C)**	**(ug C)**	**(ug C/ml)**	**Vol. (ml)**	**(ug C)**	**Wt (g)**	**(ug C)**	**% Recovery**
02-00824 MS	Total Organic Carbon Recovery	829	21	2092	0.20	418	0.00087	348	**114.0**

Reviewer/date: 7-30-02

Battelle PNNL/RSE/Inorganic Analysis --- TOC/TIC Report

PO Box 999, Richland, WA 99352

Client: J. Geeting
RPL Numbers: 02-0822 to 02-828
Analyst: MJ Steele

Charge Code/Project: W60513 / 42365
ASR Number: 6284
Analysis Date: May 15, 2002

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"
M&TE: Carbon System (WA92040); Balance (360-06-01-023)

Analysis Results

RPL Number	Sample ID	TIC MDL µgC/mL	TIC Results µgC/mL	TIC RPD	TOC MDL µgC/mL	TOC Results µgC/mL	TOC RPD	TC Results µgC/mL	TC RPD
	MRQ		150		1,500				
02-0822	AZ-A	70	8,470		180	490		8,960	
02-0822 D	AZ-A Dup	30	8,390	1%	90	710	na	9,100	2%
02-0823	AZ-C	30	4,010		90	440		4,450	
02-0824	AZ-E	20	2,020		60	230		2,250	
02-0825	AZ-G	20	1,160		50	130		1,290	
02-0826	AZ-I	20	730		50	65		795	
02-0827	AZ-K	20	310		50	< 50		310	
02-0827 D	AZ-K Dup	20	310	0%	50	< 50	na	310	0%
02-0828	AZ-M	20	950		50	< 50		950	
02-0824 MS	%Recovery		99%			104%		101%	
Blank Spike/LCS	%Recovery		98%			100%			

MRQ = minimum required quantity; RPD = Relative Percent Difference; MDL = method detection limit; na = not applicable

The TOC/TIC analyses of the seven liquid samples submitted under ASR 6284 are to be performed by both the hot persulfate and furnace methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

Q.C. Comments:

The TIC analysis uses calcium carbonate and the TOC analysis uses α-D-Glucose as the calibration, laboratory control, and matrix spike standards. (The JT Baker, Aldrich, Sigma, and Mallinckrodt Chemical Measurement System numbers are provided on the raw data benchsheets for traceability).

The QC for the method involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes per analysis batch. The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs"; the performance of the QC samples is compared to this Plan.

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyzed with the batch of samples. At 98% for TIC and 100% for TOC, the LCS/BS recoveries are well within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample AZ-E, an inorganic standard, and an organic standard. The TIC and TOC matrix spike recoveries are well within the acceptance criteria of 75% to 125% recovery. At 101%, the TC recovery (TIC + TOC standard) indicates that all the carbon added as a matrix spike was recovered.

Duplicates: The precision between the duplicates (replicates) is demonstrated by the Relative Percent Difference (RPD) between sample and duplicate. Two duplicate sample were prepared; one from samples of AZ-A and one from AZ-K. The TIC RPD results are within the QP Plan acceptance criteria of <20% RPD. The TOC RPD could not be determined since the either the sample or duplicate results are less than five times the MDL.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data. The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: ____________________ **Date** 6/4/02

Review/Approval by: ____________________ **Date** 6-5-02

Excel Archive File: ASR 6184 Geeting L HP.xls

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC/TIC Report – Furnace Oxidation Method

PO Box 999, Richland, Washington 99352

Project Number:	42365
Charge Code:	W60513
ASR Number:	6284
Client:	J. Geeting
Total Samples:	4 (solids)

	First in Series	Last in Series
RPL Numbers	02-0829	02-0832
Client IDs	AZ-0	AZ-6

Analysis Procedure	PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"
Prep Procedure	None
Analyst	M. Steele
Analysis Date	06/20/2002 (TC) and 06/28/2002 (TOC)
Cal/Verify Standards	TOC: CMS-53219, TC: CMS-161359
MS/LCS Standards	TOC: CMS-161713, TC: CMS-161732
Excel Data File	ASR 6280 6284 6378 F 700 1000.xls
M&TE Numbers	Carbon System (WD13071) Balance (360-06-01-023)
All Analysis Records	Project File

7-30-02

Prepared By **Date**

7-30-02

Reviewed By **Date**

Carbon Results

RPL Number	Sample ID	TIC Results µgC/g dry	TOC MDL µgC/g dry	TOC Results µgC/g dry	TOC RPD	TC MDL µgC/g dry	TC Results µgC/g dry	TC RPD
	MRQ			**150**			**1,500**	
00-0829	AZ-0	n/d	400	9,800		60	9,000	
00-0829 Dup	AZ-0 Dup		600	10,600	8%			
00-0830	AZ-2	n/d	500	9,300		90	8,400	
00-0831	AZ-4	6,800	300	10,200		200	17,000	
02-00831 Dup	AZ-4 Dup					100	14,900	13%
	MRQ			**30**			**60**	
00-0832	AZ-6	11,100	400	13,200		200	24,300	
00-0829 MS	MS %Rec			107%				
02-0832 MS	MS %Rec						110%	
LCS/Blank Spike 1	LCS/BS %Rec			93%			102%	
LCS/Blank Spike 2	LCS/BS %Rec			103%				

TOC: total organic carbon
MDL: method detection limit
n/d: not calculated (TC > TOC)
TC: total carbon
RPD: relative percent difference
TIC: total inorganic carbon (by difference)
MRQ: minimum reportable quantity

Sample Analysis/Results Discussion

The TOC/TIC analyses of the four solids samples submitted under ASR 6284 were to be performed by both the hot persulfate and furnace oxidation methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate oxidation method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 700 °C for 20 minutes. The total carbon (TC) is determined on another aliquot of the sample by combusting at 1000 °C for 10 minutes. The total inorganic carbon (TIC) is obtained by difference.

The table above shows the results, rounded to two or three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

Quality Control Discussion

The calibration and QC standards for TC and TOC analysis are solid pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt (calcium carbonate for TC and α-D-glucose for TOC). The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC for the method involves calibration blanks, sample duplicates (laboratory), laboratory control sample/blank spike (LCS/BS), and matrix spike (MS). The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs"; the performance of the QC samples is compared to this Plan.

The calibration of the coulometer analysis system is checked by analyzing calibration check standards at the beginning, middle, and end of each day's run. The samples were analyzed for TOC as a batch and for TC as a batch. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the TOC was 99% and for TC was 95%.

Analysis Run 06/20/2002: TC QC

Laboratory Control Sample/Blank Spike: A TC LCS/BS (inorganic standard) was analyzed with the samples. At 102% TC, the LCS/BS recovery is well within acceptance criteria of 80% to 120%.

Duplicate: The TC measurement precision is demonstrated by the RPD between duplicate analyses. No duplicate sample was provided by the client; therefore, a laboratory duplicate were prepared from sample 'AZ-4'. At 13%, the TC RPD for the laboratory duplicate is slightly elevated, but meets the QA Plan's acceptance criterion of <20%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the MS. A MS was prepared from sample 'AZ-6' by adding a know quantity of an inorganic standard. The TC MS recovery of 110% is well within the acceptance criterion of 75% to 125% recovery.

Analysis Run 06/28/2002: TOC QC

Laboratory Control Sample/Blank Spike: Two TOC LCS/BSs (organic standard) were analyzed with samples. At 93% and 102%, the LCS/BS recoveries are well within acceptance criterion of 80% to 120%.

Duplicates: A laboratory duplicate was prepared from sample 'AZ-0'; the TOC RPD meets the QA Plan's acceptance criterion of <20%.

Matrix Spike: A MS were prepared from sample 'AZ-0' by adding a know quantity of an organic standard. The TOC MS recovery of 107% is well within the acceptance criteria of 75% to 125%.

Deviations from Procedure

None.

Comparison: Furnace Oxidation and Hot Persulfate Oxidation Results

RPL Number	Sample ID	TIC-F (a) Results µgC/g dry	TIC-HP Results µgC/g dry	TOC-F Results µgC/g dry	TOC-HP Results µgC/g dry	TC-F Results µgC/g dry	TC-HP (b) Results µgC/g dry
00-0829	AZ-0	n/d	14,500	9,800	1,400	9,000	15,900
00-0830	AZ-2	n/d	9,200	9,300	1,300	8,400	10,500
00-0831	AZ-4	6,800	15,100	10,200	500	17,000	15,600
00-0832	AZ-6	11,100	10,800	13,200	1,400	24,300	12,200

HP= Hot Persulfate Method F= Furnace Combustion Method n/d= not detected (TC≤TOC)

a) TIC Furn is determined by difference (TC-F minus TOC-F)

b) TC HP is determined by sum (TIC-HP plus TOC-HP)

There is very little correlation between the TIC, TOC, and TC results between the two methods. At the 700 °C temperature used for analysis of TOC by the furnace method, some metal carbonates, such as iron, magnesium, and nickel, may fully oxidize and bias the TOC results high. Some organic compounds are difficult to oxidize by the hot persulfate method, leading to a potential low bias in the TOC results; this effect can also be seen in the furnace method (but is generally not as severe). Assuming consistent sub-sampling and sample integrity, the furnace method typically produces the highest and most accurate TC results and the hot persulfate method the most accurate TIC results; thus the best estimate for TOC is 'TC-F minus TIC-HP'. It is unlikely that the hot persulfate TIC is bias high.

However, the significant differences between the TC results suggest a complex sample matrix, loss of sample integrity (e.g., TIC change with time due to adsorption of atmospheric CO_2), and/or severe sample heterogeneity (e.g., 70 mg to 200 mg samples may not be of sufficient size to represent the average carbon concentration). The samples represent virgin, washed, and leached solids with interstitial liquid that have been dried prior to performing the carbon analysis. The interstitial liquid (dried) from the virgin solids is most likely in equilibrium with atmospheric CO_2; however, the washed and leached 'caustic' solids may re-adsorb CO_2 from the atmosphere, resulting in variable TIC results depending on length of exposure and sub-sampling location. The dates the solids were sub-sampled for analysis could result in significant differences between the methods for both TIC and TC (i.e., TC from furnace combustion determined in mid June and TC established from hot persulfate combustion in mid July). Without additional analyses or sample chemistry, it is not possible to assess which method produces the most reasonable results for these samples.

General Comments

1) The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
3) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results <5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated.
4) For both the TC and TOC, the analysis MDL is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's t values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in µgC/mL or µgC/g) are calculated by using the analysis MDL adjusted for the sample volume or mass.

PNNL Radiochemical Processing Group: TOC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Geeting	Temp.	Analyzer M&TE: WD13071 -- 701 Balance M&TE: 360-06-01-023
Project :		700 Degree C	
Work Pkg:	CMC	Run time	CAL STD - alpha-D-glucose: Aldrich, lot HY12603EY, CMS# 53219 40.00% Carbon <<[G]
Analyzed:	June 28, 2002	20 Minutes	BS/MS -alpha-D-glucose: Sigma Lot 58H01281, CMS# 161713 40.00% Carbon <<[C]
ASR:	6284		

		TOC (ug C)	
Blanks:	Calibration blank (start of batch)	40.7	
	Calibration blank (start of batch)	45.6	
	Calibration blank (end of batch)	55.1	

47.1	**<<< Blank Average (ug C)**
7.4	**<<< Blank Std Dev (ug C)**
3	***<<< # of Blanks analyzed***
33	***<<< Method Det. Limit (ug C)* [M]**

		TOC			
		[A] Raw TOC (ug)	**[B] Blk (ug)**	**[D] Std Wt (g)**	**TOC % Rec**
Standards:	Calibration Standard (start of batch)	1682	47	0.0040	102.2
	Calibration Standard (start of batch)	1300	47	0.0034	92.1
	Calibration Standard (start of batch)	1692	47	0.0040	102.8
	Calibration Standard (end of batch)	2256	47	0.0056	98.6
QC	Blank Spike/LCS	1522	47	0.0040	93.2
	Blank Spike/LCS	1480	47	0.0035	103.5

98.9   **<<<< [L] Average TOC % Rec**

Formulas:

Standard TOC % Recovery = $((A-B)/((C/100)*D))*E^{-6}*100$

QC % Recovery = $(((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)$

Sample TOC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

MS TOC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).

For any TOC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

Note: Sample weights are on "as received" basis; i.e., wet weight

Sample Results		**[I] Raw**	**[J]**	**[K] Sam**	**TOC**	
RPL Number	**Sample ID**	**TOC (ug C)**	**Blk (ug C)**	**Wt (g)**	**(ug C/g)**	**RPD (%)**
ASR 6284						
02-829	AZ-0	790	47	0.0767	9,790	
02-829	AZ-0	618	47	0.0543	10,627	8
02-829	AZ-0	418	47	0.0321	11,679	18
02-829 MS	AZ-0	1792	47	0.0713	see below	
02-830	AZ-2	685	47	0.0695	9,278	
02-831	AZ-4	1155	47	0.1102	10,162	
02-832	AZ-6	1067	47	0.0779	13,234	

Matrix Spike Results		**[Q] Raw MS**	**[R] MS Blk**	**[S] Sam**	**[T] MS Sam**	**[V] Sample**	**Spike**	**[U] Spike**	**MS**
RPL Number	**Client Sample ID**	**(ug C)**	**(ug C)**	**(ug C/g)**	**wt (g)**	**(ug C)**	**wt (g)**	**(ug C)**	**% Recovery**
02-829 MS	Total Organic Carbon Recovery	1792	47	9790	0.0713	698	0.0025	1000	**106.6**

Reviewer/date: 7-30-02

PNNL Radiochemical Processing Group: TC Calculations **Review** Report - Furnace Method PNL-ALO-380

Client:	Geeting	Temp.	Analyzer M&TE: WD13071 -- 701 Balance M&TE: 360-06-01-023
Project :		1000 Degree C	
Work Pkg:	CMC	Run time	MS/LCS: MALLINCKRODT LOT 4071 KTRT CMS#161732 11.99% Carbon <<[G]
Analyzed:	June 20, 2002	10 Minutes	Cal Check: JT BAKER LOT N30628 CMS #161359 11.99% Carbon <<[C]
ASR:	6284 solids		

		Raw TC (ug C)	
Blanks:	Calibration blank (start of batch)	45.0	
	Calibration blank (start of batch)	40.8	
	Calibration blank (end of batch)	46.8	

44.2	**<<< Blank Average (ug C)**
3.1	**<<< Blank Std Dev (ug C)**
3	***<<< Pooled Std Dev (ug C)***
13.8	***<<< Method Det. Limit (ug C)* [M]**

		Total Carbon (TC)			
		[A] Raw TC (ug)	**[B] Blk (ug)**	**[D] Std wt (g)**	**TC % Rec**
Standards:	Calibration Standard (start of batch)	3732	44	0.0321	95.8
	Calibration Standard (start of batch)	4648	44	0.0406	94.6
	Calibration Standard (end of batch)	2876	44	0.0249	94.9
QC	Blank Spike/LCS	570	44	0.0045	102.5

95.1 **<<<< [L] Average TC % Rec**

Formulas:

Standard TC % Recovery = ((A-B)/((C/100)*D))*E^{-6}*100

QC % Recovery = (((A-B)/((C/100)*D))*E^{-6}*100)/(L/100)

Sample TC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

MS TC % Recovery = ((((Q-R)/(L/100))-S*T)*100)/U

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Method Detection Limit for the batch run is the Std Deviation from the number (n) of blanks times the Student's *t* value for the number of degrees of freedom (n-1).

For any TC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit [M] by [K].

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

Note: Sample weights are on "as received" basis; i.e., wet weight

Sample Results		[I] Raw	[J]	[K] Sam	TC	TC
RPL Number	**Sample ID**	**TC (ug C)**	**Blk (ug C)**	**Wt (g)**	**(ug C/g)**	**RPD (%)**
ASR 6284						
02-00829	AZ-0	2,130	44	0.2434	9,013	
02-00830	AZ-2	1,348	44	0.1627	8,428	
02-00831	AZ-4	1,109	44	0.0658	17,020	
02-00831	AZ-4 Dup	1,074	44	0.0725	14,939	13
02-00832	AZ-6	1,770	44	0.0748	24,266	
02-00832	AZ-6 MS	1,272	44	0.0287	see below	

Matrix Spike Results		[Q] Raw MS	[R] MS Blk	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
RPL Number	**Sample ID**	**(ug C)**	**(ug C)**	**(ug C/ml)**	**wt (g)**	**(ug C)**	**wt (g)**	**(ug C)**	**% Recovery**
02-00831	Total Carbon Recovery (TC)	1,272	44	24,266	0.0287	696	0.0045	540	**110.3**

Reviewer/date: 7-30-02

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC/TIC Report – Hot Persulfate Oxidation Method

PO Box 999, Richland, Washington 99352

Project Number:	42365
Charge Code:	W60513
ASR Number:	6284
Client:	J. Geeting
Total Samples:	4 (solids)

	First in Series	**Last in Series**
RPL Numbers	02-00829	02-00832
Client IDs	AZ-0	AZ-6

Analysis Procedure	PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"
Prep Procedure	None
Analyst	M. Steele
Analysis Date	07/17/2002
Cal/Verify Standards	TOC CMS-53219, TIC CMS-161359
LCS/MS Standards	TOC CMS-161713, TIC CMS-161732
Excel Data File	ASR 6284 Geeting S HP.xls
M&TE Numbers	Carbon System (WA92040) Balance (360-06-01-023)
All Analysis Records	Project File

7-30-02

Prepared By **Date**

7-30-02

Reviewed By **Date**

Carbon Results

RPL Number	Sample ID	TIC MDL µgC/g dry	TIC Results µgC/g dry	TOC MDL µgC/g dry	TOC Results µgC/g dry	TC Results µgC/g dry
	MRQ		**150**		**1,500**	
02-00829	AZ-0	50	14,500	140	1,400	15,900
02-00830	AZ-2	70	9,200	180	1,300	10,500
02-00831	AZ-4	50	15,100	130	500	15,600
	MRQ		**30**		**60**	
02-00832	AZ-6	70	10,800	180	1,400	12,200
02-00832 MS	Matrix Spike %Rec		115%		99%	105%
Blank Spike/LCS	LCS %Rec		100%		97%	

TIC: total inorganic carbon TOC: total organic carbon TC: total carbon (sum of TIC and TOC)
MDL: method detection limit MRQ: minimum reportable quantity

Sample Analysis/Results Discussion

The TOC/TIC analyses of the four solids samples submitted under Analytical Service Request (ASR) 6284 are to be performed by both the hot persulfate and furnace oxidation methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

Quality Control Discussion

The calibration and QC standards for TIC and TOC analysis are solid pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt (calcium carbonate for TC and α-D-glucose for TOC). The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC for the method involves calibration blanks, sample duplicates (laboratory), laboratory control sample/blank spikes (LCS/BS), and matrix spikes (MS). The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs"; the performance of the QC samples is compared to this Plan.

The calibration of the coulometer analysis system is checked by analyzing calibration check standards at the beginning, middle, and end the analysis run. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recoveries were 99% and 98% for the TIC and TOC, respectively.

Laboratory Control Sample/Blank Spike: A LCS/BS was analyzed with the samples. At 100% TIC and 97% TOC, the LCS/BS recoveries are well within acceptance criterion of 80% to 120%.

Duplicate: Precision of the carbon measurements is demonstrated by the Relative Percent Difference (RPD) between sample and duplicate (or replicate). However, there was insufficient sample to perform both a duplicate analysis and a MS analysis. The decision was made to perform the MS analysis, since good recovery also demonstrated good precision.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the MS. A MS was prepared from sample 'AZ-6', an inorganic standard, and an organic standard (see cover page for standard identification). The TIC and TOC MS recoveries are within the acceptance criterion of 75% to 125% recovery. At 105%, the TC recovery (TIC + TOC standard) indicates that all the carbon added to the MS was recovered.

Deviation from Procedure

None

General Comments

1) The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
3) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results < 5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated.
4) For both the TC and TOC, the analysis MDL is based on three times the standard deviation of a set of historical 'system blank' data. The sample MDL (in µgC/mL or µgC/g) are calculated by using the analysis MDL adjusted for the sample volume or mass.

