PNNL-3050
BNFL-RPT-029, Rev. 0

Development and Demonstration of a Sulfate Precipitation Process for Hanford Waste Tank 241-AN-107

S. K. Fiskum
D. E. Kurath
B. M. Rapko

August 2000

Prepared for BNFL, Inc.
under Contract W375-LC-98-4168

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Battelle, Pacific Northwest Division
Richland, Washington 99352

Summary

A series of precipitation experiments were conducted on Hanford waste tank 241-AN-107 samples in an effort to remove sulfate from the matrix. Calcium nitrate was added directly to AN-107 sub-samples to yield several combinations of $Ca:CO_3$ mole ratios spanning a range of 0:1 to 3:1 to remove carbonate as insoluble $CaCO_3$. Similarly barium nitrate was added directly to the AN-107 aliquots, or to the calcium pretreated AN-107 aliquots, giving of $Ba:SO_4$ mole ratios spanning a range of 1:1 to 5:1 to precipitate sulfate as $BaSO_4$.

Initial bulk carbonate removal was required for successful follow-on barium sulfate precipitation. A $\geq$ 1:1 mole ratio of $Ca:CO_3$ was found to lower the carbonate concentration such that Ba would react preferentially with the sulfate. A follow-on 1:1 mole ratio of $Ba:SO_4$ resulted in 70% sulfate removal. The experiment was scaled up with a 735-mL aliquot of AN-107 for more complete testing. Calcium carbonate and barium sulfate settling rates were determined and fates of selected cations, anions, and radionuclides were followed through the various process steps. Seventy percent of the sulfate was removed in the scale-up test while recovering 63% of the filtrate volume.

Surprisingly, during the scale-up test, a sub-sample of the $CaCO_3$/241-AN-107 slurry was found to lose fluidity upon standing for $\leq$ 2 days. Metathesis with $BaCO_3$ at ambient temperature was also evaluated using batch contacts at various $BaCO_3:SO_4$ mole ratios with no measurable success.

Terms and Abbreviations

AEA	alpha energy analysis
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
EDTA	ethylenediaminetetriacetate
EPA	environmental protection agency
GEA	gamma energy analysis
HEDTA	N-(2-hydroxyethyl)ethylenediaminetriacetate
HLRF	High Level Radiation Facility
IC	ion chromatography
ICP-AES	inductively-coupled plasma atomic emission spectrometry
ICP-MS	inductively-coupled plasma mass spectrometry
LAW	low-activity waste
MRQ	minimum reportable quantity
NA	not analyzed
SAL	Shielded Analytical Laboratory
TCLP	toxicity characteristic leaching procedure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
VSL	Vitreous States Laboratory
XRD	X-ray diffraction

Units

°C	degrees centigrade or celsius
cm	centimeter
g	gram
M	molarity (moles per liter)
mL	milliliter
µg	micrograms
µCi	microcuries
µm	micrometer

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1.0 Introduction

The presence of sulfate ion in the Hanford low-activity waste (LAW) solutions generates several potential processing difficulties. Preliminary testing of the LAW vitrification system at the Vitreous States Laboratory (VSL) indicates a separate molten sulfur layer will form in the melter (nominally at 1150°C) at sufficiently high sulfate concentrations. A molten sulfur layer in the LAW melter can lead to accelerated corrosion of the melter and unacceptable operating conditions (e.g., steam explosion).

BNFL Inc. (BNFL) has been evaluating several methods to mitigate the impacts of sulfate on the LAW vitrification system including pretreatment technologies, blending of high and low sulfate LAW solutions, modification to the LAW glass formulations, and volatilization of sulfur in the LAW melter as SO_2 or SO_3. BNFL is evaluating four pretreatment technologies for separating sulfate from LAW solutions:

- Ion Exchange (SuperLig® 655)
- Evaporation
- Precipitation
- Low-temperature crystallization

This report describes the results of a series of experiments conducted by Battelle to evaluate precipitation as a process for removal of sulfate ion from the waste currently stored in tank 241-AN-107 (referred to as AN-107). The pretreated AN-107 liquid waste (supernatant) is categorized as LAW Envelope C waste. Actual tank waste samples were used in these tests.