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Geeting	Analyzer M&TE: WC01713 -- 701
Project :		Balance M&TE: 360-06-01-023
Work Pkg:	CMC	TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS) 40.00% Carbon <<[G]
Analyzed:	July 17, 2002	TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS) 11.99% Carbon <<[C]
ASR:	6284	

		Raw TIC (ug C)	**Raw TOC (ug C)**		**TIC**	**TOC**	
					13.8	**40.1**	**<<< Blank Average (ug C)**
Blanks:	Calibration blank (start of batch)	11.9	44.8		**3.0**	**6.3**	**<<< Blank Std Dev (ug C)**
	Calibration blank (start of batch)	12.1	42.6		***2.16***	***5.8***	***<<< Pooled Std Dev (ug C)***
	Calibration blank (end of batch)	17.3	32.9		***6.5***	***17.3***	***<<< Method Det. Limit (ug C)***

Is Blank Std Dev < Method Det Limit?	
TIC	**Yes**
TOC	**Yes**

		Total Inorganic Carbon (TIC)					**Total Organic Carbon (TOC)**				
		[A] Raw TIC (ug)	**[B] Blk (ug)**	**[D] Std wt (g)**	**TIC % Rec**		**[E] Raw TOC (ug)**	**[F] Blk (ug)**	**[H] Std wt (g)**	**TOC % Rec**	
Standards:	Calibration Standard (start of batch)	1885	14	0.01583	98.6		961	40	0.00232	99.2	
	Calibration Standard (start of batch)	3408	14	0.02871	98.6		1509	40	0.00380	96.6	
	Calibration Standard (end of batch)	1428	14	0.01188	99.3		1750	40	0.00441	96.9	
		[L] Average TIC % Rec >>>>			**98.8**	**<<[L]**	**[P] Average TOC % Rec >>>>**			**97.6**	**<<[P]**
QC	Blank Spike/LCS	1593	14	0.01336	99.8		1352	40	0.00345	97.4	

Formulas:

Standard TIC % Recovery = $((A-B)/((C/100)*D))*10^{-6}*100$

Standard TOC % Recovery = $((E-F)/((G/100)*H))*10^{-6}*100$

Sample TIC (ug C/ml or ug C/g) = (I-J)/(K*L/100)

Sample TOC (ug C/ml or ug C/g)= (M-N)/(O*P/100)

Matrix Spike Recoveries:

TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U

TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U

TC % Recovery = $((((Q^{TIC}-R^{TIC})/(L/100))-V^{TIC})+(((Q^{TOC}-R^{TOC})/(P/100))-V^{TOC}))*100/U^{TIC+TOC}$

Comments:

Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.

The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.

If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".

TIC and TOC are measured; TC is the sum of the TIC and TOC results.

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client:	Geeting	Analyzer M&TE: WC01713 -- 701
Project :		Balance M&TE: 360-06-01-023
Work Pkg:	CMC	TOC STD: alpha-D-glucose Aldrich CMS#53219(Cal/ICV), Sigma CSM#161713(MS/LCS) 40.00% Carbon <<[G]
Analyzed:	July 17, 2002	TIC STD: Calcium Carbonate Baker CMS#161359(Cal/ICV), Mallinckrodt CSM#161732(MS/LCS) 11.99% Carbon <<[C]
ASR:	6284	

Sample Results

Note: Sample weights are on "as received" basis; i.e., wet weight

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam wt (g)	TIC (ug C/g)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam wt (g)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
02-00829	AZ-0	1847	14	0.1278	14,515		210	40	0.1278	1,362		15,877	
02-00830	AZ-2	909	14	0.0982	9,225		169	40	0.0982	1,345		10,570	
02-00831	AZ-4	2107	14	0.1400	15,129		104	40	0.1400	468		15,597	
02-00832	AZ-6	1085	14	0.1003	10,807		178	40	0.1003	1,409		12,216	
02-00832 MS	AZ-6	2110	14	0.1095	see below		1426	40	0.1095	see below		see below	

(Note: For any TOC or TIC result displayed as "# (<mdl)" the final reported "less than" concentration is calculated by dividing the Method Detection Limit by [K])

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/g)	[T] MS Sam wt (g)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery	
02-00832 MS	TIC Recovery	2110	14	10807	0.1095	1183	0.00679	814	**115.2**	**TIC**
	TOC Recovery	1426	40	1409	0.1095	154	0.00319	1276	**99.2**	**TOC**
	Total Carbon Recovery (TIC + TOC)							2090	**105.4**	**TC**

Reviewer/date: 7/30/02

Battelle

. . . Putting Technology To Work

Client:	John Geeting	Date:	3/11/02
Subject:	Hydroxide Analyses for: 02-0832		
ASR:	6284		

Two samples of **AZ-101 SW/CL Slurry tank waste** were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. These samples were analyzed using a Brinkman 636 Auto-Titrator. A 0.0103 M NaOH (ChemRec_72) solution was used as a standard and sample spike and the titrant used was a 0.02030 M HCl standardized solution. The attached Report Summary indicates good RPD on the OH molarity (1st inflection point) on the sample and replicate results on both samples. The RPD are higher on the sample than on duplicate because the first run volume was 0.5 mL whereas all remaining runs were at 1mL volume. The overall average RPD was about 2% on the 3 - 1mL analyses. The hydroxide results were all well below the required MRQ value of 7.5E+04 ug/mL. The hydroxide standard recovery was 93% on the 2 standards run on this day and the matrix spike recovery was 99%. No hydroxide was detected in the reagent blank or process blank. The second and third inflection points frequently associated with carbonate and bicarbonate, showed excellent RPD, again with excepton to the 1st run (0.5mL vol analyzed) and overall average of 3 - 1mL was < 5% and well below the +/- 20% required. All of the results meet the QC acceptance criteria for spike recovery and RSD of duplicate measurements.

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.

Prepared by: Date: 3/11/02

Reviewed by: Date: 3/11/02

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

ASR	**6284**
WP#	**W60513**

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

Report Summary for ASR # -- 6284

				Concentration, moles / Liter					
RPG #	**Client ID**		OH conc ug/mL	**First Point**	**RPD**	**Second Point**	**RPD**	**Third Point**	**RPD**
	AZ-6								
02-0832	**AZ-101 SW/CL Slurry**	**Rep**	2.1E+03	**0.126**		**0.010**		**0.002**	
02-0832	**AZ-101 SW/CL Slurry**	**Rep**	2.6E+03	**0.152**	19%	**0.008**	27%	**0.003**	27%
02-832-dup	**AZ-101 SW/CL Slurry**		2.7E+03	**0.159**		**0.007**		**0.003**	
02-832-dup	**AZ-101 SW/CL Slurry**	**Rep**	2.7E+03	**0.159**	1%	**0.006**	2%	**0.003**	4%
Overall average (3 -1mL analysis runs) =			**2.66E+03**	**0.157**		**0.007**		**0.003**	

OH conc (ug/mL) = M (g/L) * 17,000

02-0832-BS		**0**
Reag. Blk.1		**0**
Standard 1		**94%**
Standard 2		**92%**
MS 02-832	**Matrix spike**	**99%**

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: 3/11/02

Reviewer: 3/11/02

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # **6284** File: R:\radchem\hydroxide\asr
Client: John Geeting Analysis Da **3/6/02**
WP# W60513 Report Dat **3/11/02**

6284

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

Analyst: 3/11/02

Equip # **WB76843** Lab Loc. 525

	Titrant	Molarity	Rec#	Std. & Spike	Molarity
Strong	HCl	**0.0203**	72	NaOH	**0.0103**

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	
02-0832-BS	process blank			0.5			4	5.959	0.035	4.126	0.001	0.001		
02-0832	AZ-101 SW/CL Slurry		15.4035	0.500	0.5017	1.003	5	10.563	0.201	9.117	0.004	0.126		
02-0832	AZ-101 SW/CL Slurry	Replicate	15.4035	1.000	1.0043	1.004	6	11.200	0.485	9.700	0.0098	0.152	18.71%	
02-832-dup	AZ-101 SW/CL Slurry		15.5545	1.000	1.0016	1.002	7	11.033	0.505	9.332	0.0103	0.159		
02-832-dup	AZ-101 SW/CL Slurry	Replicate	15.5545	1.000	1.0037	1.004	8	11.085	0.502	9.222	0.0102	0.159	0.60%	
												0.1590		
Reag. Blk.1				5.00			2						**Total**	
Reag. Blk.2				5.00		3					**OH Recovery**			
Standard 1	0.0103 N NaOH			5.000	4.964	1	11.953	2.386	7.804	0.0484	**94.1%**			Standard 1
Standard 2	0.0103 N NaOH			5.000	4.9751	14	11.414	2.342	7.579	0.0475	**92.3%**			Standard 2
MS 02-832	+ 2.5mL 0.01N NaOH	2.4967		0.500	0.5011	1.002	9	11.553	1.508	8.742	0.0306	**99.0%**	MS	

Buffer	JT Baker Lot #	CMS#	Expire Date
10	J38505	161304	Sep-02
4	V0510	161306	Jan-03
7	J34512	161305	Aug-02

Initial	pH 7.0 reading =	6.995
Continuing	pH 7.0 reading =	7.009

Performance checks us 360--01-06-037

Pipet #	Vol.	Wt.	Pipet #	Vol.	Wt.
F04171	2.00	1.9987	120737	0.500	0.4997
F04171	2.00	2.0074	120737	0.500	0.5001
F04171	2.00	1.9971	120737	0.500	0.4981

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # 6284

WP# W60513

File: R:\radchem\hydroxide\asr

Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
0
Equip # WB76843

Analyst: 3/11/02

Titrant	Molarity
HCl	0.0203
	0

RPG #		Sample Vol. (mL	2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
02-0832	0	0.500	0.458	6.270	0.005	0.0104		0.513	4.469	0.001	0.00	
02-0832	Replica	1.000	0.875	7.134	0.008	0.0079	27.43%	1.020	4.705	0.003	0.00	27.5%
02-832-dup	0	1.000	0.831	7.048	0.007	0.0066		0.986	4.716	0.003	0.00	
02-832-dup	Replica	1.000	0.820	6.991	0.006	0.0065	2.48%	0.982	4.849	0.003	0.00	4.4%
						2-nd Recovered						
					2-nd Recovered							
	5.000	2.638	4.148	0.00512	9.9%	sample						
	5.000	2.638	4.096	0.00601	11.7%	sample						
MS 02-832		0.500	1.747	7.154	0.00485		2.978	2.029	4.846			

Chem Rec_72

Prep date: 6/21/01

Preparation and Standardization 0.02 and 0.005 M HCl

WP# K88426 for: RPL-CMC- OH analysis

Prepare 1- liter supply of 1M HCL and 0.2M HCl
Calculation: 1000 mL * 1.0 N HCl / 12M HCl = 83.33 mL of 12 N HCl diluted to 1liter with H20.
0.2 M HCl is a 1 : 5 dilution of 1M HCl
Used 83.5 mL reagent grade conc HCl (Barcode # 58914) and diluted to 1000 mL using nanopure (Type II ASTM grade) water. The 0.02M HCl was prepared by diluting 20 mL of 1M HCl to 1Liter
The 0.005 M HCl was prepared by diluting 5 mL of 1M HCl to 1Liter
The 0.02 M HCl will be titrated against standardized 0.1005M NaOH solution (Chem Rec_64), then used to standardized ~0.01M NaOH and then used to set
50 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalien endpoint using the recently standardized 0.1005 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be <0.3% @ 1s. Thus total error @ 1sigma is combined pipetting and titration error (i.e. Molarity error plus pipeting error ~0.3%)

NaOH Molarity veification --- from Chem Rec -64

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.40071	19.5	0.10062	
2	0.43252	21.1	0.10037	
3	0.41742	20.35	0.10044	
		Ave=	**0.10047**	0.00013
			certified value	0.13%

Hydrochloric Acid Molarity

Titration Id.	aliquot of acid	Vol. of **0.1005M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	50.00	10.10	0.0203	
2	50.00	10.04	0.0202	
3	50.00	10.15	0.0204	
		Ave Molarity =	**0.0203**	0.00011
				0.55%

Expires 6-21-2002

using Hydrochloric Acid set Molarity of more dilute NaOH

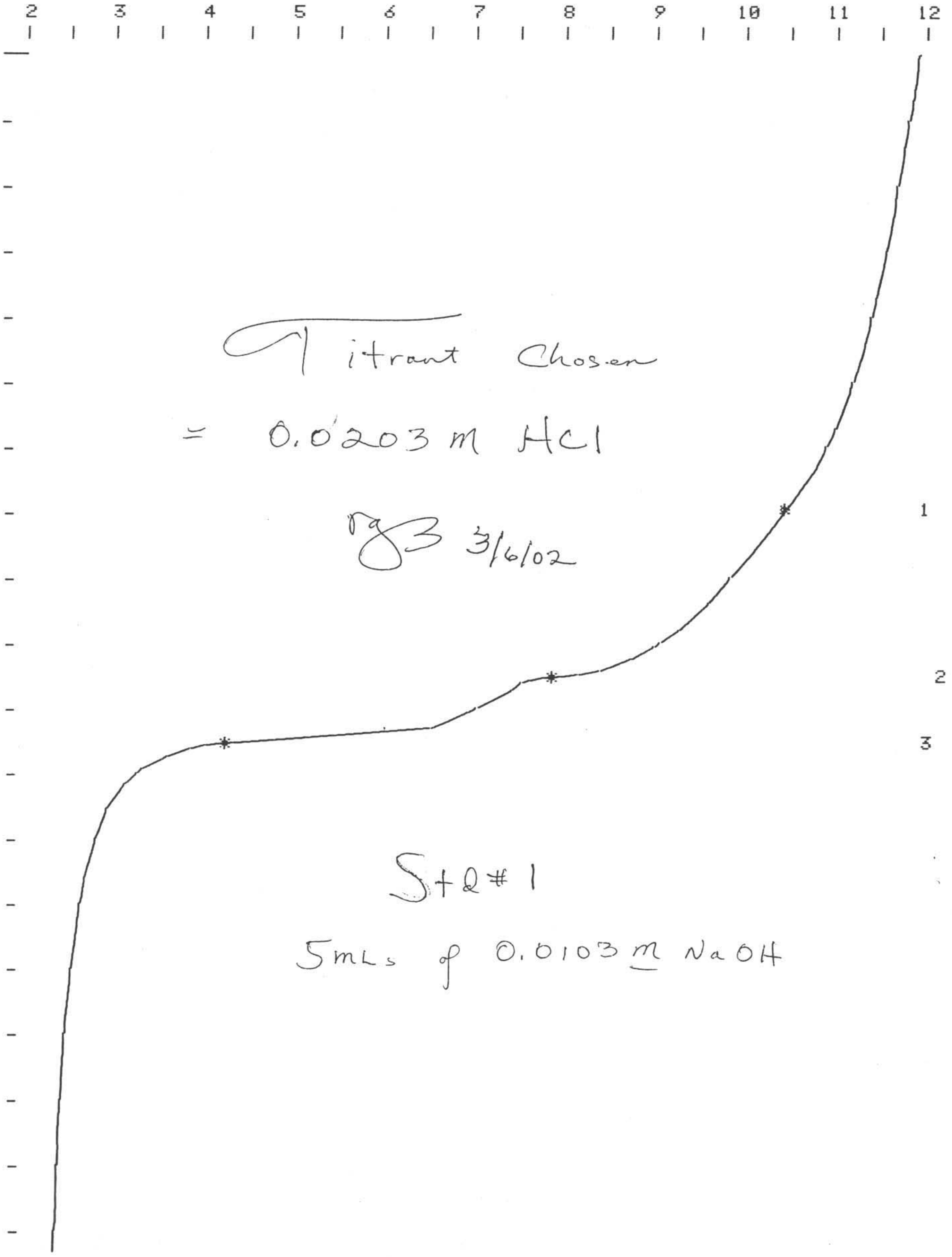
Titration Id.	aliquot of acid	Vol. of **0.01M NaOH** to neutralize	**Molarity of NaOH** in Sample	Molarity Error +/- @ 1 s
1	10.00	19.50	0.0104	
2	10.00	19.80	0.0102	
3	20.00	39.20	0.0103	
		Ave Molarity =	**0.0103**	0.00008
				0.78%

Hydrochloric Acid Molarity

Titration Id.	aliquot of acid	Vol. of **0.0103 M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	20.00	9.95	0.0051	
2	40.00	19.65	0.0051	
3	40.00	19.80	0.0051	
		Ave Molarity =	**0.0051**	0.00003
				0.64%

Analyst/Date rg Swoboda ---- 6-21-2001 3/11/02

```
CALIBR.ELECTRODES 1
PH(S)     4.000   10.000
TEMP/C     25.0    SLOPE     0.968  U(AS)/MV -     5.9

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



Titrant Chosen

= 0.0203 M HCl

3/6/02

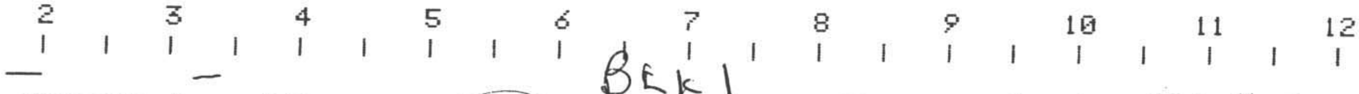
Std #1

5mLs of 0.0103 M NaOH

```
#      1   PH(INIT)  11.953   V(TE)/ML   4.813
1  V/ML    1.747  PH(M)  10.391
2  V/ML    2.386  PH(M)   7.804
3  V/ML    2.638  PH(M)   4.148
```

— Not OH⁻ equivalence pt.

```
                                   DATE    0.00.02   NAME

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



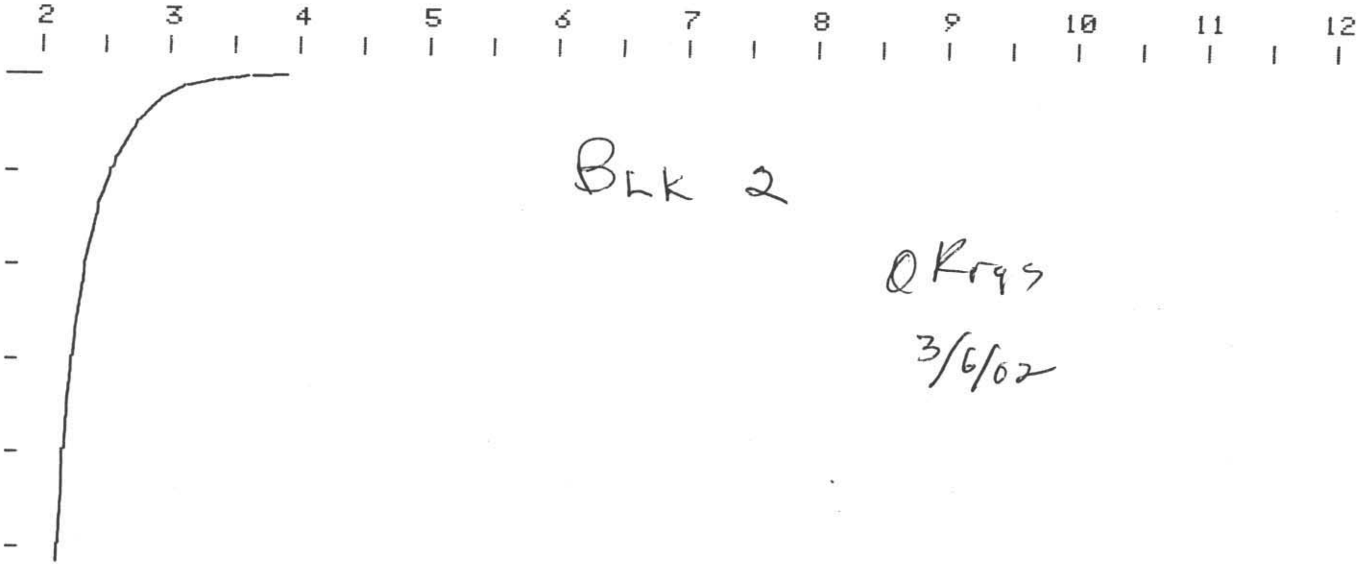
BLK 1

```
ROUTINE #    101
#      2   PH(INIT)   3.303   V(TE)/ML   0.015

                                   DATE    0.00.02   NAME
```

Maybe didn't rinse Acid Purge inlet enough

rgs 3/6/02

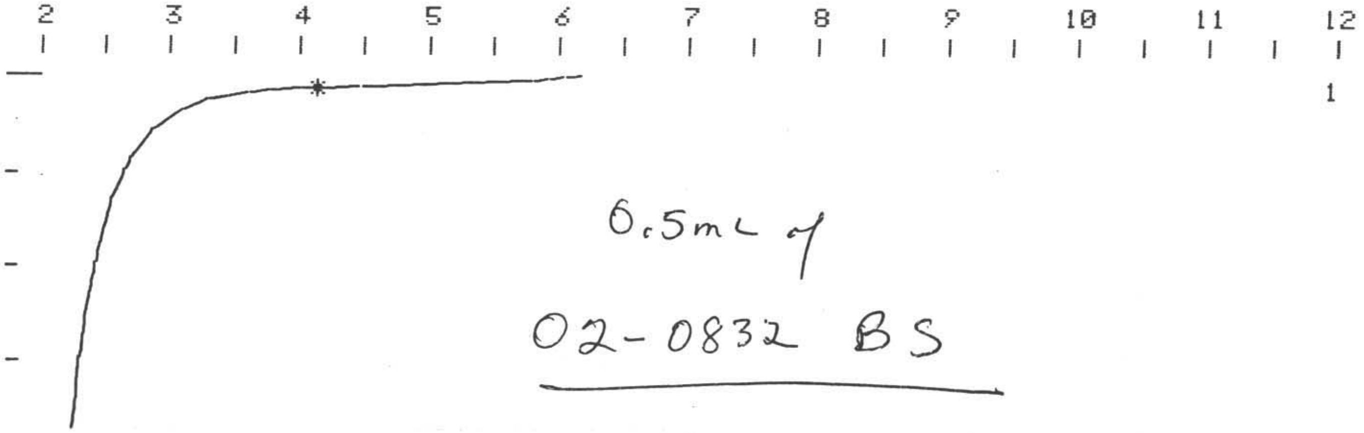
```
    0.25ML/DIV  V(START)/ML     0.000       PH
```



BLK 2

OK rgs 3/6/02

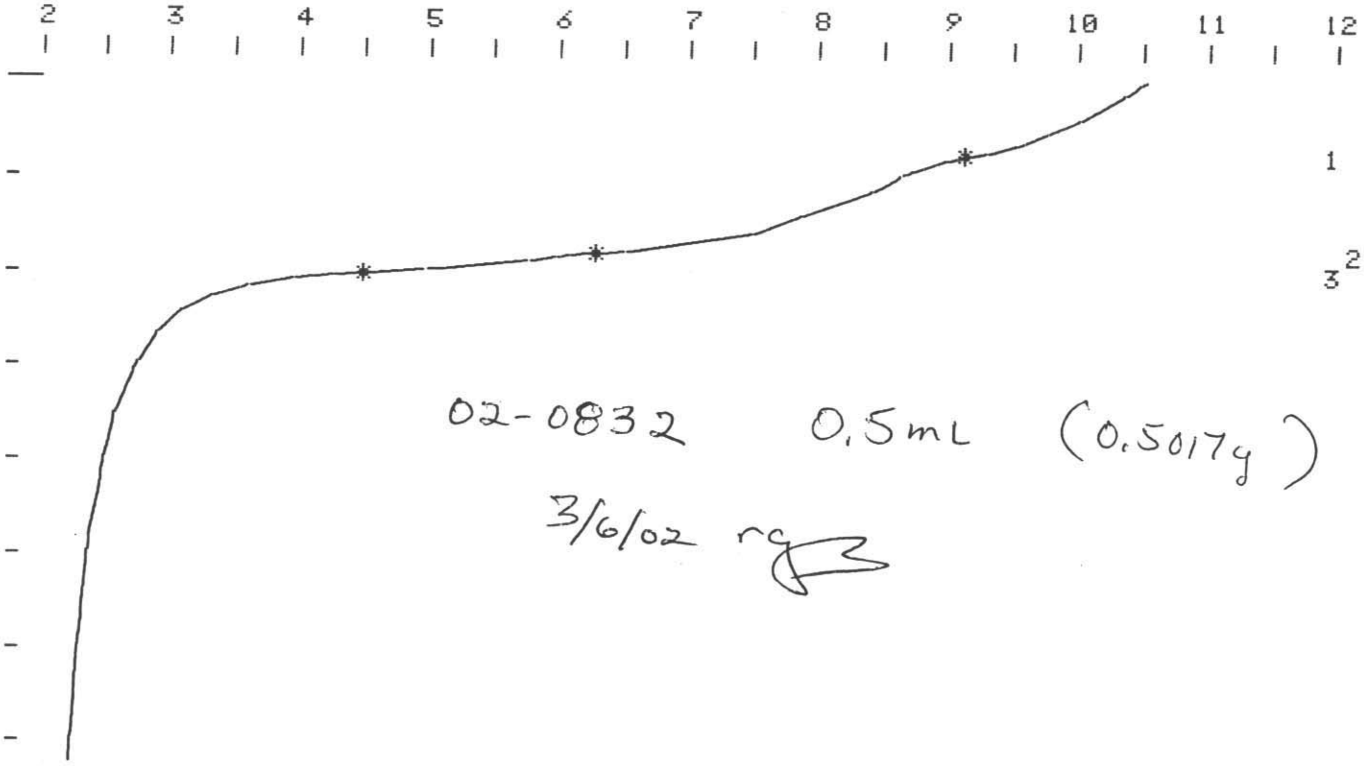
```
ROUTINE #    101
#      3   PH(INIT)   4.255   V(TE)/ML   1.522

                                   DATE    0.00.02   NAME

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



0.5mL of

02-0832 BS

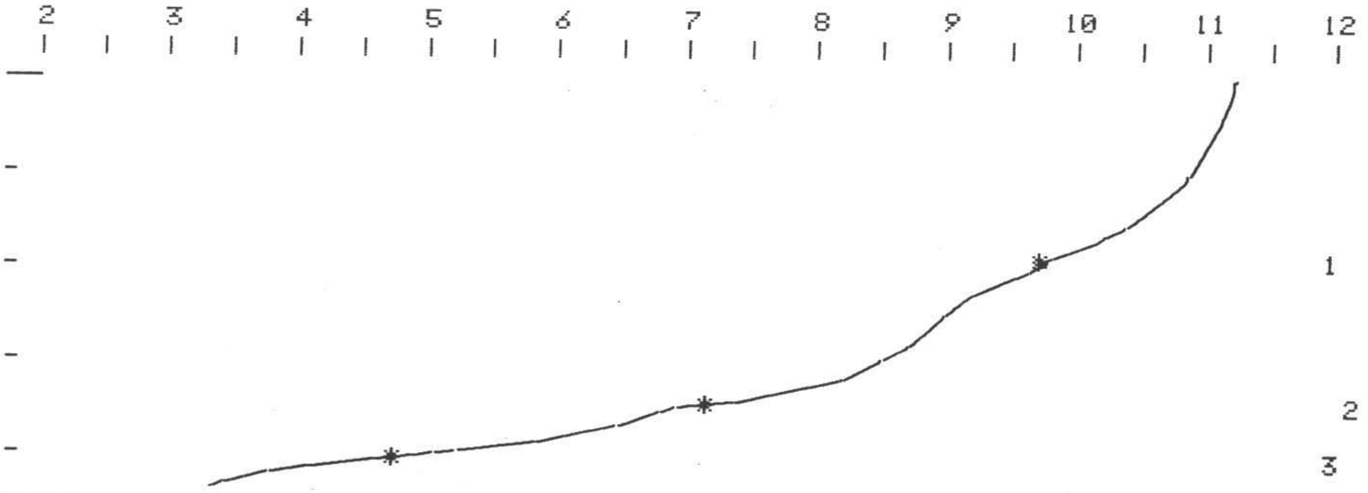
```
ROUTINE #    101
#       4   PH(INIT)   5.959   V(TE)/ML    1.460
1   V/ML    0.035  PH(M)    4.126

                                         DATE     0.00.02    NAME


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



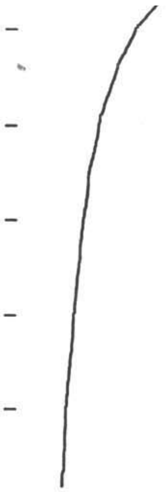
02-0832 0.5mL (0.5017g)

3/6/02 rg

```
ROUTINE #    101
#       5   PH(INIT)  10.563   V(TE)/ML    2.038
1   V/ML    0.201  PH(M)    9.117
2   V/ML    0.458  PH(M)    6.270
3   V/ML    0.513  PH(M)    4.469

                                         DATE     0.00.02    NAME


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



BRINKMANN CAT # 2025015-1

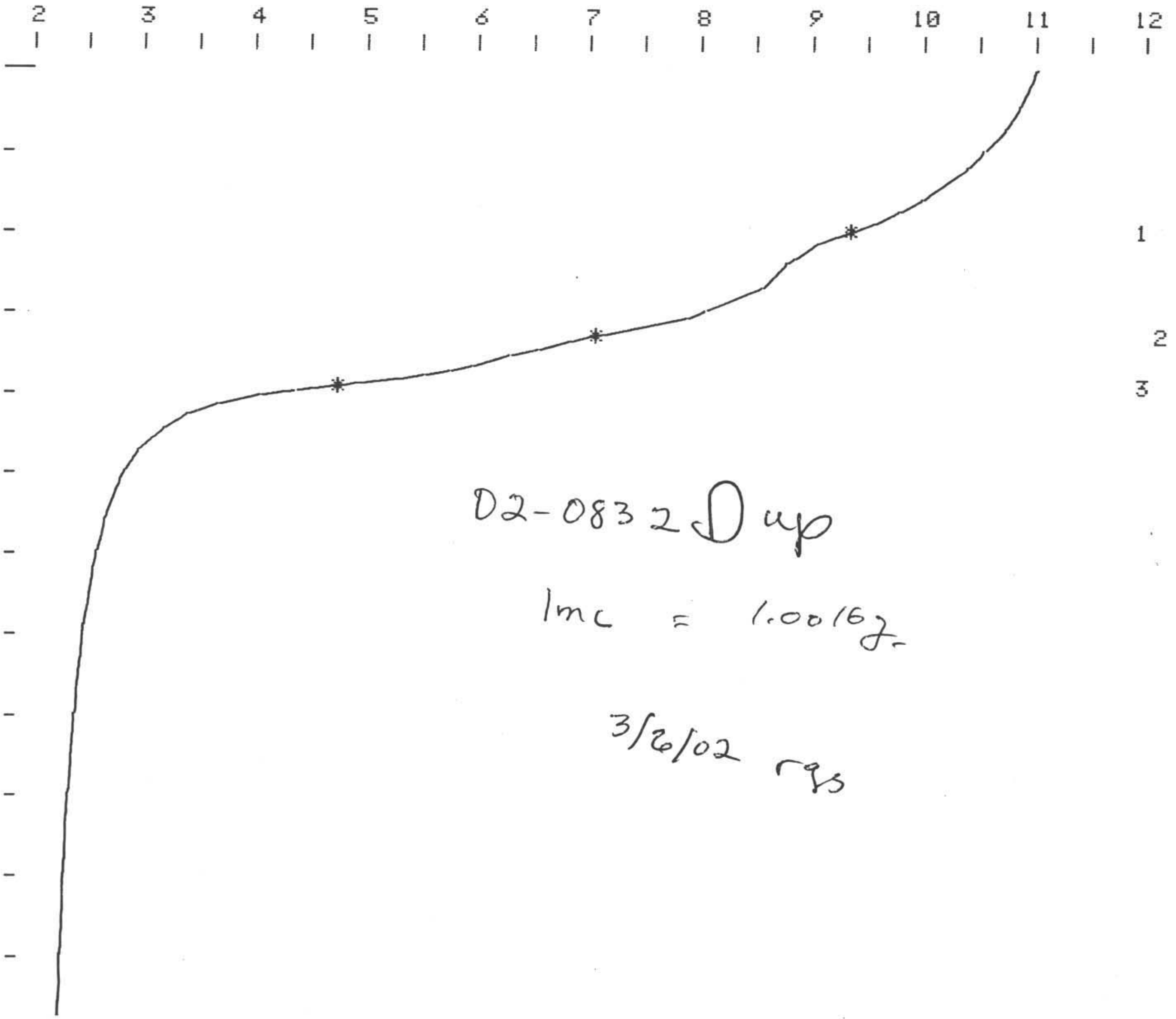


02-0832 Rep

1mL (1.0043g)

3/6/02 rgs

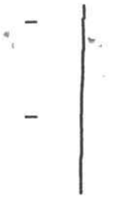
```
ROUTINE #     101
#       6   PH(INIT)  11.200   V(TE)/ML   2.686
1  V/ML   0.485  PH(M)    9.700
2  V/ML   0.875  PH(M)    7.134
3  V/ML   1.020  PH(M)    4.705


                                    DATE    0.00.02    NAME


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



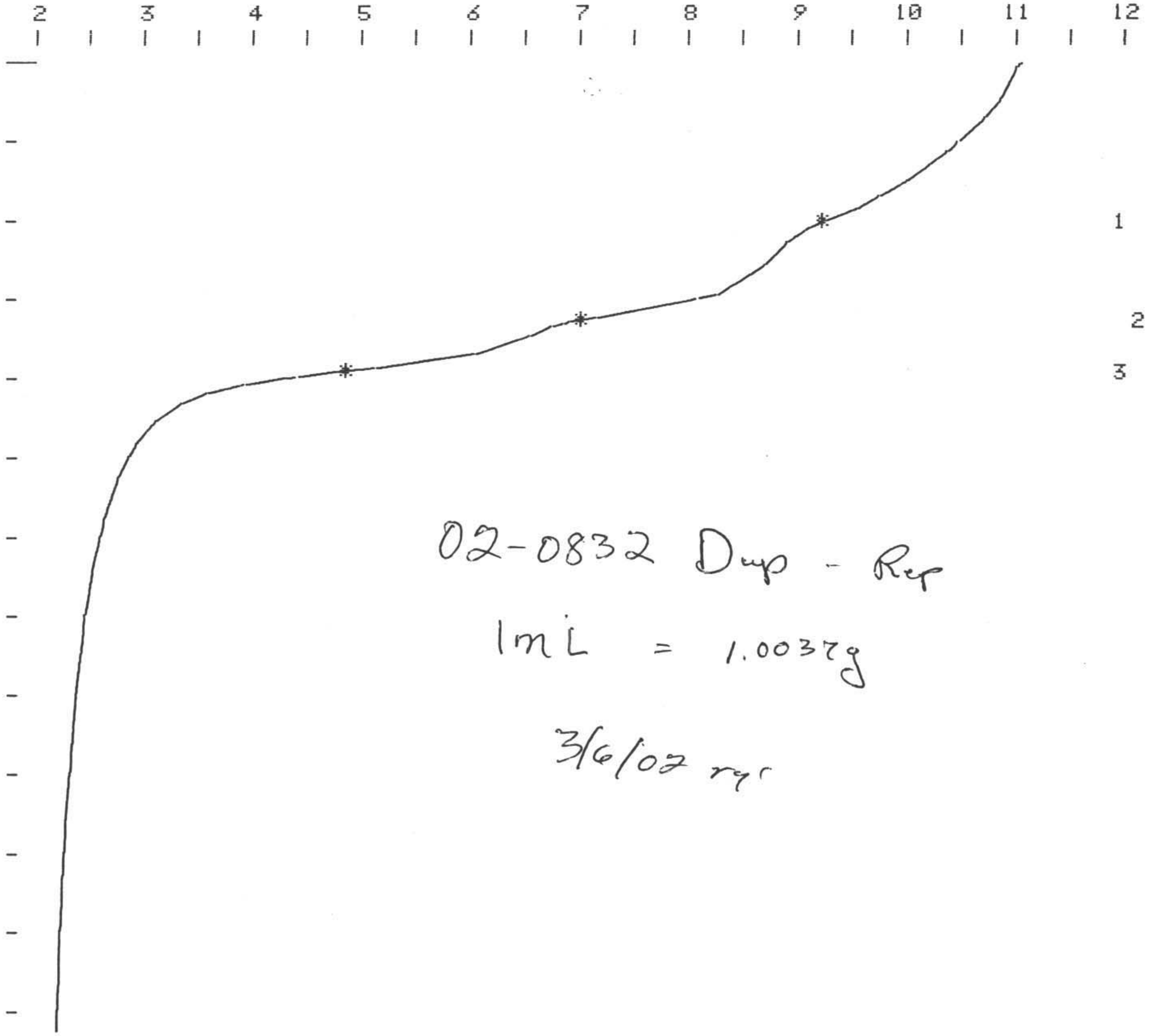
02-0832 Dup

1mL = 1.0016g.

3/6/02 rgs



```
ROUTINE #     101
#       7   PH(INIT)  11.033   V(TE)/ML   3.702
1  V/ML    0.505  PH(M)    9.332
2  V/ML    0.831  PH(M)    7.048
3  V/ML    0.986  PH(M)    4.716

                                          DATE    0.00.02   NAME

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



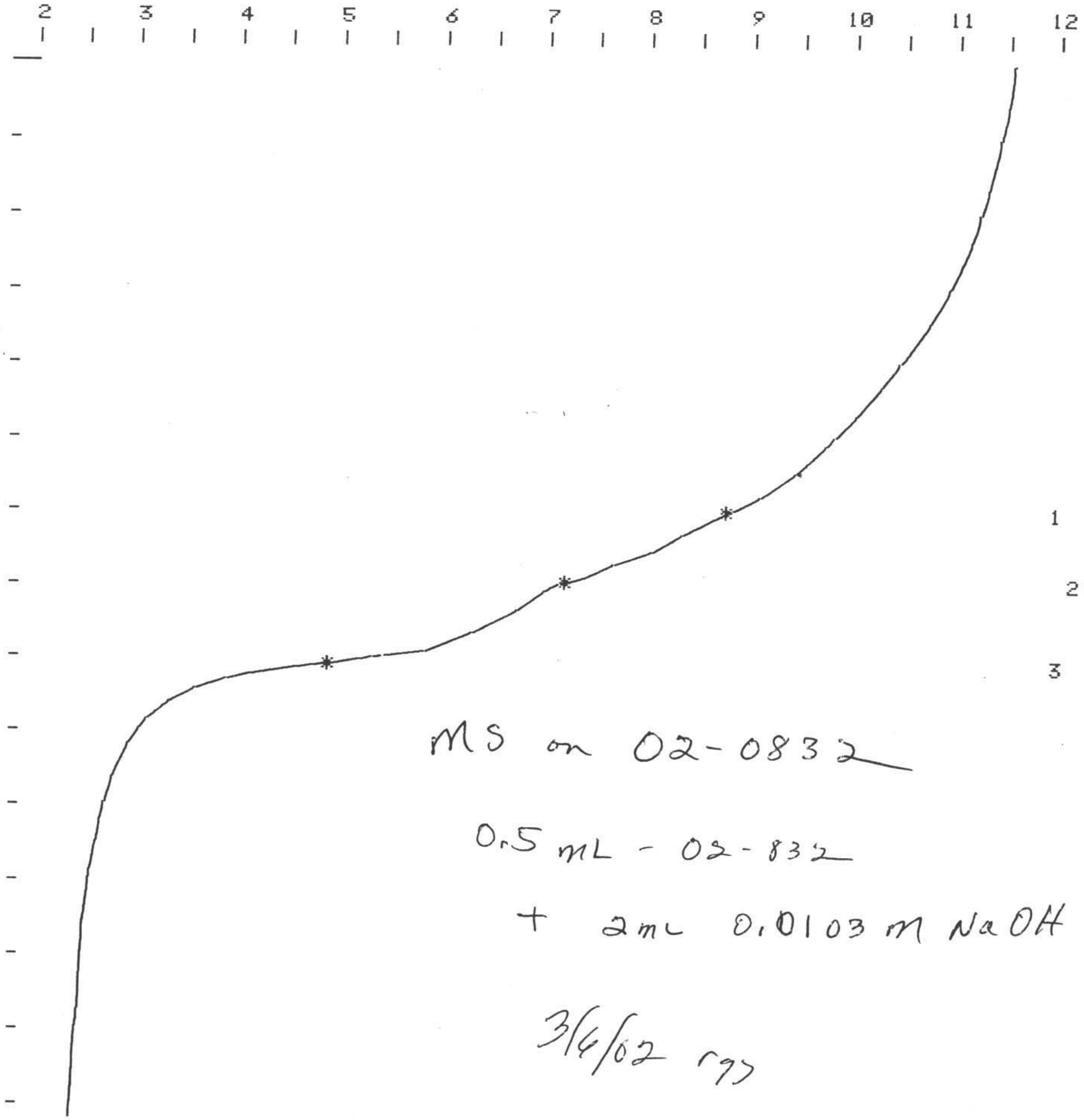
02-0832 Dup - Rep

1mL = 1.0037g

3/6/02 ryc

```
ROUTINE #     101
#       8   PH(INIT)  11.085   V(TE)/ML   3.302
1  V/ML    0.502  PH(M)    9.222
2  V/ML    0.820  PH(M)    6.991
3  V/ML    0.982  PH(M)    4.849

                                          DATE    0.00.02   NAME
```

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



MS on 02-0832

0.5 mL - 02-832

+ 2mL 0.0103 M NaOH

3/6/02 rg

```
ROUTINE #    101
#       9   PH(INIT)  11.553   V(TE)/ML   3.778
1  V/ML   1.508  PH(M)   8.742
2  V/ML   1.747  PH(M)   7.154
3  V/ML   2.029  PH(M)   4.846