2.0 Experimental

2.1 Sample Description

The initial testing was conducted with a sample of waste from tank 241-AN-107 archived at Battelle after previous testing (Hendrickson, 1997). This sample was originally obtained as a multiple grab samples in January of 1997. It was diluted with 0.53M NaOH to approximately 5M Na and 0.125M OH. Cesium was removed using a crystalline silico-titanate ion exchanger. The cesium-decontaminated samples were transferred to the Radiochemical Processing Laboratory (RPL) on May 23, 1997 and were archived in the Shielded Analytical Laboratory (SAL). One-liter aliquots of this archive material were processed with two slightly different Sr/TRU removal processes (Hallen et al., 2000a). The first treatment process described provided material called "Archive 1 AN-107" in this report and was used for phase 1 studies. The second treatment process described by Hallen provided material called "Archive 2 AN-107" in this report and was used for phase 2 studies.

A sample of the waste from 241-AN-107 (Envelope C) was received in the High Level Radiation Facility (HLRF) during the fourth quarter of 1998. The homogenization, dilution, caustic adjustment, and sub-sampling of this sample was described (Urie et al., 1999). The diluted AN-107 sample (AN-107 Diluted Feed) was then processed for Sr/TRU removal with a precipitation process. The precipitate and entrained solids were removed with a single-tube cross-flow filter using a 0.1-micron sintered metal Mott filter (Hallen 2000b). The clarified AN-107 sample was then processed for cesium removal using an ion exchange process (Kurath et. al., 2000). The cesium-decontaminated sample was then processed for removal of ^{99}Tc (Blanchard, 2000). The effluent from the ^{99}Tc removal testing provided the feed, called "Actual AN-107," for phase 3 sulfate removal tests described in this report.

The AN-107 feed compositions for each experimental phase are provided in Table 2.1.

2.2 Reagents

All reagents were ACS reagent grade, with the exception of $Ba(NO_2)_2 \cdot H_2O$ (Aesar) which did not specify a grade. A 5M Ca^{2+} solution (density = 1.531g/mL) was prepared from $Ca(NO_3)_2 \cdot 4H_2O$. A 0.33M Ba^{2+} solution (density = 1.063g/mL) was prepared from $Ba(NO_3)_2$, two Ba stock solutions were prepared from $Ba(NO_2)_2 \cdot H_2O$. The $Ba(NO_2)_2$ solutions were analyzed by ICP-AES, to ascertain the actual Ba concentration.

2.3 Apparatus

Settling studies were conducted in a 1-L glass graduated mixing cylinder (6-cm internal diameter). Nalgene® filter assemblies with 20-cm^2, 0.45-µm nylon filter connected to 125-mL upper and lower reservoirs were used for phase 1 and phase 2 experiments. Millipore® filter assemblies with 40-cm^2 surface area, 0.45-µm polyvinylidene fluoride filter, connected to a polycarbonate 500-mL, graduated upper reservoir and 1-L, graduated lower reservoir were used for the third phase. Syringe filters (0.45-µm) were used to process small-volume samples. Vacuum was supplied by the laboratory vacuum system at 19.8-mm Hg pressure.

Table 2.1. Composition of AN-107 Feed Materials

Analyte	Units	Archive 1 AN-107 Phase 1	Archive 2 AN-107 Phase 2	Actual AN-107 Phase 3
density	g/mL	1.20	1.259	1.231
SO_4	M	0.041	0.053	0.042
CO_3/titration	M	0.7	0.81	
CO_3/TIC	M		0.70	0.66
NO_3	M		2.2	1.7
NO_2	M		0.73	0.62
OH	M		0.87	
HEDTA	M	0.0039		
EDTA	M	0.01		
Na	M		4.4	4.8
Ca	M		0.0041	0.0043
Al	µg/mL		127	2340
Ba	µg/mL		<0.4	<0.4
Cr	µg/mL		35	43
Sr	µg/mL		120	130
Cs-137	µCi/mL		2.02E-2	8.22E-2
Eu-154	µCi/mL		2.66E-2	3.26E-2
Am-241	µCi/mL		9.73E-3	1.19E-2
Co-60	µCi/mL		6.44E-2	4.92E-2
Sr-90	µCi/mL		1.07E+0	2.46E-1

Note: Blank spaces represent non-analyzed analytes. Concentrations are expected to be similar to the other AN-107 feed materials.