                                    DATE     0.00.02     NAME
```

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Radiological Processing Group (RPG)

Inorganic Analysis - Mercury Data Report

Project / WP#: 42365 / K88408
ASR#: 6284
Client: John Geeting
Total Samples: 4

RPL #	Client ID
02-829	AZ-0
02-830	AZ-2
02-831	AZ-4
02-832	AZ-6

DRY BASIS

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number:	WD30853	CETAC, Mercury Analyzer, Model M-6000A
	1113052270	Mettler AT400 Balance

Digestion Date: 6/12/02
Analysis Date: 6/12/02

Analysis File: 02061202.DB

Analyst: LMP Thomas

For Calibration and Maintenance Records, see Chemical Measurement Center 98620 RIDS

LMP Thomas 7-5-02
Prepared By

7-24-02
Reviewed By

Four slurry samples were submitted for mercury analysis. The samples were aliquoted and prepared by digestion in the hot-cell, and analyzed by cold vapor atomic absorption spectroscopy (CVAA) in a fume hood. Preparative and analytical quality control included a preparation blank, laboratory control standard, sample, duplicate, and matrix spike.

1. Analysis

Results from the analysis of the slurry samples are provided in the table below. The concentration is reported in μg of mercury per g of sample.

RPD success criteria: < 20%

RPL ID	Sample ID	Det Lim μg/g	Measured μg/g	Average μg/g	RPD %
02-829	AZ-0	0.036	9.45	6.86	
02-829	AZ-0 DUP	0.030	4.28		75
02-830	AZ-2	0.041	14.1		
02-831	AZ-4	0.041	17.5		
02-832	AZ-6	0.049	21.2		
Est MDL (1)			0.033		
EQL (2)			0.22		
Preparation DF (L/g)			212		
Analysis DF (L/g)			20		

DF - dilution factor

(1) The estimated MDL is based on an MDL evaluated for solid samples (ASR 6145) adjusted by the appropriate dilution factors.

(2) The EQL is based on the lowest calibration standard, 0.05 μg/L, multiplied by the total dilution factor.

2. Quality Control

Duplicate (DUP). The RPD of the sample duplicate is the only QC sample which did not meet the QC success criteria. Possible causes may be due to the sample preparation or the sample matrix. During the sample preparation, none of the digests retained the purple color (due to the potassium permanganate reagent). The persistence of the purple color may be an indication that the digests are providing the oxidizing environment needed during sample preparation. However, the LCS met the QC criteria, which may indicate the digestion was adequate. A more likely cause for the QC failure is the sample matrix. Very small sample sizes were used due to ALARA considerations. The samples were aliquoted as hard chunks and could not be mixed to homogenize. There may also be components in the sample that interfere with the preparation.

Matrix Spike (MS). The matrix spike recovery meets the QC success criteria. A post spike was performed on the matrix spike sample and it also meets the QC success criteria.

MS success criteria: 75% to 125% of expected value

RPL ID	Sample ID	Det Lim μg/g	Spike μg/g	Sample μg/g	Measured μg/g	Recovery %
02-829	AZ-0 MS	0.034	9.85	6.86	17.5	108
02-829	AZ-0 MS PS	0.067	21.5	17.5	38.5	97

Preparation Blank (PB) and Laboratory Control Standard (LCS). The results of the PB and LCS analyses are presented in the table below. The results of the PB are based on the average sample size. The PB and LCS meet the success criteria.

Sample ID	PB success criteria: < EQL		LCS success criteria: 80% - 120% of expected value		
	Success Criteria µg/g	Measured µg/g	Expected µg/g	Measured µg/g	Recovery %
PB/LCS liquid	< 0.22	< MDL	1.40	1.51	108

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

ICB/CCB success criteria: < EQL		
Sample ID	Criteria µg/L [(1)]	Measured µg/L [(1)]
ICB	< 0.05	< 0.05
CCB 1	< 0.05	< 0.05
CCB 2	< 0.05	< 0.05
CCB-3	< 0.05	< 0.05
CCB 4	< 0.05	< 0.05

(1) Units are based on per liter of sample at the instrument.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

ICV/CCV success criteria: 80% to 120% recovery Calibration Range: 0 - 5 µg/L		
Sample ID	Measured µg/L(1)	Recovery %
Expected	2.00	
ICV	2.02	101
CCV 1	2.00	100
CCV 2	1.96	98
CCV-3	1.99	99
CCV-4	1.96	98

(1) Units are based on per liter of sample at the instrument.

Low-Level Standard (LLS). The LLS meets the success criteria.

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.05 µg/L		
Expected µg/L(1)	Measured µg/L(1)	Recovery %
0.050	0.046	93

(1) Units are based on per liter of sample at the instrument.

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on detection limit studies using water (for the determination of the detection limit in liquid matrices) and sand (for the determination of the detection limit in solid matrices) and documented in ASR 6145. The estimated quantitation limit (EQL) is defined as the lowest calibration standard.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Radiological Processing Group (RPG)

Inorganic Analysis - Cyanide Data Report

Project / WP#: 42365 / W60513
ASR#: 6284
Client: John Geeting
Total Samples: 4

RPL #	Client ID
02-829	AZ-0
02-830	AZ-2
02-831	AZ-4
02-832	AZ-6

DRY BASIS

Procedure: PNL-ALO-287 Rev. 0, *Midi and Micro Distillation of Cyanide in Liquid and Solid Samples*
PNL-ALO-289 Rev. 0, *Total Cyanide Determination by Spectrophotometry (Manual or Automated) or Argentometric Titration*

M&TE Number:	WC36517	Lachet QuikChem Analyzer
	1113052270	Mettler AT400 Balance

Analyst: LMP Thomas

Analysis Date: 3/6/02, 7/7/02

Analysis Files: Calibration - 02030601, 02070701
Sample Trays - 02030603.RS, 02030605.RS, 02030609.RS
02070701.RS, 02070703.RS, 02070704.RS

For Calibration and Maintenance Records, see Chemical Measurement Center 98620 file:

LMP Thomas 7-9-02
Analyst

7/12/02
Reviewed By

Four samples, AZ-101 slurries, were submitted for cyanide analysis. The samples were aliquoted and prepared by micro-distillation in a hot cell, and analyzed by automated spectrophotometry in a fume hood. Quality control samples included a preparation blank, laboratory control standard, duplicate, and matrix spike. Quality control check standards relevant to the instrument performance were also prepared and analyzed.

1. Analysis

The results from the analysis of the AZ-101 slurry samples are provided in Table 1 below. The concentrations are reported in µg of cyanide per g of sample. Because some of the quality control samples did not meet the QC success criteria, a second run was performed. However, there was insufficient material of the AZ-6 sample to perform a second analysis.

Table 1. Cyanide Results for AZ-101 Slurry Samples

RPD success criteria: < 15%							
RPL ID#	**Sample ID**	**Measured µg/g**	**Average µg/g**	**RPD %**	**Measured µg/g**	**Average µg/g**	**RPD %**
		Run 1			*Run 2*		
02-829	AZ-0	< MDL	1.01	200	10.2	10.2	0.67
02-829	AZ-0 DUP	2.01			10.1		
02-830	AZ-2	4.82			7.00		
02-831	AZ-4	1.49			2.50		
02-832	AZ-6	7.14					
	Est. MDL(1)	0.11			0.16		
	Target MRQ	3.0			3.0		
	EQL(2)	0.40			0.60		
	Preparation DF (mLs/g)	40			60		
	Analysis DF (v/v)	1			1		
	Total DF	40			60		
	The MDL (at the instrument) is 0.00263 mg/L						

DF = dilution factor

(1) The estimated MDL is based on an MDL evaluated for sand samples (ASR 6091) adjusted by the appropriate dilution factors.

(2) The EQL is based on the lowest calibration standard, 0.01 mg/L, multiplied by the total dilution factor.

2. Quality Control Comments

Duplicate (DUP). The duplicate QC success criterion was met in the second run, but not the first run. In previous analysis of tank waste material, cyanide losses have been observed. Normally, sulfamic acid (to reduce the nitrite and nitrate interference) and sulfuric acid (releasing agent) are added to the sample tube (containing the sample), after which the sample tube is capped with the distillation tube containing the trapping solution. With some tank waste materials, the reaction between the sample and releasing agent is so vigorous, the cyanide may be released before the distillation tube can be capped onto the sample tube or the trapping solution may not have enough time to capture all of the cyanide. In Run 1, the releasing agent was added in little vials which floated in the sample tube and mixed into the sample after the distillation tube was secured over the sample tube. However, a reaction between the sulfamic acid (which was added directly to the sample prior to the releasing agent) and sample was observed, which may account for the loss of cyanide. In Run 2, the sulfamic acid and releasing agent were added in little vials and every effort was made to mix these reagents slowly into the sample.

Matrix Spike (MS). A matrix spike of the AZ-0 sample was prepared and analyzed. The spike recovery met the QC success criterion for the second run, but not the first run.

Table 2. Cyanide Results for the AZ-101 Slurry MS Sample

MS success criteria: 75% to 125% Recovery					
RPL ID	**Sample ID**	**Measured µg/g**	**Sample µg/g**	**Spike Expected µg/g**	**Rec %**
02-829	AZ-0 MS - Run 1	< MDL	1.01	7.40	0
02-829	AZ-0 MS - Run 2	18.7	10.2	7.33	116

Preparation Blank (PB) and Laboratory Control Standard (LCS). Table 3 presents the PB and LCS results, which meet their respective QC success criteria, except for the LCS in Run 2. The PB is a 0.25 M NaOH solution and the results are based on the average of the sample sizes. The LCS is a soil (ERA Priority PollutnT) containing a certified concentration of 177 µg/g of cyanide (71 - 301 µg/g advisory range). The LCS in Run 2 is outside the success criteria of its certified value, but within the vendor's advisory range. Because the concentration of cyanide is so high, only a very small amount of standard is prepared, which may account for the variability seen in the results. Both PB and BS samples were distilled.

Table 3. Cyanide Results for the PB and LCS

PB sucess criteria: < EQL			**LCS success criteria: 80% to 120% Recovery**		
Sample ID	**Success Criteria µg/g**	**Measured µg/g**	**Measured µg/g**	**Expected µg/g**	**Recovery %**
Run 1	< 0.40	< MDL	154	177	87
Run 2	< 0.60	< MDL	224	177	126

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards are a 0.25 M NaOH solution and were not distilled. The results of the ICB/CCB standards meet the QC success criteria of < EQL and are presented in Table 4.

Table 4. Cyanide Results for the ICB and CCB Standards

ICB/CCB success criteria: <EQL	
Sample ID	**Measured mg/L (1)**
Run 1	
ICB	< MDL
CCB 1	< MDL
CCB 2	< MDL
Run 2	
ICB	< MDL
CCB 1	< MDL
CCB 2	< MDL

(1) Units are based on per liter of sample at the instrument. The MDL at the instrument is 0.00263 mg/L

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. Calibration verification standards are prepared from a KCN salt and were not distilled. The results of the ICV/CCV standards are presented in Table 5 and meet the QC success criteria of 85% to 115% recovery.

Table 5. Cyanide Results for the ICV and CCV Standards

ICV/CCV success criteria: 85% to 115% Recovery **Calibration Range: 0 - 0.30 mg/L**				
Sample ID	**ICV/CCV - low**		**ICV/CCV - high**	
	Measured mg/L [1]	**Recovery %**	**Measured mg/L [1]**	**Recovery %**
Run 1				
Expected Value	0.0753	n.a.	0.224	n.a.
ICV	0.0711	94	0.211	94
CCV-1	0.0744	99	0.224	100
CCV-2	0.0741	98	0.224	100
Run 2				
Expected Value	0.0754	n.a.	0.226	n.a.
ICV	0.0754	100	0.221	98
CCV-1	0.0754	100	0.221	98
CCV-2	0.0757	100	0.223	99

n.a. = not applicable

(1) Units are based on per liter of sample at the instrument.

Low-Level Standard (LLS). A 0.01 mg/L standard, prepared from a KCN salt, was used as the LLS. This standard was not distilled and was analyzed only once per analytical batch. The results for the LLS are presented in Table 6 and meet the QC success criteria of 75% to 125% recovery.

Table 6. Cyanide Results for the LLS

LLS success criteria: 75% to 125% recovery **Lowest calibration std: 0.010 mg/L**			
Sample ID	**Measured mg/L [1]**	**Expected mg/L [1]**	**Recovery %**
Run 1	0.0079	0.0099	80
Run 2	0.0103	0.0101	102

(1) Units are based on per liter of sample at the instrument.

3. Comments

a). The cyanide results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

Title: *Evaluation of Chelators and Degradation Products in Tank Matrix* *Page* 1 of ~~5~~ 6 RS 7-29-02

Battelle, PNNL / RPG / Organic Analysis … Chelator Data Report

Project / WP#: 42365 / W60513
ASR#: 6284
Client: John Geeting
Total Samples: 1 (in duplicate)

RPL #	Client ID
02-0832	AZ-6

DRY BASIS

Procedure: TP-RPP-WTP-049, *Ion Exchange for Activity Reduction*
TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products*

M&TE Number:		
	Gas Chromatograph/Flame Ionization Detector	WD14807
	Mettler PC4400 Balance	SN: 41100
	Mettler AC100 Balance	SN: 821319

Analyst(s): BR Valenzuela and AM Aman

Analysis Date: 6/4,11/02

Analysis Files:
Calibration – 060402CH
Sample Analysis - 060402CH
061102CH (reruns)

For Calibration and Maintenance Records, see Calibration Data Packet 060402CH and Instrument Logbook

for
Blandina Valenzuela
Prepared By 7-29-02

7/21/02
Reviewed By

CHELATOR RESULTS

1. Sample Analysis
AZ-6 Results

			Sample		Duplicate	
Analyte	**CAS #**	**MDL mg/kg**	**02-0832 mg/kg**	**Data Flag**	**02-0832D mg/kg**	**Data Flag**
Chelators and Degradation Products						
EDTA	60-00-4	4.9	4.9	U	4.9	U
HEDTA	150-39-0	8.8	8.8	U	8.8	U
ED3A(a)	(b)	4.9	4.9	U	4.9	U
NTA	139-13-9	5.6	5.6	U	5.6	U
NIDA/IDA(c)	25081-31-6	11	11	U	11	U
Citric acid(d)	77-92-9	5.8	5.8	U	5.8	U
Succinic acid	110-15-6	6.1	110		100	
AA (surrogate)	124-04-9	(e)	67%(e)		83%(e)	

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) IDA completely converted to NIDA in the presence of nitrite in tank waste.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids.Value represents percent recovery of the surrogate standard.
(e) Value represents percent recovery of the surrogate standard; no MDL calculated.

Narrative

Analysis was performed on the "AZ-6" for chelators and chelator-degradation products. The chelators, ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), ethylenediaminetriacetic acid (ED3A), iminodiacetic acid (IDA), nitrosoiminodiacetic acid (NIDA), succinic acid, and nitrilotriacetic acid (NTA), have low volatility and high polarity precluding direct analysis by GC/FID. Derivatizing the chelators with a BF_3/methanol mixture results in a methyl ester product that is amenable to GC/FID separation and analysis. The derivatization process and analysis are still considered experimental.

Samples of "AZ-6" were submitted for chelator analysis. An approximate 1-gram sub-sample (in duplicate) of AZ-6 was diluted with 5 mL of DI water and allowed to mix overnight. The following day the sample was centrifuged and the water decanted in to a pre-weighed sample vial and subjected to an IX procedure, TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* to reduce the sample dose. The diluted AZ-6 samples were removed from the hot cell and derivitization of the samples according to procedure TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products* was performed in a fume hood in 329. Adipic acid was added to 2-mL aliquots of each sub-sample (prior to derivatization step) as a derivatization monitor. The analysis of these samples included a duplicate. Only succinic acid was present in the samples. Quality control check standards relative to the sample preparation and instrument performance were also prepared and analyzed.

The EQL was calculated using the lowest calibration standard (10 mg/kg; 20 mg/kg for NIDA) and multiplied by the preparation dilution factor recorded for the batch preparation blank.

Historically, the EQL was 10 times the value of the estimated MDL. Therefore, for the current analysis the MDL will be calculated by dividing the EQL by 10. The results above the MDL and below the EQL will be "J" flagged.

2. Quality Control Criteria

MS and MSD QC Results

Analyte	CAS #	MDL (mg/kg)	02-0832 Average Native Amt. (mg/kg)	Data Flag	Spiked Conc. (MS/MSD) (mg/kg)	Analyzed Conc. (mg/kg)	Matrix Spike (MS) % Rec	Analyzed Conc. (mg/kg)	Matrix Spike Duplicate(MSD) % Rec.
Acceptance Criteria							*75-125*		*75-125*
Chelators									
EDTA	60-00-4	4.9	4.9	U	530/490	550	104	500	102
HEDTA	150-39-0	8.8	8.8	U	1100/980	1200	116	1000	106
ED3A[a]	(b)	4.9	4.9	U	(c)		(c)		(c)
NTA	139-13-9	5.6	5.6	U	660/610	720	109	720	119
NIDA	25081-31-6	11	11	U	670[d]/620[d]	82	**12**	81	**13**
Citric Acid [e]	77-92-9	5.8	5.8	U	660/610	630	95	610	100
Succinic Acid	110-15-6	6.1	100		670/620	690	88	670	90
AA (surrogate)	124-04-9	(f)	75%[f]				108 [f]		106 [f]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

Bolded values denote acceptance criteria failures

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A not spiked into MS and MSD samples.
(d) Assumes spiked IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.
(e) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(f) Value represents percent recovery of the surrogate standard; no MDL calculated.

Process Blank and LCS Results

Analyte	CAS #	MDL (mg/kg)	Process Blank (mg/kg)	Data Flag	Spiked LCS concentration (mg/kg)	Analyzed LCS concentration (mg/kg)	Data Flag	Lab Control (LCS/BS) % Rec.
Acceptance Criteria								*80-120*
Chelators								
EDTA	60-00-4	4.9	4.9	U	480	440		90
HEDTA	150-39-0	8.8	8.8	U	970	900		92
ED3A(a)	(b)	4.9	4.9	U	(c)	(c)		(c)
NTA	139-13-9	5.6	5.6	U	610	610		101
NIDA	25081-31-6	11	11	U	(c)	(c)		(c)
Citric Acid (d)	77-92-9	5.8	5.8	U	610	510		84
Succinic Acid	110-15-6	6.1	6.1	U	620	560		91
AA (surrogate)	124-04-9	(e)	100%(e)					106 (e)

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A and NIDA not spiked into LCS samples; no nitrite from tank sample to convert IDA into NIDA.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(e) Value represents percent recovery of the surrogate standard; no MDL calculated.

Narrative

The preparation blank results meet the success criteria of <EQL.

For the solid batch chelator preparation, AZ-6 (RPL#: 02-0832) was used for the matrix spike and duplicate. Of the seven analytes, only the NIDA results failed the spiked recovery criterion. This recovery failure can be explained for the MS and MSD. An AZ-6 sample size of approximately 0.5 grams was aliquoted for each of the MS and MSD and then extracted with 5 mL of deionized water prior to being subjected to the IX procedure, TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* to reduce the sample dose. By performing the water extraction step with the solid sample first (and not using the actual tank waste), the nitrite concentration was not high enough to completely convert the IDA in the matrix spiking solution to NIDA. Therefore, a low recovery for NIDA was expected.

The LCS results passed the recovery acceptance criteria (80-120%) without exception.

3. Recommendations

Although HEDTA results met the acceptance criteria in this analytical batch, recoveries of that compound were higher than the acceptance limit in other batches. In those batches, HEDTA results were X flagged and should be considered qualitative. In the reaction of chelators with BF_3/methanol, methylation occurs at the carboxylic acid ligand sites. However, methylation does not occur at hydroxy sites in the reaction of BF_3/methanol, for example with HEDTA. The hydoxyethyl group of HEDTA resists methylation by BF_3/methanol and, instead, forms a cyclic, or intramolecular lactone with one of the neighboring ligand sites. Depending on the pH, the lactone accounts for only approximately 30% of the species detected. The trimethylated non-cyclic HEDTA with a free hydroxyethyl group accounts for the remaining 70% of the HEDTA. It is speculated that the non-cyclic species is simply too polar to migrate through the GC column (Lokken et al. 1986; Grant, Mong, Lucke, and Campbell 1996) and, hence, is not detected in this analysis.

Therefore, the recovery of HEDTA is greatly affected by the pH of the solution. A slight change in the final pH may greatly affect the recovery of HEDTA. It is possible that the conditions under which this particular batch was derivatized closely matched the conditions under which calibration standards were derivatized, resulting in acceptable recoveries.

To increase the reliability of this analytical method, further research is required to understand the variation in recoveries, particularly for HEDTA.

An isotope dilution approach where deuterated analogs of the chelators could be added to the solution prior to sample workup is recommended. Analysis of the samples would then be performed using GC/MS after derivatization. Additional compounds to monitor extraction and sample preparation performance may be advisable.

Existing techniques for EDTA and HEDTA using Cu complexation and ion-pair chromatography (W.R. Grace 1988) could be employed to further validate the performance of this method. This technique could also be used as a confirmatory method for sample matrices although substantial ALARA limitations exist when using liquid chromatographic methods. Additionally, preliminary results using capillary electrophoresis appear promising for the direct analysis of chelators and their associated degradation products without the use of derivatization.

References

Grant KE, GM Mong, RB Lucke, JA Campbell. 1996. Quantitative Determination of Chelators and Their Degradation Products in Mixed Hazardous Wastes from Tank 241-SY-101 Using Derivatization GC/MS. J. of Radioanalytical and Nuclear Chemistry, 211, 383-402.

Lokken RO, RD Scheele, DM Strachan, AP Toste. 1986. Complex Concentrate Pretreatment FY 1986 Progress Report. PNL-7687, Pacific Northwest Laboratory, Richland, Washington.

Organic Chemical Division, General Procedure for the Determination of NTA, HEDTA, EDTA, and DTPA in Chelater and Metal Chelate Mixtures. W.R. Grace and Company, Nashua, New Hampshire, RDSAP Number 0021, 1988, pg. 1.

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Advanced Organic Analysis Group (AOAM)

Organic Anions by Ion Chromatography Report

Project / WP#: 42365 / W60513
ASR#: 6284
Client: John Geeting
Total Samples: 1

RPL #	Client ID
02-00832	AZ-6

Procedure: TP-RPP-WTP-024, *MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste.* (See narrative for adaptation descriptions)

M&TE Number:

WD12888	Dionex IC Analyzer System
P37596	Mettler AE50 Balance

Analyst: GM Mong

Analysis Date: 7/10/02

Analysis Files:
Data: gm0710E01, gm0711E1,
Standards: BNW14062, p. 49
Spreadsheets: IC Std 14062-49a asr6284
IC Std 14062-49b asr6284

Jerry Mong 8-9-02
Prepared By

Blandina Valenzuela
Reviewed By 8-9-02

One sample, AZ-6, was submitted for organic anion analysis. The Radiological Processing Group (RPG) prepared the samples by dilution with 5 mLs of DI water, leaching for 2 hours, and elution through a bed of Bio-Rad AG 50W-X8 cation exchange resin (50-100 mesh, sodium form) to relieve the samples of some of their fission product activity. Ion chromatography was performed with 2 separations by using 2 columns, which had different loading characteristics. The first separation, using a Dionex AS-15 column, was for the determination of glycolate and acetate. The second separation, using a Dionex AS-11 column, was for the determination of formate, oxalate, and citrate. The analytical dilutions of the ion exchange eluant (IEX) solution chosen, were those determined to be within the capacity of the columns. The dilutions of the IEX solutions were 400μL/25 mL (62.5x) for the AS-15 separation and 200 μL/25 mL (125x) for the AS-11 separation. The analytical dilutions were sample dependent and were a principle effect on the overall method MDL estimation.

The results were corrected for the density of the IEX solution (1.025 g/mL). All other dilutions were done on a v/v basis.

This work utilizes the QC acceptance criteria developed in test plan TP-RPP-WTP-024 MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste. The analyte list for this work differs substantially from that developed for the test plan; consequently, the analytical method has to be adapted to meet these new requirements.

One of the requested analytes (gluconate) cannot be reliably determined by the analysis developed here. Two critical analytical hurdles prevent direct analysis for gluconate: (1) Gluconate suffers from low sensitivity to conductivity detection. (2) Gluconate co-elutes with other anions native to tank waste materials in the analytical systems used. For the AS-11 column, gluconate and fluoride are both nearly non-retained and co-elute. For the AS-15 separation, there is great selectivity for the weakly retained analytes fluoride, glycolate, and acetate; however, gluconate was found to co-elute with glycolate. This point will be addressed in the comments below.

1. Analysis

The results from the analysis of the Tc-AP1-EFF comp sample are provided in Table 1 below. The concentration values are given in mg/L and are corrected for the density of theIEX solution. The data reported are the average of duplicate injections.

Table 1. Organic Anion Results for Tc-AP1-EFF comp (01-01613)

RPD success criteria: < 20% when > 10x MDL
RSD success criteria: < 15% when > 10x MDL

Sample ID	Glycolate(1) mg/L	Acetate mg/L	Formate mg/L	Oxalate mg/L	Citrate mg/L
AZ-6	<100 U	<130 U	<170 U	<240 U	<460 U
AZ-6 DUP	<100 U	<130 U	<170 U	<240 U	<460 U
MDL (est)(2)	100	130	170	240	460
EQL (est)(3)	300	380	510	710	1400
Preparation DF	24.7	24.7	24.7	24.7	24.7
Analysis DF	62.5	62.5	125	125	125
Total DF	1500	1500	3000	3000	3000
MDL (at the instrument)	0.067	0.084	0.057	0.079	0.15

DF = dilution factor

U =. Analyte is either not observed or the response is below the include MDL value.

(1) In the AS-15 separation, glycolate and gluconate co-elute. Separate analysis found that the signal response for gluconate is approximately 25% - 38% of the signal response for glycolate. Thus, the results in this column could be interpreted as glycolate/gluconate. Quantitation was based on the calibration curve of glycolate.

(2) The estimated MDL is based on the MDL at the instrument (taken to be one-third of the LLS) multiplied by the total dilution factor.

(3) The estimated EQL is taken to be the LLS concentration multiplied by the total dilution factor.

The results reported for acetate and glycolate in Table 1 were determined by the AS-15 separation. Formate, oxalate, and citrate were determined by the AS-11 separation.

2. Quality Control Sample Comments

Duplicate (DUP) and Triplicate (TRIP). A duplicate sample was created for this sample set; none of the desired analytes exhibited a response at the dilutions used for analysis. Duplicate instrument injections were done for the sample and sample duplicate.

Post Spike (PS) and Post Spike Duplicate (PSD). These samples cannot be successfully spiked in the matrix so that the included volume of spike would be <10% of the sample. Several of the analytes would exceed their respective solubilities in water if this exercise was undertaken. Therefore, a spike was added to the IEX solution delivered for analyses. The only QC available is therefore a **Post Spike**. The spike recoveries as well as the RPD between the PS and PSD meet the QC success criteria.

Table 2. Organic Anion Results for the AZ-6 Solid PS and PSD samples (ASR 6284)

Post Spike Success Criteria: 75% to 125% of expected value						
Analyte/Sample	**MDL* mg/L**	**Spike mg/L**	**Sample mg/L**	**Measured mg/L**	**Rec %**	**RPD %**
Glycolate PS	4.2	120	0	140	119	
Glycolate PSD		120	0	140	118	1
	mg/L	mg/L	mg/L	mg/L	%	%
Acetate PS	5.3	43	0	45	103	
Acetate PSD		43	0	40	92	9
	mg/L	mg/L	mg/L	mg/L	%	%
Formate PS	7.1	35	0	40	114	
Formate PSD		35	0	41	117	3
	mg/L	mg/L	mg/L	mg/L	%	%
Oxalate PS	9.8	45	0	52	115	
Oxalate PSD		45	0	49	108	6
	mg/L	mg/L	mg/L	mg/L	%	%
Citrate PS	19	89	0	101	114	
Citrate PSD		89	0	90	101	11

*MDL's are derived from the instrument MDL multiplied by the dilution factor applied to the post spike

For the purposes of the post spike, the sample value is calculated as zero if the analytical result is below the MDL.

Preparation Blank (PB) and Laboratory Control Standard (LCS).

Two LCS samples were prepared. LCS-1 contained glycolate, acetate, formate, oxalate, citrate, and gluconate. LCS-2 contained glycolate, acetate, formate, oxalate, and citrate only. These samples were constructed to demonstrate the co-elution problems associated with gluconate; the LCS-1 sample contained 8.7 times more gluconate than glycolate. In the AS-15 analysis gluconate and glycolate co-elute. There is no discernable retention time difference between fluoride and gluconate in the AS-11 analysis.
The LCS yield data (for LCS-1 below) indicates the co-elution of glycolate and gluconate .

LCS data is reported as the concentration delivered to the hot cell. LCS data is assembled from the AS-15 and AS-11 data in the same fashion that PS, PSD, analytical samples, and other QC are obtained. The LCS data clearly demonstrates the overlap between gluconate and glycolate. With this exception, the PB and LCS data all meet the success criteria.

Table 3. Organic Anion Results for the PB and LCS

PB success criteria: < EQL **LCS success criteria: 80% to 120% Recovery**					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
PB	<100 U	< 130 U	< 170 U	< 240 U	< 460 U
LCS-1 measured	**29000** **(587%)**	5200 (118%)	4900 (106%)	3600 (104%)	10000 (101%)
LCS-1 expected	4900	4400	4600	3500	10000
LCS-2 measured	8700 (119%)	4800 (110%)	5900 (112%)	3700 (110%)	11000 (100%)
LCS-2 expected	7300	4400	5300	3400	11000

Explanation of flag: U: analyte is either not observed or the determination was below the included MDL level.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards.

ICB and CCB standards met the success criteria. All analytes were below the MDL levels shown below. Since the analysis was done using two column separations, two sets of ICB and CCB data were compiled for the table below.

Table 4. Organic Anion Results for the ICB and CCB Standards

ICB/CCB success criteria: < MDL (at the instrument)					
Sample ID	**Glycolate mg/L**	**Acetate mg/L**	**Formate mg/L**	**Oxalate mg/L**	**Citrate mg/L**
MDL	0.067	0.084	0.057	0.079	0.15
ICB	< 0.067 U	< 0.084 U	< 0.057 U	< 0.079 U	< 0.15 U
CCB	< 0.067 U	< 0.084 U	< 0.057 U	< 0.079 U	< 0.15 U

Explanation of flag: U: analyte is either not observed or the determination was below the included MDL level.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards.

The ICV/CCV analysis met the success criteria. Since the analysis was done using two column separations, two sets of ICV and CCV data were examined.

Table 5. Organic Anion Results for the ICV and CCV Standards

ICV/CCV success criteria: 90% to 110% Recovery (at the instrument)					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
ICV-AS11 measured	n.a.	n.a.	0.51 (105%)	0.54 (91%)	1.20 (96%)
ICV-AS11 expected			0.48	0.59	1.20
CCV-AS11 measured	n.a.	n.a.	0.44 (101%)	0.53 (94%)	1.20 (104%)
CCV-AS11 expected			0.44	0.56	1.10
ICV-AS15 measured	0.72 (99%)	0.62 (108%)	0.49 (100%)	0.64 (107%)	n.a.
ICV-AS15 expected	0.72	0.57	0.48	0.59	
CCV-AS15 measured	0.67 (103%)	0.54 (101%)	0.46 (104%)	0.52 (91%)	n.a.
CCV-AS15 expected	0.65	0.54	0.44	0.56	
n.a. = not applicable					

Low-Level Standard (LLS).

The LLS met the success criteria. Since the analysis was done using two separations, two sets of data were examined. The LLS level is comparable to the EQL level. By examination of the integrated area of the LLS analytes, the MDL level is approximated by 1/3 of the LLS or EQL level. This criteria is applied to all data released by this laboratory.

Table 6. Organic Anion Results for the LLS

LLS success criteria: 75% to 125% Recovery (at the instrument)					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
LLS-AS11 measured	n.a.	n.a.	0.17 (97%)	0.24 (104%)	0.46 (104%)
LLS-AS11 expected			0.17	0.23	0.44
LLS-AS15 measured	0.20 (77%)	0.25 (117%)	0.18 (100%)	0.24 (106%)	n.a.
LLS-AS15 expected	0.26	0.22	0.17	0.23	
n.a. = not applicable					

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 115
Client:	John Geeting
Total Samples:	10

Analysis: Ta, Pt, amu-241, Np-237, Pu-239, Pu-240 and Pu-242

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 27FEB02
Procedure – 27FEB02
Element Menu – 27FEB02

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 18Aug02
Reviewed By Date

18 Aug 02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
02-832-B1-Ni	PROCESS BLK-1
02-832-B2-Ni	PROCESS BLK-2
02-829-Ni	AZ-0
02-830-Ni	AZ-2
02-831-Ni	AZ-4
02-832-Ni	AZ-6
02-832-DUP-Ni	AZ-6-Dup
02-832-BS-Ni	ICP/MS Blank Spike
02-832-MS-Ni	ICP/MS Matrix Spike
02-832-LCS-Ni	LCS SRM2710

The samples (AZ-101 slurry) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Ta, Pt, amu-241, Np-237, Pu-239, Pu-240 and Pu-242.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Results

Ta Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	3.73E-04	2.40E-05	3.60E-05
02-00832-B2-Ni	1.95E-03	1.05E-04	3.52E-05
02-00832-Ni	6.50E+00	1.11E-01	1.45E-02
02-00832-DUP-Ni	6.70E+00	9.92E-02	1.08E-02
02-00829-Ni	4.39E-01	1.14E-02	1.35E-02
02-00830-Ni	9.58E-01	8.00E-03	1.11E-02
02-00831-Ni	7.28E-01	2.72E-02	1.05E-02
02-00832-BS-Ni	1.78E-02	2.53E-04	2.28E-05
02-00832-MS-Ni	8.71E+00	1.38E-01	1.24E-02
02-00832-LCS-Ni	9.49E-01	8.66E-02	1.15E-02

Pt Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<1.88E-04		1.88E-04
02-00832-B2-Ni	<1.83E-04		1.83E-04
02-00832-Ni	1.59E-01	2.10E-02	7.54E-02
02-00832-DUP-Ni	1.38E-01	3.24E-02	5.65E-02
02-00829-Ni	<7.03E-02		7.03E-02
02-00830-Ni	<5.78E-02		5.78E-02
02-00831-Ni	5.82E-02	3.09E-02	5.50E-02
02-00832-BS-Ni	<1.19E-04		1.19E-04
02-00832-MS-Ni	1.92E-01	5.90E-02	6.48E-02
02-00832-LCS-Ni	<5.98E-02		5.98E-02

AMU-241 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<6.37E-06		6.37E-06
02-00832-B2-Ni	<6.22E-06		6.22E-06
02-00832-Ni	5.95E+01	9.10E-01	2.56E-03
02-00832-DUP-Ni	6.12E+01	1.33E+00	1.92E-03
02-00829-Ni	1.23E+01	3.10E-01	2.38E-03
02-00830-Ni	2.11E+01	2.39E+00	1.96E-03
02-00831-Ni	1.86E+01	7.58E-01	1.86E-03
02-00832-BS-Ni	1.96E-05	1.01E-05	4.03E-06
02-00832-MS-Ni	4.42E+01	9.23E-01	2.20E-03
02-00832-LCS-Ni	<2.03E-03		2.03E-03

Np-237 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<9.83E-06		9.83E-06
02-00832-B2-Ni	<9.60E-06		9.60E-06
02-00832-Ni	1.91E+02	2.81E+00	3.95E-03
02-00832-DUP-Ni	1.92E+02	4.98E+00	2.96E-03
02-00829-Ni	3.94E+01	4.86E-01	3.68E-03
02-00830-Ni	7.08E+01	7.25E+00	3.02E-03
02-00831-Ni	6.21E+01	1.97E+00	2.88E-03
02-00832-BS-Ni	2.42E-05	5.30E-06	6.23E-06
02-00832-MS-Ni	1.48E+02	3.62E+00	3.39E-03
02-00832-LCS-Ni	<3.13E-03		3.13E-03

Pu-239 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	8.89E-05	1.58E-05	6.87E-06
02-00832-B2-Ni	8.75E-05	2.67E-05	6.71E-06
02-00832-Ni	1.27E+02	1.02E+00	2.76E-03
02-00832-DUP-Ni	1.30E+02	4.19E+00	2.07E-03
02-00829-Ni	2.63E+01	4.53E-01	2.57E-03
02-00830-Ni	4.92E+01	1.75E+00	2.11E-03
02-00831-Ni	4.13E+01	1.46E+00	2.01E-03
02-00832-BS-Ni	6.19E-05	3.12E-05	4.35E-06
02-00832-MS-Ni	1.00E+02	3.82E+00	2.37E-03
02-00832-LCS-Ni	1.30E-01	6.46E-03	2.19E-03

Pu-240 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<6.08E-06		6.08E-06
02-00832-B2-Ni	<5.94E-06		5.94E-06
02-00832-Ni	9.77E+00	1.20E-01	2.44E-03
02-00832-DUP-Ni	9.96E+00	1.88E-01	1.83E-03
02-00829-Ni	2.01E+00	4.31E-02	2.28E-03
02-00830-Ni	3.52E+00	3.57E-01	1.87E-03
02-00831-Ni	3.10E+00	1.50E-01	1.78E-03
02-00832-BS-Ni	<3.85E-06		3.85E-06
02-00832-MS-Ni	7.37E+00	2.84E-01	2.10E-03
02-00832-LCS-Ni	1.12E-02	9.58E-03	1.94E-03

Pu-242 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-832-B1-Ni	<1.06E-05		1.06E-05
02-832-B2-Ni	<1.03E-05		1.03E-05
02-829-Ni	1.14E-01	9.42E-03	4.25E-03
02-830-Ni	1.10E-01	1.68E-02	3.18E-03
02-831-Ni	2.63E-02	8.07E-04	3.96E-03
02-832-Ni	6.59E-02	2.55E-02	3.26E-03
02-832-DUP-Ni	4.14E-02	6.10E-03	3.10E-03
02-832-BS-Ni	<6.71E-06		6.71E-06
02-832-LCS-Ni	1.14E-01	5.66E-03	3.65E-03
02-832-MS-Ni	<3.37E-03		3.37E-03

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-832-Ni) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-832-Ni) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-832-Ni) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-832-Ni) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-832-Ni) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-832-B1-Ni) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

All analytes in the LCS/BS failed to meet the success criteria of ±20%. However a PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements met the success criteria of ± 20% RPD except for Pt, which was <10XMDL. However, the ICS and ICSD instrument QC samples met the success criteria of ± 20% RPD for all analytes analyzed.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis. All analytes analyzed in the MS failed to meet the success criteria of ±25%. However an ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM-2710)

Narrative:

There are no certified values for these elements in the LCS/SRM(2710).

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 128
Client:	John Geeting
Total Samples:	10

Analysis: Rb, Cs, and Tc-99

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 14FEB02
Procedure – 14FEB02
Element Menu – 14FEB02

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 18Aug02
Reviewed By Date

16 aug 02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
02-832-B1-Ni	PROCESS BLK-1
02-832-B2-Ni	PROCESS BLK-2
02-829-Ni	AZ-0
02-830-Ni	AZ-2
02-831-Ni	AZ-4
02-832-Ni	AZ-6
02-832-DUP-Ni	AZ-6-Dup
02-832-BS-Ni	ICP/MS Blank Spike
02-832-LCS-Ni	LCS SRM2710
02-832-MS-Ni	ICP/MS Matrix Spike

The samples (AZ-101 slurry) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Rb, Cs, andTc-99.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Results

Rb Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-832-B1-Ni	4.14E-01	4.08E-03	1.80E-04
02-832-B2-Ni	5.02E-01	8.31E-03	1.83E-04
02-829-Ni	2.51E+02	1.41E+00	9.49E-02
02-830-Ni	2.46E+02	4.97E+00	7.84E-02
02-831-Ni	2.04E+02	3.18E-01	7.07E-02
02-832-Ni	2.41E+02	1.74E+00	7.36E-02
02-832-DUP-Ni	2.60E+02	1.77E+00	8.06E-02
02-832-BS-Ni	5.38E-01	7.63E-03	1.53E-04
02-832-LCS-Ni	3.58E+02	6.70E+00	1.75E-01
02-832-MS-Ni	2.69E+02	8.54E-01	9.13E-02

Cs Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-832-B1-Ni	<5.34E-05		5.34E-05
02-832-B2-Ni	<5.42E-05		5.42E-05
02-829-Ni	3.82E+01	7.02E-02	2.82E-02
02-830-Ni	1.75E+01	2.13E-01	2.33E-02
02-831-Ni	1.20E+01	1.48E-01	2.10E-02
02-832-Ni	1.23E+01	1.56E-01	2.18E-02
02-832-DUP-Ni	1.22E+01	2.46E-01	2.39E-02
02-832-BS-Ni	8.97E-03	1.17E-04	4.54E-05
02-832-LCS-Ni	1.04E+02	4.65E-01	5.20E-02
02-832-MS-Ni	1.61E+01	2.68E-01	2.71E-02

Tc-99 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-832-B1-Ni	9.23E-04	4.25E-05	1.30E-04
02-832-B2-Ni	9.28E-04	6.74E-05	1.32E-04
02-829-Ni	3.00E+01	2.47E-01	6.87E-02
02-830-Ni	9.00E+00	6.12E-02	5.67E-02
02-831-Ni	8.77E+00	7.79E-02	5.12E-02
02-832-Ni	2.26E+00	8.74E-02	5.33E-02
02-832-DUP-Ni	2.79E+00	3.93E-02	5.84E-02
02-832-BS-Ni	4.49E-04	2.39E-05	1.11E-04
02-832-LCS-Ni	5.66E-01	7.31E-02	1.27E-01
02-832-MS-Ni	2.29E+00	1.02E-01	6.60E-02

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-829-Ni) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-829-Ni) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-829-Ni) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-829-Ni) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-829-Ni) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-832-B1Ni) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

All analytes in the LCS/BS met the success criteria of ±20% except Tc-99, which was not spiked in this QC. However a PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements met the success criteria of ± 20% RPD except Tc-99. However, the ICS and ICSD instrument QC samples met the success criteria of ± 20% RPD for all analytes analyzed.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis; all analytes analyzed in the MS met the success criteria of ±25% except Tc-99, which was not spiked in the MS solution. However an ICSDS (matrix post spike) was analyzed and Tc-99 and met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM-2710)

Narrative:

The LCS/SRM (2710) was analyzed for Cs only and met the success criteria of ±25%.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 115
Client:	John Geeting
Total Samples:	10

Analysis: U and U-iso

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 23 April 2002

Analysis Files:
Experiment – 02 April 02
Procedure – 02 April 02
Element Menu – 02 April 02

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 18Aug02
Reviewed By Date

18-Aug 02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
02-00832-B1-Ni	PROCESS BLK-1
02-00832-B2-Ni	PROCESS BLK-2
02-00832-Ni	AZ-6
02-00832-Ni-Dup	AZ-6-Dup
02-00829-Ni	AZ-0
02-00830-Ni	AZ-2
02-00831-Ni	AZ-4
02-00832-MS-Ni	ICP/MS Matrix Spike
02-00832-BS-Ni	ICP/MS Blank Spike
02-00832-LCS-Ni	LCS/SRM2710

The samples (AZ-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) total U and U-isotopic.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using the 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Results

Total U Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	8.10E-04	4.45E-05	6.72E-05
02-00832-B2-Ni	8.99E-04	7.79E-05	6.91E-05
02-00832-Ni	1.26E+04	1.19E+02	6.32E-01
02-00832-Ni-Dup	1.16E+04	1.08E+02	6.88E-01
02-00829-Ni	3.36E+03	8.27E+01	1.74E-01
02-00830-Ni	1.33E+04	2.57E+02	3.14E-01
02-00831-Ni	1.10E+04	1.59E+02	2.71E-01
02-00832-MS-Ni	1.21E+04	1.67E+02	7.06E-01
02-00832-BS-Ni	1.57E-02	5.97E-04	5.51E-05
02-00832-LCS-Ni	3.17E+01	6.01E-01	2.67E-02

U-233 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	5.09E-06		5.09E-06
02-00832-B2-Ni	5.24E-06		5.24E-06
02-00832-Ni	4.65E-01	5.33E-02	4.79E-02
02-00832-Ni-Dup	4.74E-01	8.20E-02	5.22E-02
02-00829-Ni	1.26E-01	2.47E-02	1.32E-02
02-00830-Ni	5.16E-01	2.92E-02	2.38E-02
02-00831-Ni	4.26E-01	4.74E-02	2.06E-02
02-00832-MS-Ni	4.71E-01	4.57E-02	5.35E-02
02-00832-BS-Ni	4.18E-06		4.18E-06
02-00832-LCS-Ni	2.02E-03		2.02E-03

U-234 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	4.55E-06		4.55E-06
02-00832-B2-Ni	4.68E-06		4.68E-06
02-00832-Ni	8.91E-01	3.08E-02	4.28E-02
02-00832-Ni-Dup	8.31E-01	1.03E-01	4.66E-02
02-00829-Ni	2.46E-01	1.12E-02	1.18E-02
02-00830-Ni	9.52E-01	7.76E-02	2.13E-02
02-00831-Ni	8.13E-01	4.77E-02	1.84E-02
02-00832-MS-Ni	9.00E-01	3.67E-02	4.78E-02
02-00832-BS-Ni	3.73E-06		3.73E-06
02-00832-LCS-Ni	3.91E-03	1.65E-03	1.81E-03

U-235 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<8.03E-06		8.03E-06
02-00832-B2-Ni	8.70E-06	4.93E-06	8.26E-06
02-00832-Ni	1.10E+02	2.09E+00	7.55E-02
02-00832-Ni-Dup	9.82E+01	1.29E+00	8.22E-02
02-00829-Ni	2.90E+01	4.20E-01	2.08E-02
02-00830-Ni	1.13E+02	1.08E+00	3.75E-02
02-00831-Ni	9.48E+01	2.08E+00	3.24E-02
02-00832-MS-Ni	1.03E+02	1.21E+00	8.44E-02
02-00832-BS-Ni	4.88E-05	7.71E-06	6.58E-06
02-00832-LCS-Ni	2.43E-01	1.30E-02	3.19E-03

U-236 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	<5.13E-06		5.13E-06
02-00832-B2-Ni	<5.27E-06		5.27E-06
02-00832-Ni	8.07E+00	2.14E-01	4.82E-02
02-00832-Ni-Dup	7.34E+00	1.72E-01	5.25E-02
02-00829-Ni	2.09E+00	2.86E-02	1.33E-02
02-00830-Ni	8.23E+00	1.15E-01	2.40E-02
02-00831-Ni	6.91E+00	3.28E-02	2.07E-02
02-00832-MS-Ni	7.78E+00	1.25E-01	5.39E-02
02-00832-BS-Ni	<4.20E-06		4.20E-06
02-00832-LCS-Ni	3.89E-03	1.70E-03	2.04E-03

U-238 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Ni	8.08E-04	1.80E-05	6.15E-05
02-00832-B2-Ni	8.88E-04	9.80E-06	6.33E-05
02-00832-Ni	1.24E+04	2.02E+00	5.79E-01
02-00832-Ni-Dup	1.15E+04	1.28E+00	6.30E-01
02-00829-Ni	3.33E+03	4.28E-01	1.59E-01
02-00830-Ni	1.32E+04	1.15E+00	2.88E-01
02-00831-Ni	1.09E+04	2.09E+00	2.48E-01
02-00832-MS-Ni	1.20E+04	1.09E+00	6.47E-01
02-00832-BS-Ni	1.56E-02	1.77E-05	5.04E-05
02-00832-LCS-Ni	3.14E+01	1.27E-02	2.44E-02

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards for total U met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-00832-Ni) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-00832-Ni) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-00832-Ni) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-00832-Ni) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-00832-Ni) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-00832-B1-Ni) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

No LCS/BS was submitted for analysis, however a PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All analytes met the success of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MS or MSD was submitted for analysis.

Laboratory Control Standard (LCS/SRM)

Narrative:

The LCS/SRM-2710 was submitted for analysis and failed the success criteria of ± 25%, the recovery of the LCS/SRM was 127%.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 116
Client:	John Geeting
Total Samples:	9

Method developed Specifically for Analytes tested

Analysis: Pd, Ru, Rh, Pr and Pt

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 23 April 2002

Analysis Files:
Experiment – 01 April 02
Procedure – 01 April 02
Element Menu – 01 April 02

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): James Bramson / Teresa Wilson

Orvill Thomas Farmer III 24Apr02
Reviewed By Date

4.24.02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
02-00832-B1-Zr (116)	PROCESS-BLK-1
02-00832-B2-Zr (116)	PROCESS-BLK-2
02-00832-Zr (116)	AZ-6
02-00832-Dup-Zr (116)	AZ-6-Dup
02-00829-Zr (116)	AZ-0
02-00830-Zr (116)	AZ-2
02-00831-Zr (116)	AZ-4
02-00832-BS-Zr (116)	ICP/MS Blank Spike
02-00830-MS-Zr (116)	ICP/MS Matrix Spike

The samples (AZ-101 supernate) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Ru, Pd, Rh, Pr and Pt.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

Both Ru and Pd were determined to be fission yield production; selected isotopes for each analyte were used after correcting for both atomic interferences and altered isotopic abundances.

2. Results

Ru Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (116)	1.67E-03	2.77E-04	5.21E-04
02-00832-B2-Zr (116)	<5.53E-04		5.53E-04
02-00832-Zr (116)	1.49E+03	9.89E+00	2.90E-01
02-00832-Dup-Zr (116)	1.59E+03	2.13E+01	3.02E-01
02-00829-Zr (116)	4.16E+02	2.50E+00	3.10E-01
02-00830-Zr (116)	8.13E+02	1.39E+01	3.35E-01
02-00831-Zr (116)	9.00E+02	1.05E+01	2.67E-01
02-00832-BS-Zr (116)	2.38E-02	1.07E-03	4.13E-04
02-00830-MS-Zr (116)	7.49E+02	9.29E+01	2.76E-01

Pt Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (116)	<6.85E-04		6.85E-04
02-00832-B2-Zr (116)	<6.89E-04		6.89E-04
02-00832-Zr (116)	<3.56E-01		3.56E-01
02-00832-Dup-Zr (116)	<3.65E-01		3.65E-01
02-00829-Zr (116)	<3.65E-01		3.65E-01
02-00830-Zr (116)	<4.03E-01		4.03E-01
02-00831-Zr (116)	<3.25E-01		3.25E-01
02-00832-BS-Zr (116)	9.61E-03	7.15E-04	5.83E-04
02-00830-MS-Zr (116)	4.49E+00	8.65E-01	3.60E-01

Pd Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (116)	4.64E-02	2.45E-03	1.89E-03
02-00832-B2-Zr (116)	4.85E-02	2.11E-03	2.00E-03
02-00832-Zr (116)	1.37E+03	8.07E+01	1.05E+00
02-00832-Dup-Zr (116)	1.07E+03	4.05E+01	1.10E+00
02-00829-Zr (116)	1.71E+03	6.86E+01	1.12E+00
02-00830-Zr (116)	1.99E+02	2.81E+01	1.22E+00
02-00831-Zr (116)	2.05E+02	3.01E+01	9.68E-01
02-00832-BS-Zr (116)	1.09E+00	3.11E-02	1.50E-03
02-00830-MS-Zr (116)	3.79E+02	5.42E+01	1.00E+00

Pr Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (116)	9.91E-04	7.30E-04	2.85E-04
02-00832-B2-Zr (116)	<2.86E-04		2.86E-04
02-00832-Zr (116)	8.54E+02	1.46E+01	1.48E-01
02-00832-Dup-Zr (116)	9.11E+02	9.23E+00	1.52E-01
02-00829-Zr (116)	2.36E+02	2.42E+00	1.52E-01
02-00830-Zr (116)	4.50E+02	1.14E+01	1.67E-01
02-00831-Zr (116)	5.07E+02	1.93E+00	1.35E-01
02-00832-BS-Zr (116)	2.88E-04	1.62E-04	2.42E-04
02-00830-MS-Zr (116)	4.00E+02	2.63E+01	1.49E-01

Rh Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (116)	<3.93E-04		3.93E-04
02-00832-B2-Zr (116)	<4.16E-04		4.16E-04
02-00832-Zr (116)	3.11E+02	7.13E+00	2.18E-01
02-00832-Dup-Zr (116)	3.20E+02	1.20E+01	2.28E-01
02-00829-Zr (116)	9.09E+01	3.32E+00	2.33E-01
02-00830-Zr (116)	1.82E+02	3.16E+00	2.53E-01
02-00831-Zr (116)	1.95E+02	5.20E+00	2.01E-01
02-00832-BS-Zr (116)	3.26E-02	1.55E-03	3.11E-04
02-00830-MS-Zr (116)	1.76E+02	2.16E+01	2.08E-01

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 2% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes when the DT is greater then 10 X MDL.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-00832-Zr-116) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-00832-Zr-116) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-00832-Zr-116) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-00832-Zr-116) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-00832-Zr-116) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-00832-B1-Zr-116) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes except Pd which suffered from Zr contamination.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

All analytes failed the LCS/BS success criteria of ±20%. A PBS was analyzed and all analytes of interest met the success criteria of ±20% except Rh (73% recovery).

Duplicate (DUP)

Narrative:

All elements met the success of ± 20% RPD except Pd which suffered from Zr interference and contamination. The ICS and ICSD also met the success criteria of ± 20% RPD for all analytes when the response was above 10 X MDL.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis All analytes failed the success criteria of ± 25% recovery. The ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 115
Client:	John Geeting
Total Samples:	10

Analysis: Sn-126

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 26 April 02
Procedure – 26 April 02
Element Menu – 26 April 02

Analyst(s): James Bramson / Teresa Wilson

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Orville Thomas Farmer III 18Aug02
Reviewed By Date
19/Aug 02

Samples Submitted for Analysis:

RPL #	Client I.D.
02-00823-B1-Ni	PROCESS BLK-1
02-00823-B2-Ni	PROCESS BLK-2
02-00823-BS-Ni	ICP/MS Blank Spike
02-00823-LCS-Ni	LCS/SRM2710
02-00832-Ni	AZ-6
02-00832-DUP-Ni	AZ-6-Dup
02-00829-Ni	AZ-0
02-00830-Ni	AZ-2
02-00831-Ni	AZ-4
02-00832-MS-Ni	ICP/MS Matrix Spike

The samples (AZ-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Sn-126.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis. The Sn-126 results were calculated using the Sn-120 (32.8% abundance) isotope calibration response curve. The MS, BS and LCS/SRM were not spiked with Sn-126 at the time of sample preparation, also the LCS/SRM does don't have Sn-126 as an impurity analyte. An instrument post spike of the MS and BS using the Sn-120 isotope in the natural calibration solutions was performed to evaluate analyte recovery.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using the seven (7) instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Results

Sn-126 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00823-B1-Ni	<2.12E-06		2.12E-06
02-00823-B2-Ni	<2.24E-06		2.24E-06
02-00823-BS-Ni	9.48E-06	2.71E-06	2.12E-06
02-00823-LCS-Ni	4.00E-03	9.70E-04	9.19E-04
02-00832-Ni	2.11E-01	4.64E-03	8.69E-04
02-00832-DUP-Ni	2.08E-01	1.65E-03	9.64E-04
02-00829-Ni	7.80E-02	4.71E-03	9.25E-04
02-00830-Ni	1.44E-01	4.28E-03	8.15E-04
02-00831-Ni	9.46E-02	2.32E-03	7.66E-04
02-00832-MS-Ni	2.04E-01	1.02E-02	9.84E-04

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-00832-Ni) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-00832-Ni) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-00832-Ni) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-00832-Ni) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-00832-Ni) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-00832-B1-Ni) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

The LCS/BS that was submitted for analysis was not spike at the time of sample dissolution in the hot cell, however a PBS was analyzed and Sn-120 met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements met the success criteria of ± 20% RPD. Also, the ICS and ICSD instrument QC samples met the success criteria of ± 20% RPD for all analytes analyzed.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

The MS that was submitted for analysis was not spiked at the time of sample dissolution in the hot cell, however an ICSDS (matrix post spike) was analyzed and Sn-120 met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM-2710)

Narrative:

The LCS/SRM was submitted for analysis Sn-126 is not a certified analyte in this standard.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W58950
ASR#:	6284 / 128
Client:	John Geeting
Total Samples:	10

Analysis: Tc-99

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 05feb02
Procedure – 020205a, 020201a
Element Menu – rbtc, 1feb02

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 15 Apr 02
Reviewed By Date

T Wilson 4.15.02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
02-00822-B	Reagents Only
02-00822	AZ-A
02-00822-DUP	AZ-A DUP
02-00823	AZ-C
02-00824	AZ-E
02-00825	AZ-G
02-00826	AZ-I
02-00827	AZ-K
02-00828	AZ-M

The samples (AZ-101 supernate) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Tc-99.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using all the instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.75. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

2. Results

µCi/g -correct

Tc-99 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
02-00822-B	5.57E-06	9.57E-08	9.67E-07
02-00822	3.70E-01	1.03E-02	1.98E-04
02-00822-DUP	3.19E-01	2.08E-03	2.00E-04
02-00823	1.57E-01	7.22E-04	2.03E-04
02-00824	7.09E-02	7.57E-04	2.44E-04
02-00825	3.81E-02	9.91E-04	1.96E-05
02-00826	1.85E-02	2.00E-05	2.22E-05
02-00827	8.98E-03	6.95E-05	2.20E-05
02-00828	4.81E-03	4.65E-05	2.22E-05

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 2% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-777) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-777) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-777) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-777) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-777) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-00822-B) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

No LCS/BS was submitted for analysis, however a PBS was analyzed and Tc-99 met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements met the success criteria of ± 20% RPD. Also, the ICS and ICSD instrument QC samples met the success criteria of ± 20% RPD for all analytes analyzed.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis, however an ICSDS (matrix post spike) was analyzed and Tc-99 and met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM-2710)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60513
ASR#:	6284 / 114
Client:	John Geeting
Total Samples:	9

Analysis: I-129

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: WB36913 ICP/MS, VG Elemental
512-06-01-014 Mettler AJ100 Balance

Point of Contact: **Orville Thomas Farmer III**

Report Date: 28 April 02

Analysis Files: Experiment - 03 April 02
Procedure – 03 April 02
Element Menu – 03 April 02

Analyst(s): James Bramson / Teresa Wilson

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Orville Thomas Farmer III 18Aug02
Reviewed By Date

19 Aug 02
CONCUR

Samples Submitted for Analysis:

RPL #	Client I.D.
02-00832-B1-Zr (114)	PROCESS BLK-1
02-00832-B2-Zr (114)	PROCESS BLK-2
02-00832-Zr (114)	AZ-6
02-00832-Dup-Zr (114)	AZ-6-Dup
02-00829-Zr (114)	AZ-0
02-00830-Zr (114)	AZ-2
02-00831-Zr (114)	AZ-4
02-00832-BS-Zr (114)	ICP/MS Blank Spike
02-00832-MS-Zr (114)	ICP/MS Matrix Spike

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) I-129.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

All samples solutions were modified with (1% HCl / 5 mM P-Cyanophenol) to reduce memory effects and stabilize the Iodine signal. The major interference for the determination of I-129 by ICP/MS is the atomic ion of Xe-129, which was subtracted using the Xe-131 atomic ion. This correction was applied to all sample solutions in the analytical run.

2. Results

I-129 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
02-00832-B1-Zr (114)	<1.73E-04		1.73E-04
02-00832-B2-Zr (114)	1.67E-04	1.38E-04	1.67E-04
02-00832-Zr (114)	<7.81E-02		7.81E-02
02-00832-Dup-Zr (114)	<6.68E-02		6.68E-02
02-00829-Zr (114)	8.38E-02	2.18E-02	5.28E-02
02-00830-Zr (114)	1.06E-01	3.82E-02	6.20E-02
02-00831-Zr (114)	<5.91E-02		5.91E-02
02-00832-BS-Zr (114)	<1.10E-04		1.10E-04
02-00832-MS-Zr (114)	5.66E-02	9.34E-02	4.74E-02

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 1% HCl / 5 mM P-Cyanophenol solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes when Greater then 10 X MDL.

Instrument Control Solution (ICS)

Narrative:

Sample solution (02-00832-Zr-114) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-00832-Ni) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (02-00832-Zr-114) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-00832-Zr-114) met the instrument QC success criteria of ± 20% RPD for all analytes when greater then 10 X MDL.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (02-00832-Zr-114) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (02-00832-B1-Zr-114) and failed the QC success criteria of ± 20% recovery for all analytes, the PBS recovery was 75%.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

The LCS/BS submitted for analysis was spiked with I-127 and failed the success criteria of ±20% , however a PBS using I-129 was analyzed and met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

The sample duplicate failed the success criteria of ± 20% RPD , and the ICS and ICSD failed the success criteria of ± 20% RPD because the analyte concentration was below 10 X MDL.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

The MS submitted for analysis was spiked with I-127 and met the success criteria of ±25%, also a PBS using I-129 was analyzed and met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle
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Project No. 42365

Internal Distribution
File/LB

Date June 3, 2002

To J. Geeting

From L. R. Greenwood

Subject Radiochemical Analyses for ASR 6284

Samples of the supernate, washes, rinses, leaches and slurries for tank AZ101 (02-0822-832) were analyzed for gamma emitters, ^{90}Sr, total alpha, Am/Cm, Pu, and Tc according to ASR 6284. The analyses were performed on sample materials prepared by acid digestion (liquids) or fusions (dried slurries) in the hot cells. The attached reports list measured analyte activities in units of μCi/ml (liquids, corrected for density) or uCi/g of dried solids. 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.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. Only ^{134}Cs and ^{137}Cs could be detected in the liquids; however, most of the requested isotopes were detected in the solids. In a number of cases, it was not possible to meet the requested MRQ values in extended counting due to the very high levels of ^{137}Cs activity. Minimum detectable activity (MDA) values are reported for all of the requested isotopes including the additional request list for the slurry sample AZ-6. Hot cell process blanks showed negligible activity relative to the samples. The ^{241}Am results for the solids are in reasonable agreement with the alpha energy analysis results reported below, although the AEA results have lower uncertainties in most cases.

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). ^{137}C was detected in a few of the samples and suitable small corrections were made to the beta counting results. The process blank prepared with the acid digestion in the laboratory was found to have a low level of ^{90}Sr contamination that corresponds to 20% of the activity in sample AZ-E, 16% for AZ-M, 14% for sample AZ-K, 12% for AZ-C, and less than 10% for the other samples. The solids hot cell process blank was negligible with respect to the samples. The reagent blank did not show any contamination. The LCS and matrix spike showed good recovery at 103% and 93%, respectively. Duplicate analyses showed acceptable agreement taking into account the uncertainties in the data. Some of the uncertainties are relatively high for some of the liquid samples due to the large correction to the beta counting from the ^{85}Sr tracer. The detection limits as well as many of the measurements were well below the requested MRQ values, except in cases where the measured ^{90}Sr activities greatly exceeded the MRQ values.

Total Alpha

The total alpha activity was determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. Alpha activity could not be detected in the liquid samples; the detection limits are quite low, although there was no MRQ value requested for the liquids. A better estimate of the total alpha activity is given by the sum of the alpha emitters, as discussed below. Relatively high levels of total alpha activity were detected in all of the slurry samples, well above the requested MRQ value. Duplicate results are in good agreement for the slurry sample AZ-6. No significant alpha activity was seen in the hot cell or laboratory blanks. Blank and matrix spikes gave good recoveries at 105% and 110%, respectively.

Plutonium, Americium and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and counted by alpha spectrometry according to PNL-ALO-422. The plutonium activities were determined with a ^{242}Pu tracer. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. For the liquid samples, only Am/Cm analyses were requested. The hot cell process blank was found to contain significant contamination with both Am and Cm isotopes, generally exceeding the alpha activities in the samples except for sample AZ-M. This hot cell contamination probably explains the poor agreement for sample AZ-A, which is below the hot cell level, compared to the good agreement for sample AZ-M, which is somewhat above the hot cell level of contamination. In any case, all of the results for the liquids (including the hot cell blank) are below the requested MRQ value of 7.2E-4 uCi/ml. Sample AZ-M is only slightly below the requested MRQ value; however, this sample is the least affected by the hot cell contamination. Negligible contamination was seen in the lab blank. The LCS recovery was 95% and the matrix spike recovery was 89%.

Both Am/Cm and Pu analyses were requested for the solid samples. In this case, the alpha activities in the hot cell process blanks as well as the lab blank were negligible with respect to the sample activities. Duplicate results for samples AZ-0 and AZ-6 were in reasonable agreement, taking into account the measurement uncertainties. The Pu, Am, and Cm activities in the samples were well above the requested MRQ values. The LCS recoveries were 103% and 102% for ^{242}Pu and ^{243}Am, respectively. Matrix spike recoveries were about 90% in both cases. The sum of the individual alpha activities is in good agreement with the total alpha activities. The alpha emitter sum is the best estimate of the total alpha activity due to the lower uncertainties for this method.

Tritium

Tritium was distilled from direct slurry samples according to procedure PNL-ALO-418 and measured by liquid scintillation counting according to procedure PNL-ALO-474. The procedure was modified to include a cation exchange and a second distillation to ensure the removal of relatively high levels of ^{90}Sr and ^{137}Cs in these samples. This procedural modification is documented in the raw data file. This resulted in very clean tritium beta spectra with no detectable beta contamination. Tritium was detected in all of the slurry samples above the requested MRQ value of 1.5E-2 uCi/g. Duplicate analyses were in excellent agreement. The laboratory LCS recovery was 87% and the hot cell LCS recovery was somewhat low at 72%. The matrix hot cell spike recovery was 101%. No tritium was detected in either the hot cell or laboratory blanks.

Tc-99 as Pertechnetate

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated fractions were then counted according to procedure RPG-CMC-408. The LCS blank spike recovery of a ^{99}Tc standard was 83%, and a matrix spike of sample AZ-M gave a standard recovery of 95%. Sample duplicates showed good agreement with an RPD of 3%. The ^{99}Tc activities in the samples were well above the requested MRQ value of 1.5E-3 uCi/ml.

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

06/03/02

Client : Geeting
ASR: 6284

Cognizant Scientist: L.R. Greenwood Date : 6/3/02

Concur : T Trang-le Date : 6/3/02

Procedure: PNL-ALO-432 for Tc-99 (Pertechnetate)

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

RPL ID Client ID		Pertechnetate Tc-99 Error +/-
02-822 AZ-A		3.86E-1 3%
	MDA	<7.E-4
02-823 AZ-C		1.62E-1 3%
	MDA	<7.E-4
02-824 AZ-E		7.77E-2 3%
	MDA	<7.E-4
02-825 AZ-G		3.90E-2 3%
	MDA	<7.E-4
02-826 AZ-I		1.80E-2 3%
	MDA	<7.E-4
02-827 AZ-K		8.84E-3 5%
	MDA	<7.E-4
02-828 AZ-M		5.65E-3 6%
	MDA	<7.E-4
02-828 DUP AZ-M		5.50E-3 6%
	MDA	<7.E-4
RPD		3%
Matrix Spike 02-828 AZ-M		95%
Blank Spike		83%
Lab Blank		<7.E-4

Battelle Pacific Northwest National Laboratory
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3/11/2002

Client : Geeting
ASR: 6284

Cognizant Scientist: L. R. Greenwood Date : 3/12/02

Concur : T Trang-le Date : 3/11/02

Procedure: PNL-ALO-418/424 for Tritium; Reference Date: 2/19/02

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

ALO ID Client ID	H-3	Error%	MDA
02-832B Process Blank			4E-4
02-829 Ni AZ-0	3.27E-2	4%	6E-4
02-830 Ni AZ-2	2.62E-2	4%	9E-4
02-831 Ni AZ-4	6.62E-2	4%	7E-4
02-832 Ni AZ-6	5.96E-2	4%	6E-4
02-832 Ni DUP AZ-6	5.89E-2	4%	6E-4
RPD	1%		
Lab Blank			4E-5
Lab reagent spike	87%		
MS 02-832 AZ-6	101%		
Cell blank spike	72%		

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02-822
03/26/02

Client : Geeting
ASR: 6284

Cognizant Scientist: L. R. Greenwood Date : 3/26/02

Concur : T Trang-le Date : 3/26/02

Reference Date: Feb. 4, 2002 for Gea
Procedure: PNL-ALO-450 for GEA
Procedure: PNL-ALO-476 For Sr-90

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

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Density g/ml
02-822BLK Process Blank	5.93E-2 3%					2.89E-3 4%					1.000
MDA	<2.E-3	<8.E-5	<2.E-4	<6.E-5	<6.E-5	<7.E-5	<3.E-4	<2.E-4	<4.E-4	<8.E-4	
02-822 AZ-A	1.38E+0 41%				4.98E-1 5%	1.39E+3 4%					1.189
MDA	<1.E+0	<2.E-2	<9.E-1	<2.E-1	<3.E-2	<2.E-1	<2.E-2	<3.E-2	<8.E-1	<3.E0	
02-822 DUP AZ-A	1.00E+0 56%				5.17E-1 5%	1.37E+3 4%					1.189
MDA	<1.E+0	<2.E-2	<9.E-1	<2.E-1	<3.E-2	<2.E-1	<3.E-2	<3.E-2	<8.E-1	<3.E0	
RPD	32%				4%	2%					
02-823 AZ-C	7.10E-1 17%				2.19E-1 5%	5.75E+2 4%					1.092
MDA	<3.E-1	<1.E-2	<6.E-1	<9.E-2	<2.E-2	<9.E-2	<2.E-2	<2.E-2	<5.E-1	<2.E0	
02-824 AZ-E	2.73E-1 22%				1.15E-1 3%	3.12E+2 3%					1.033
MDA	<2.E-1	<1.E-3	<2.E-1	<6.E-2	<4.E-3	<3.E-2	<4.E-3	<5.E-3	<8.E-2	<8.E-2	

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

ALO ID Client ID		Sr-90 Error %	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Density g/ml
02-825 AZ-G		1.42E+0 5%					1.50E+2 4%					1.121
	MDA	<2.E-1	<1.E-2	<3.E-1	<5.E-2	<2.E-2	<5.E-2	<2.E-2	<2.E-2	<3.E-1	<9.E-1	
02-826 AZ-I		9.56E-1 3%				3.21E-2 3%	8.84E+1 2%					1.027
	MDA	<3.E-2	<8.E-4	<6.E-2	<3.E-2	<2.E-3	<2.E-2	<3.E-3	<2.E-3	<5.E-2	<7.E-2	
02-827 AZ-K		4.09E-1 4%				1.53E-2 7%	4.42E+1 2%					1.033
	MDA	<2.E-2	<2.E-3	<7.E-2	<3.E-2	<2.E-3	<2.E-2	<5.E-3	<3.E-3	<7.E-2	<9.E-2	
02-828 AZ-M		3.65E-1 4%				7.66E-3 7%	2.08E+1 3%					1.003
	MDA	<2.E-2	<5.E-4	<4.E-2	<3.E-2	<8.E-4	<8.E-3	<3.E-3	<2.E-3	<2.E-2	<2.E-2	

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

ALO ID Client ID		Sr-90 Error %	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
02-829 Ni AZ-0		1.58E+4 3%	2.06E+0 2%	8.89E+0 5%		7.47E-1 5%	2.13E+3 3%		2.45E+1 2%	2.87E+1 5%	4.77E+1 4%
	MDA	<2.E+2	<4.E-2	<1.E+0	<5.E-1	<7.E-2	<3.E-1	<2.E-1	<2.E-1	<1.E+0	<2.E+0
02-830 Ni AZ-2		3.04E+4 3%	3.75E+0 4%	1.82E+1 7%			8.07E+2 4%		4.29E+1 2%	5.81E+1 8%	5.66E+1 15%
	MDA	<3.E+2	<2.E-1	<4.E+0	<6.E-1	<5.E-1	<7.E-1	<8.E-1	<6.E-1	<6.E+0	<2.E+1
02-831 Ni AZ-4		2.36E+4 3%	3.26E+0 4%	1.42E+1 7%			5.34E+2 4%		3.64E+1 2%	5.09E+1 8%	5.11E+1 14%
	MDA	<2.E+2	<2.E-1	<3.E+0	<5.E-1	<4.E-1	<7.E-1	<7.E-1	<6.E-1	<5.E+0	<2.E+1

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

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
02-832B1 Ni Process Blank	6.70E-2 16%					9.12E-2 4%				
MDA	<2.E-2	<3.E-3	<8.E-3	<3.E-3	<3.E-3	<3.E-3	<2.E-2	<6.E-3	<1.E-2	<2.E-2
02-832B2 Ni Process Blank	6.66E-2 17%					1.24E-1 3%				
MDA	<2.E-2	<2.E-3	<5.E-3	<2.E-3	<2.E-3	<2.E-3	<6.E-3	<4.E-3	<7.E-3	<2.E-2
02-832 Ni AZ-6	6.41E+4 3%	8.66E+0 2%	3.88E+1 3%			6.41E+2 3%	1.45E+0 14%	1.03E+2 2%	1.22E+2 5%	2.04E+2 4%
MDA	<8.E+2	<2.E-1	<2.E+0	<6.E-1	<3.E-1	<5.E-1	<4.E-1	<4.E-1	<3.E+0	<4.E+0
02-832 Ni DUP AZ-6	5.78E+4 3%	8.19E+0 2%	3.84E+1 3%			6.41E+2 3%	1.71E+0 10%	9.93E+1 2%	1.17E+2 4%	1.91E+2 4%
MDA	<9.E+2	<2.E-1	<2.E+0	<6.E-1	<3.E-1	<5.E-1	<4.E-1	<5.E-1	<3.E+0	<4.E+0
RPD	10%	6%	1%			0%	16%	4%	4%	7%
MS 02-828 AZ-M	93%									
Blank Spike	103%									
Lab Blank	<1.E-4									

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

02-822
05/30/02

Client : Geeting
ASR: 6284

Cognizant Scientist: LR Greenwood Date : 5/30/02

Concur : T Trang-le Date : 5/31/02

Reference Date: Feb. 4, 2002 for Gea
Procedure: PNL-ALO-450 for GEA

Additional Gamma Emitters Requested for Sample AZ-6
Measured Activities (uCi/g)

RPL ID Client ID		Cr-51	Fe-59	Y-88	Nb-95	Ru-103	Ru-106	Sn-113	Ce-144	Th-232
02-832B1 Ni Process Blank	MDA	< 3.E-2	< 5.E-3	< 3.E-3	< 3.E-3	< 3.E-3	< 3.E-2	< 4.E-3	< 2.E-2	< 6.E-3
02-832B2 Ni Process Blank	MDA	< 2.E-2	< 3.E-3	< 2.E-3	< 2.E-3	< 2.E-3	< 2.E-2	< 2.E-3	< 2.E-2	< 3.E-3
02-832 Ni AZ-6	MDA	< 4.E+0	< 4.E-1	< 3.E-1	< 3.E-1	< 5.E-1	< 3.E+0	< 6.E-1	< 5.E+0	< 1.E+0
02-832 Ni DU AZ-6	MDA	< 5.E+0	< 4.E-1	< 3.E-1	< 3.E-1	< 5.E-1	< 3.E+0	< 7.E-1	< 5.E+0	< 1.E+0

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

04/11/02

Client : Geeting
ASR: 6284

Cognizant Scientist: L.R. Greenwood Date : 4/11/02

Concur : T Trang-le Date : 4/12/02

Procedure: PNL-ALO-4001/408 for Alpha/Beta
Procedure: PNL-ALO-417/422 for Am/AEA

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

		Alpha Energy Analysis				
RPL ID Client ID	Alpha Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Sum of Alpha* Error %	Density g/ml
02-822BLK Process Blank	4.41E-4 18%	2.00E-4 5%	7.04E-5 8%		2.70E-4 4%	1.00
MDA	<3.E-4	<6.E-6	<4.E-6	<3.E-6		
02-822 AZ-A		1.65E-5 19%	7.06E-6 29%		2.36E-5 16%	1.189
MDA	<4.E-3	<6.E-6	<4.E-6	<3.E-6		
02-822 DUP AZ-A		3.70E-5 12%	8.06E-5 8%		1.18E-4 7%	1.189
MDA	<4.E-3	<6.E-6	<5.E-6	<3.E-6		
RPD		76%	168%		133%	
02-823 AZ-C		1.74E-5 19%			1.74E-5 19%	1.092
MDA	<4.E-3	<6.E-6	<3.E-6	<3.E-6		
02-824 AZ-E		1.22E-5 27%			1.22E-5 27%	1.033
MDA	<5.E-3	<8.E-6	<6.E-6	<4.E-6		
02-825 AZ-G		1.89E-5 16%	7.60E-6 27%		2.65E-5 14%	1.121
MDA	<4.E-3	<4.E-6	<4.E-6	<3.E-6		
02-826 AZ-I		9.72E-5 7%	5.98E-5 9%		1.57E-4 6%	1.027
MDA	<4.E-3	<6.E-6	<5.E-6	<3.E-6		
02-827 AZ-K		3.50E-5 15%	1.28E-5 24%		4.78E-5 13%	1.033
MDA	<5.E-3	<8.E-6	<5.E-6	<4.E-6		

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

		Alpha Energy Analysis				
RPL ID Client ID	Alpha Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Sum of Alpha* Error %	Density g/ml
02-828 AZ-M		6.47E-4 4%	4.78E-4 5%		1.13E-3 3%	1.003
MDA	<6.E-3	<1.E-5	<7.E-6	<6.E-6		
02-828 Dup AZ-M		6.20E-4 4%	4.95E-4 5%		1.12E-3 3%	1.003
MDA	<6.E-3	<2.E-5	<6.E-6	<7.E-6		
RPD		4%	4%		1%	
Matrix Spike 02-828 AZ-M	110%	89%				
Blank Spike	105%	95%				
Lab Blank		7.95E-6 38%				
MDA	<5.E-5	<8.E-6	<7.E-6	<5.E-6		

*Note: The sum of the alpha emitters does not represent the total alpha activity since Pu analyses were not requested for these samples.

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Radiochemical Science & Engineering -325 Building

Client : Geeting
ASR: 6284

04/12/02

Cognizant Scientist: LRGreenwood Date : 4/12/02

Concur : T Trang-le Date : 4/12/02

Procedure: PNL-ALO-417/422 for Pu and Am/AEA
Procedure: PNL-ALO-4001 for Alpha/Beta

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

			Alpha Energy Analysis						
RPL ID Client ID		Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Sum of Alpha Error %
02-829 Ni AZ-0		4.12E+1 12%	2.62E+0 11%	2.74E-1 42%		4.36E+1 2%			4.65E+1 2%
	MDA	<1.E+1	<4.E-1	<4.E-1	<3.E-1	<2.E-1	<2.E-1	<7.E-2	
02-829 Ni DUP AZ-0			2.26E+0 8%	1.98E-1 27%		3.92E+1 2%			4.17E+1 2%
	MDA		<9.E-2	<2.E-1	<8.E-2	<2.E-1	<2.E-1	<9.E-2	
RPD			15%	32%		11%			11%
02-830 Ni AZ-2		9.67E+1 7%	4.13E+0 9%	5.13E-1 25%		7.79E+1 2%	1.92E-1 28%	8.42E-2 40%	8.28E+1 2%
	MDA	<9.E+0	<3.E-1	<3.E-1	<2.E-1	<2.E-1	<2.E-1	<8.E-2	
02-831 Ni AZ-4		7.64E+1 8%	3.35E+0 7%	4.56E-1 19%		6.60E+1 2%	1.49E-1 34%		7.00E+1 2%
	MDA	<9.E+0	<2.E-1	<2.E-1	<2.E-1	<2.E-1	<2.E-1	<7.E-2	
02-832 Ni AZ-6		1.95E+2 5%	9.38E+0 5%	1.07E+0 13%		1.66E+2 3%			1.76E+2 3%
	MDA	<8.E+0	<3.E-1	<2.E-1	<2.E-1	<5.E-1	<5.E-1	<3.E-1	

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

RPL ID Client ID	Alpha Error %	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %	Sum of Alpha Error %
		Alpha Energy Analysis						
02-832 Ni DUP AZ-6	1.80E+2 5%	9.78E+0 5%	1.12E+0 11%		1.64E+2 3%	2.98E-1 40%	2.98E-1 40%	1.75E+2 3%
MDA	<1.E+1	<1.E-1	<2.E-1	<7.E-2	<4.E-1	<3.E-1	<3.E-1	
RPD	8%	4%	5%		1%			1%
02-832B1 Ni Process Blank		5.44E-4 6%	8.54E-4 5%		8.21E-4 5%	2.04E-4 10%		2.42E-3 3%
MDA	<5.E-3	<3.E-5	<3.E-5	<2.E-5	<2.E-5	<2.E-5	<2.E-5	
02-832B2 Ni Process Blank		8.20E-4 5%	9.90E-4 5%		7.70E-4 5%	1.03E-4 14%		2.68E-3 3%
MDA	<5.E-3	<2.E-5	<2.E-5	<9.E-6	<3.E-5	<2.E-5	<2.E-5	
Matrix Spike*	110%	99%			90%			
Blank Spike	105%	112%			102%			
Lab Blank		1.72E-5 35%						
MDA	<5.E-5	<2.E-5	<2.E-5	<2.E-5	<3.E-5	<2.E-5	<1.E-5	

*Note: The matrix spike was performed on sample AZ-M for total alpha, AZ-O for Pu and AZ-6 for Am.

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Project No. 42365

Internal Distribution
File/LB

NH3
SM-151

Date July 11, 2002

To J. Geeting

From L. R. Greenwood

Subject Radiochemical Analyses for ASR 6284

Samples of the supernate, washes, rinses, leaches and slurries for tank AZ101 (02-0822-832) were analyzed for gamma emitters, ^{90}Sr, total alpha, Am/Cm, Pu, and Tc according to ASR 6284. The analyses were performed on sample materials prepared by acid digestion (liquids) or fusions (dried slurries) in the hot cells. The attached reports list measured analyte activities in units of µCi/ml (liquids, corrected for density) or uCi/g of dried solids. 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.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. Only ^{134}Cs and ^{137}Cs could be detected in the liquids; however, most of the requested isotopes were detected in the solids. In a number of cases, it was not possible to meet the requested MRQ values in extended counting due to the very high levels of ^{137}Cs activity. Minimum detectable activity (MDA) values are reported for all of the requested isotopes including the additional request list for the slurry sample AZ-6. Hot cell process blanks showed negligible activity relative to the samples. The ^{241}Am results for the solids are in reasonable agreement with the alpha energy analysis results reported below, although the AEA results have lower uncertainties in most cases.

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). ^{137}C was detected in a few of the samples and suitable small corrections were made to the beta counting results. The process blank prepared with the acid digestion in the laboratory was found to have a low level of ^{90}Sr contamination that corresponds to 20% of the activity in sample AZ-E, 16% for AZ-M, 14% for sample AZ-K, 12% for AZ-C, and less than 10% for the other samples. The solids hot cell process blank was negligible with respect to the samples. The reagent blank did not show any contamination. The LCS and matrix spike showed good recovery at 103% and 93%, respectively. Duplicate analyses showed acceptable agreement taking into account the uncertainties in the data. Some of the uncertainties are relatively high for some of the liquid samples due to the large correction to the beta counting from the ^{85}Sr tracer. The detection limits as well as many of the measurements were well below the requested MRQ values, except in cases where the measured ^{90}Sr activities greatly exceeded the MRQ values.

Total Alpha

The total alpha activity was determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. Alpha activity could not be detected in the liquid samples; the detection limits are quite low, although there was no MRQ value requested for the liquids. A better estimate of the total alpha activity is given by the sum of the alpha emitters, as discussed below. Relatively high levels of total alpha activity were detected in all of the slurry samples, well above the requested MRQ value. Duplicate results are in good agreement for the slurry sample AZ-6. No significant alpha activity was seen in the hot cell or laboratory blanks. Blank and matrix spikes gave good recoveries at 105% and 110%, respectively.

Plutonium, Americium and Curium

The Pu and Am/Cm separations were performed according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and counted by alpha spectrometry according to PNL-ALO-422. The plutonium activities were determined with a ^{242}Pu tracer. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. For the liquid samples, only Am/Cm analyses were requested. The hot cell process blank was found to contain significant contamination with both Am and Cm isotopes, generally exceeding the alpha activities in the samples except for sample AZ-M. This hot cell contamination probably explains the poor agreement for sample AZ-A, which is below the hot cell level, compared to the good agreement for sample AZ-M, which is somewhat above the hot cell level of contamination. In any case, all of the results for the liquids (including the hot cell blank) are below the requested MRQ value of 7.2E-4 uCi/ml. Sample AZ-M is only slightly below the requested MRQ value; however, this sample is the least affected by the hot cell contamination. Negligible contamination was seen in the lab blank. The LCS recovery was 95% and the matrix spike recovery was 89%.

Both Am/Cm and Pu analyses were requested for the solid samples. In this case, the alpha activities in the hot cell process blanks as well as the lab blank were negligible with respect to the sample activities. Duplicate results for samples AZ-0 and AZ-6 were in reasonable agreement, taking into account the measurement uncertainties. The Pu, Am, and Cm activities in the samples were well above the requested MRQ values. The LCS recoveries were 103% and 102% for ^{242}Pu and ^{243}Am, respectively. Matrix spike recoveries were about 90% in both cases. The sum of the individual alpha activities is in good agreement with the total alpha activities. The alpha emitter sum is the best estimate of the total alpha activity due to the lower uncertainties for this method.

Tritium

Tritium was distilled from direct slurry samples according to procedure PNL-ALO-418 and measured by liquid scintillation counting according to procedure PNL-ALO-474. The procedure was modified to include a cation exchange and a second distillation to ensure the removal of relatively high levels of ^{90}Sr and ^{137}Cs in these samples. This procedural modification is documented in the raw data file. This resulted in very clean tritium beta spectra with no detectable beta contamination. Tritium was detected in all of the slurry samples above the requested MRQ value of 1.5E-2 uCi/g. Duplicate analyses were in excellent agreement. The laboratory LCS recovery was 87% and the hot cell LCS recovery was somewhat low at 72%. The matrix hot cell spike recovery was 101%. No tritium was detected in either the hot cell or laboratory blanks.

Tc-99 as Pertechnetate

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated fractions were then counted according to procedure RPG-CMC-408. The LCS blank spike recovery of a ^{99}Tc standard was 83%, and a matrix spike of sample AZ-M gave a standard recovery of 95%. Sample duplicates showed good agreement with an RPD of 3%. The ^{99}Tc activities in the samples were well above the requested MRQ value of 1.5E-3 uCi/ml.

Pu-241

The ^{241}Pu activity in the samples was determined by placing the co precipitation alpha mounts from the Pu/AEA separation procedure, as described above, into liquid scintillation cocktail. The beta activity was then measured by liquid scintillation counting according to procedure PNL-ALO-474. The chemical yield was taken from the ^{242}Pu yields measured during the alpha counting. The 241Pu counting efficiency was determined by co precipitating a ^{241}Pu standard. Although results were not requested for sample AZ-0, this sample had to be analyzed since this was used for the matrix spike measurement. Duplicate results are in good agreement. Negligible activity was seen in the hot cell process blanks or the lab blank. The matrix spike yield was 77%; however, the 1-sigma uncertainty is 11%. The LCS yield was 91%. No MRQ value was specified.

Sm-151

Rare earths are separated along with Am/Cm in our procedure PNL-ALO-417, as described above. The precipitation mounts that were used for the Am/Cm alpha energy analyses were thus counted on a liquid scintillation counter according to procedure PNL-ALO-474 to determine the beta activity due to ^{151}Sm. The recovery of the ^{243}Am tracer used for the alpha measurements was used to correct the ^{151}Sm data. LCS and matrix spike samples prepared at the time of the Am/Cm chemical separations gave recoveries of 75% and 72%, respectively. Duplicate results for sample AZ-6 are in good agreement. The hot cell process blanks as well as the lab blank had negligible activity compared to the samples. Other rare earths are also separated along with the Sm. However, the only significant activity in highly decayed Hanford tank waste is due to ^{154}Eu. The ^{154}Eu activity was measured by gamma energy analysis, as described above. The beta energy spectrum measured during the liquid scintillation counting shows a weak, higher-energy component that appears to be due to ^{154}Eu. The ratio of the ^{154}Eu activity under the ^{151}Sm beta peak was estimated to be about 15-20% and suitable subtractions were made to determine the final ^{151}Sm activities. Most of the stated uncertainty in the data is due to this correction. The MRQ value was not specified for this isotope.

Ammonia

Ammonia was measured by ion selective electrode, using procedure RPG-CMC-226. The liquid samples were preserved in a hot cell by acidifying them with dilute sulfuric acid. The samples, preserved by acidification, were removed from the cell for ammonia measurement in a laboratory hood. The solid samples dissolved in dilute sulfuric acid, then surveyed out of the hot cell and taken to a laboratory for ammonia analysis. Laboratory control samples, matrix spikes, duplicates, and hot cell blanks were prepared with the samples.

The ammonia concentration in the samples was measured by both direct measurement and by standard addition. The detection limit of the ammonia probe was around 10^{-7} molar, and the lower end of the linear range of the probe was about 1.5×10^{-5} molar. All the samples had detectable ammonia, but below the linear range of the probe.

The samples were analyzed in three batches. Out of eleven instrument standards analyzed in these three batches, two fell outside the ±10% limit (123% and 78%). None of the instrument blanks had significant ammonia.

The laboratory control standards and matrix spikes all fell within limits. The hot cell blanks had ammonia well below the MRQ and below all but one sample result.

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

File: 02-0822.xls
7/3/2002

Client : Geeting
ASR: 6284

Cognizant Scientist: C Soderquist Date : 7-10-02

Concur : LR Greenwood Date : 7-10-02

Procedure RPG-CMC-4014, Rev 0, *Ammonia Analysis by Ion Selective Electrode*

RPL ID Client ID	Measured Concentration with 1-sigma error µg NH_3 per mL		EQL	MRQ
02-822 AZ-A	5.25E-1	± 20%	2.0E+0	1.00E+2
02-822 Dup AZ-A	2.85E+0	± 20%	1.0E+1	1.00E+2
02-823 AZ-C	1.10E+1	± 20%	1.0E+1	1.00E+2
02-823 Dup AZ-C	3.61E+0	± 20%	1.0E+1	1.00E+2
02-824 AZ-E	2.12E+0	± 23%	1.0E+1	1.00E+2
02-825 AZ-G	2.65E+0	± 23%	1.0E+1	1.00E+2
02-826 AZ-I	8.10E+0	± 23%	1.0E+1	1.00E+2
02-827 AZ-K	1.58E+0	± 23%	1.0E+1	1.00E+2
02-828 AZ-M	2.03E+0	± 23%	1.0E+1	1.00E+2
Hot cell blank 1	9.84E-1	± 23%	1.0E+1	1.00E+2
Hot cell blank 2	8.00E-1	± 23%	1.0E+1	1.00E+2
LCS 1	97%			
LCS 2	75%			
Matrix spike 824	121%			
Matrix spike 825	116%			

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

File: 02-0822.xls
7/3/2002

Client : Geeting
ASR: 6284

RPL ID Client ID	Measured Concentration with 1-sigma error µg NH_3 per g 2M solids		EQL	MRQ
02-829 AZ-0	4.57E+1	± 9%	7.6E+1	6.00E+1
02-830 AZ-2	5.79E+1	± 10%	6.0E+1	6.00E+1
02-831 AZ-4	7.48E+1	± 10%	9.2E+1	6.00E+1
02-832 AZ-6	3.18E+1	± 28%	6.5E+1	6.00E+1
02-832 Dup AZ-6	3.25E+1	± 8%	6.5E+1	6.00E+1
Hot cell blank 832	1.95E+1	± 16%	6.2E+1	6.00E+1
Matrix spike 832	122%			
LCS 832	119%			

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

7/2/2002

Client : Geeting
ASR: 6284

Cognizant Scientist: LRGreenwood Date : 7-2-02

Concur : C-Soderquist Date : 7-2-02

Procedure: PNL-ALO-417/474
Reference Date: 5/22/2002

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

RPL ID	Sm-151		
Client ID	uCi/g	Error +/-	MDA
02-832 AZ-6	1.40E+3	10%	<1.E+1
02-832 dup AZ-6	1.57E+3	10%	<1.E+1
RPD	11%		
02-832 B1 Process Blank	2.08E-3	10%	<1.E-3
02-832 B1 Process Blank	1.71E-3	12%	<1.E-3
Matrix Spike 02-832 AZ-0	72%		
LCS	75%		
Lab Blank			<1.E-3

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Date: 02/19/02

Subject:	**Se-79** Analysis Report on:	Client ID AZ-101 SW/CL slurry AZ-6	
Project:	42365		

ASR **6284**
Sample RPG ID **02-0832**
WP# W-60513

To: John Geeting

Se-79 was measured in duplicate on the Fusion Fraction of AZ101 -SW/CL Slurry and its lab duplicate according to procedure PNL-ALO-440 and the results are listed below. Since Se-79 is not available as a radioactive standard, it was not possible to provide a LCS or matrix spike. Se carrier was used in the analysis for establishing the yield and C-14 was used to establish the instrument efficiency since it has a very similar beta max energy (156 Kev vs 149 Kev for Se-79). One mL aliquots of the KOH fused diluted slurry material provided to the lab were analyzed. The gravimetric recoveries for the reagent blank, lab blank, and samples, are listed below. The Se-79 activities were measured by liquid scintillation counting according to procedure PNL-ALO-474. No peaks were observed in the Se-79 R.O.I. beta energy spectral plots provided and no other higher energy beta contaminants were observed. A second count was performed to determine reproducablity and this data showed excellent agreement with the first count data. The average MDC value was < 1.6E-3 uCi/g. No MRQ value was provided for Se-79.

		Se	Se-79 Result	Se-79 1s TPU	Se-79 MDC	
I.D.	Client ID	Recovery	uCi/g	uCi/g	uCi/g	TPU,%
Reag Blk		0.68	-3.42E-7	8.36E-7	2.88E-6	244%
02-0832-B	Process Blk	0.57	1.77E-6	1.09E-6	3.44E-6	61%
02-0832-B-2	Process Blk	0.61	1.87E-7	9.49E-7	3.19E-6	508%
02-0832	AZ-101 SW/CL	0.74	1.83E-4	3.66E-4	1.21E-3	200%
02-0832-rep	AZ-101 SW/CL	0.57	2.24E-5	4.64E-4	1.57E-3	2074%
02-0832-dup	AZ-101 SW/CL	0.62	-2.71E-4	4.61E-4	1.60E-3	170%
2-0832-dup re	AZ-101 SW/CL	0.48	4.98E-4	6.19E-4	2.03E-3	124%
	AZ-6	Ave =	1.08E-4		**1.6E-3**	
		Std. Dev.=	3.21E-4			

Prepared by: ____________________ Date: 2/19/02

Reviewed by: LR Greenwood Date: 2/19/02

Revd
2-22-02

Se-79 Analysis --- via RPG-CMC-440 ASR # 6284

This procedure involves an anion/cation exchange to remove most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences elimination by the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. The Se is finally dissolved and counted by liquid scintillation using C-14 (prep'd in the same matrix configuration) for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV.

Se-79 Procedure Flow Diagram

Add 20 mg elemental Se (2 mL of 10mgSe/mL to each aliquot for analysis

↓

.Prep plastic disposable columns:
1mL AG--50W: X 8 100-200 mesh H+ +
1ml AG-1, X-4 100-200 Cl-

↓

Pass sample through column and collect in 35 mL vial. Wash with 4-1ml portions of 0.5 M HNO3

↓

Add 10ml hydroxlamaine hydrochloride to sample in a glass vial. Cap and place the vial in a heating block and heat @ 90C for ~30 min (I.e. just until black ppt forms).

↓

.Dissove ppt in a few drops of conc HNO3. Add 5 mL of water and repeat the NH2OH.HCl addition and precipitation. Then dissolve in ~ 0.5 M HNO3 for transfer to distillation flask.

Set up still by placing Nitrogen purge ~100ml boiling flask in heating mantle connected to a variable power supply and supported on a ring stand.

↓

Transfer the solution to the boiling flask rinsing the 35mL vial with 2-5mL portions of concentrated HBr.

↓

Place a rinsed (clean) 35ml vial on the exit port of the boiling flask. Add 5ml of 25% hydroxlamaine hydrochloride solution to this vial. Cool this vial in an ice bath.

↓

Connect nitrogen supply to top of flask and set flow to about 5 bubbles/sec. Then turn on power supply and adjust to 110 V amd distill for at least 35 min.

↓

Add 5 ml additional hydroxlamaine hydrochloride solution to the collection vial. Cap and place the vial in a heating block and heat for a couple hours. Black ppt. forms

Prep. and tare- weigh filter disks Centrifuge, decant most of solution and transfer black ppt. using ethanol to the top of filter mounted on vaccum system.

↓

Dry ppt. and obtain final wt. for Se grav. recovery. 20 mg =100% .

↓

Transfer filter + ppt. To glass LCS vial, add 2-3 drops of conc HNO3, allow to dissolve completely, then take to dryness. Add 2 mL 0.1 M HCL and 10 ml of LSC cocktail, place on labeled caps.

↓

Prepare C-14 reference stds. similarly and count all on Packard LSC 2550 for 2 X 30 min. (Prog. # 6).

Analysis raw data:

Sample ID.	Aliqout Vol. (mL)	Diluted to	Tare Wt (mg) Filter + holder	Final Wt. (mg)	Net Se Wt.	Recovery %
Reag BlK	1.00		745.8	759.3	13.5	68%
02-0832-B	1.00	Process Blk	746.8	758.1	11.3	57%
02-0832-B-2	1.00	Process Blk	748.1	760.3	12.2	61%
02-0832	1.00		753.8	768.5	14.7	74%
02-0832-rep	1.00		757.9	769.3	11.4	57%
02-0832-dup	1.00		747.4	759.7	12.3	62%
02-0832-dup	1.00		746.2	755.9	9.7	48%

2/15/02
Entered by Date

L.R. Greenwood 2/19/02
Reviewed by Date

Pipet verify check	0.1 mL	1.0 mL
Pipet #	78868	125592
	0.0998	0.9987
	0.1009	1.0032
	0.1012	1.0011
Ave	0.1006	1.0010
Std Dev.	0.0007	0.0023

WP# **W-60513**
ASR # **6284**

Se-79 Analysis

AZ-6---- AZ101-SW/CL Slurry
Fusion Fraction

Data File: r:\radchem\se79\se-**6284**.xls

Analysis for Se-79 was performed using PNL-ALO-440. This procedure involves an anion/cation exchange to remove of most radiochemical interferences followed by a Selenium Bromide distillation and minor interferences completely eliminated in the reduction of Se to elemental form. The ppt. recovered is used for gravimetric yield correction. No vendor supplied Se-79 source material is available, therefore C-14 was chosen for calibration since its beta energy max. of 156 keV is very similiar to Se-79 at 149 keV. W-115-1, a secondary dilution of NIST C-14 SRM 4222 , was used for the efficiency calibration of the liquid scintillation counter. These calibration standards were prepared in the same geometry as the prepared samples and at the same time the batch was prepared to monitor efficiency of the cocktail over time. Volume of W-115-1 used was 0.1ml.

Process Dat Start Date: 9/4/01
Se Carrier: Selenium Std. @ 10,000 ppm
CMS # 126666 Inor. Ventures Std
Vol. added: Expires: 1-Mar-02
2 mL = 20.0 mg

Performance checks
Balance # 360-06-01-026
Pipet # 125592
Lab Loc. 525

Sample ID	Leached Sample Vol (g)	Total dil. volume (g)	dil factor	Diln. Aliquot (mL)	Sample Analyzed (g)	Tare Wt. of filter & older (mg	Gross Wt. f filter plu e ppt.(mg	Net Wt. of Se (mg)	Wt. of Se added (mg)	Grav. Recovery %
Reag Blk	1.000				1.00	745.8	759.3	13.5	20.00	67.5
02-0832-B	na				1.00	746.8	758.1	11.3	20.00	56.5
02-0832-B-2	na				1.00	748.1	760.3	12.2	20.00	61.0
02-0832	0.2177	100	459.348	1.00	0.00218	753.8	768.5	14.7	20.00	73.5
02-0832-rep	0.2177	100	459.348	1.00	0.00218	757.9	769.3	11.4	20.00	57.0
02-0832-du	0.1978	100	505.561	1.00	0.00198	747.4	759.7	12.3	20.00	61.5
02-0832-du	0.1978	100	505.561	1.00	0.00198	746.2	755.9	9.7	20.00	48.5

2/15/02
Entered by Date

LR Greenwood 2/19/02
Reviewed by Date

SELENIUM-79 CALCULATIONS FOR LIQUID SAMPLES

Procedure PNL-ALO-440

Data file name: r:\radchem\se79\se-6284.xls
ate Counted 02/14/02
Sample Counting Time,min. 30.00

Entered by: rgs
Date Calc'd 11/13/01
LSC progam # 6

C-14 is used for efficiency since Beta Emax is very similar to Se-79.

T1/2 = 5715. yr

C-14 Std No. W-115-1 Activity= 266700 dpm/ml
Ref. date: 12/01/90 error= 4000 dpm/ml

I.D.	aliquot ml	error ml	Se-79 R.O.I. cpm		1S%
Reag. Blank #1	1.000	0.0000	12.64		5.14
Blank 2	1.000	0.0000	13.39		
		Avg =	**13.02**	cpm	
				cpm	

					1S%	efficiency	efficiency error
100ul Std - C-14 Spk 1	0.100	0.0003	19325.0		0.13	***0.726***	***0.0111***
100ul Std - C-14 Spk 1	0.100	0.0003	19928.4		0.13	***0.748***	***0.0115***
100ul Std -C-14 Spk 3	0.100	0.0003	19142.6		0.13	***0.719***	***0.0110***
					Avg efficiency =	***0.731***	
					1s error =	***0.0078***	
					%error	***1.07***	

SAMPLES

Requested activity units: uCi
Sample quantity units: g

.D.	Sample Vol. / mass	Sample units g or mL	Se Recovery	dilution Factor	aliquot frac. anal.	Se -79 R.O.I. cpm	ct. error 1 sigma %	***Se-79 Result*** ***uCi/g***	***Se-79 1s TPU*** ***uCi/g***	***Se-79 MDC*** ***uCi/g***	**TPU,%**
Reag BIK	1.000	mL	0.675	na	1	12.64	247	***-3.42E-07***	***8.36E-07***	***2.88E-06***	**244%**
02-0832-B	1.000	mL	0.565	na	1	14.64	59	***1.77E-06***	***1.09E-06***	***3.44E-06***	**61%**
02-0832-B-2	1.000	mL	0.610	na	1	13.20	505	***1.87E-07***	***9.49E-07***	***3.19E-06***	**508%**
02-0832	1.000	mL	0.735	459.3	0.00218	13.49	198	***1.83E-04***	***3.66E-04***	***1.21E-03***	**200%**
02-0832-rep	1.000	mL	0.570	459.3	0.00218	13.06	2071.8	***2.24E-05***	***4.64E-04***	***1.57E-03***	**2074%**
02-0832-dup	1.000	mL	0.615	505.6	0.00198	12.48	172.3	***-2.71E-04***	***4.61E-04***	***1.60E-03***	**170%**
02-0832-dup	1.000	mL	0.485	505.6	0.00198	13.79	122.0	***4.98E-04***	***6.19E-04***	***2.03E-03***	**124%**
							Ave	***1.08E-04***			
							Std dev.	***3.21E-04***			
							% 1s	***297%***			

Bowen 2/19/02
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LR Greenwood 2/19/02
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Date March 25, 2002

To J. Geeting

From L. R. Greenwood LRG

Subject ^{14}C Analyses for AZ101 Samples – ASR 6284

Direct slurry samples (02-0829-0832) from tank AZ-101 were subaliquoted in the hot cells and analyzed in the laboratory for ^{14}C according to procedure PNL-ALO-482. Following sample combustion, the collected $^{14}CO_2$ was determined by liquid scintillation counting according to procedure PNL-ALO-474. The attached report lists the measured ^{14}C activities in units of μCi/g of wet slurry. Results have not been corrected for the wt% solids. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. The ^{14}C spike recovery through the combustion and collection procedure averaged 94% for three standards. A matrix spike showed excellent recovery at 100%. Duplicate analyses for sample AZ-6 showed good agreement with an RPD of 7%. No activity was detected in the laboratory blank. A hot cell blank was not provided when the samples were aliquoted for distribution to the laboratory. Periodic rinses analyzed with the batch of samples did not show any significant ^{14}C retention by the furnace and gas collection system. The ^{14}C measured values were less than the requested MRQ value of 1.8E-3 uCi/g except for sample AZ-0 which was slightly higher than the MRQ value.

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Radiochemical Science & Engineering -325 Building

File: 02-0822.xls

Client : Geeting
ASR: 6284

03/25/02

Cognizant Scientist: L. R. Greenwood Date : 3/25/02

Concur : Marilyn J. Steele Date : 3/25/02

C-14 Procedure PNL-ALO-482; Reference Date: 3/5/02

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

ALO ID Client ID	C-14	Error%	MDA
02-829 Ni AZ-0	2.22E-3	5%	2E-4
02-830 Ni AZ-2	1.02E-3	6%	2E-4
02-831 Ni AZ-4	1.09E-3	5%	1E-4
02-832 Ni AZ-6	6.53E-4	12%	2E-4
02-832 Ni Dup AZ-6	7.00E-4	10%	2E-4
RPD	7%		
Lab Blank			2E-4
Lab reagent spike	94%	Average of two LCS spikes	
Matrix Spike1845	100%		

Note: Results are currently reported per wet weight.

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