2.4 Experimental

The experimental work was conducted in three phases. The first phase was broad spectrum testing for sulfate removal including precipitation with direct Ba^{2+} addition; a Ca^{2+} addition (pre-strike) followed by Ba^{2+} addition; and metathesis of added $BaCO_3$ to $BaSO_4$. The second phase targeted the calcium pre-strike for initial carbonate removal followed by Ba^{2+} addition for $BaSO_4$ precipitation, and metathesis of $BaCO_3$ to $BaSO_4$. The third phase incorporated the Ca^{2+} pre-strike followed by Ba^{2+} addition on a scale-up test with 756-mL of Actual AN-107 tank waste[1]. The tests are delineated in Test Instructions BNFL-TI-29953-061 (Appendix A), -062 (Appendix B), and -071 (Appendix C), respectively. All testing was conducted at room temperature, nominally 21°C. In the following discussion, all indicated $Ca:CO_3$ and $Ba:SO_4$ ratios represent mole ratios.

[1] Test Specification Section 5.0, TSP-W375-99-00016, Revision 1, *Test Specification for Separating Sulfate from Pretreated AN-107, AZ-102, and AN-102 Solutions by Precipitation*, BNFL, Inc., Richland, Washington dated December 3, 1999 revised on December 9, 1999 and transmitted to PNNL according to CCN: 008798.

2.4.1 Survey of Calcium Carbonate and Barium Sulfate Precipitations, Phase 1

The experimental process flow sheet is provided in Figure 2.1. Five aliquots of nominally 30-g (25-mL) of Archive 1 AN-107 were sampled into 35-mL glass vials. Aliquots of 5M $Ca(NO_3)_2$ solution were added to four samples in mole ratios of $Ca:CO_3$ of 0.5:1, 1:1 (in duplicate), and 2:1; one sample had no added Ca^{2+}. Additional DI water was added as make-up volume such that all final sample volumes were identical at 32-mL, which diluted the matrix by a factor of 1.28. The solutions were mixed for 5 minutes and allowed to stand for 60 minutes. Solid and liquid phases were separated by centrifuging at 1500 rpm for 3 min. The supernatant was removed, filtered through a syringe filter, and analyzed for anions. The supernatants from the duplicate 1:1 $Ca:CO_3$ contact were combined in order to provide a larger working volume for subsequent Ba^{2+} contact studies. Aliquots of the supernatants were then further processed by adding 0.33 M $BaNO_3$ in various mole ratios of $Ba:SO_4$ (1:1, 1.4:1, 3:1, and 5:1). However, in the case of the 2:1 $Ca:CO_3$ contact, the supernatant volume was sufficient for only one follow-on Ba^{2+} contact; the 1.4:1 $Ba:SO_4$ contact was selected for study. After the Ba^{2+} contact, the samples were stirred for 5 minutes, then allowed to stand for 60 minutes. The samples were centrifuged and the supernatant was filtered through a syringe filter and submitted for anion analysis. Sub-samples from the 1:1 $Ca:CO_3$ contact and subsequent Ba^{2+} contacts were further analyzed for inorganic carbon and selected radionuclides.

2.4.2 Targeted 1:1 Calcium:Carbonate Pre-strike, Phase 2

The 1:1 $Ca:CO_3$ mole ratio was selected for further study based on the results from phase 1 testing. The process flow sheet provided in Figure 2.2 summarizes the general sample manipulation scheme. A 25-mL aliquot of 5M $Ca(NO_3)_2$ was mixed into 200-mL of Archive 2 AN-107 feed material over a 10 minute period with stirring. The slurry was stirred for an additional 15 minutes, then allowed to stand for 60 minutes. The slurry was resuspended and split into two fractions. The filter flux was measured by passing the slurries through two Nalgene filter apparatuses and recording the filtration times. The filter cake masses remaining in the upper filter units were recorded, as were the filtrate masses. The filtrates were combined and sampled for density, IC, TIC, GEA, and ICP measurements.

This study evaluated the use of $Ba(NO_2)_2$ in place of $Ba(NO_3)_2$; the advantages of the former reagent is it's much higher water solubility with a corresponding reduction in added aqueous volume. A 100-mL aliquot of the Ca^{2+} pre-strike filtrate was mixed with 2.7-mL 2M $Ba(NO_2)_2$ (targeted 1.4:1, actual 1.1:1 $Ba:SO_4$); a 10-mL aliquot of filtrate was contacted with 0.18-mL 2M $Ba(NO_2)_2$ (targeted 1:1, actual 0.8:1 $Ba:SO_4$). Each of these latter two solutions were mixed for 15 minutes and allowed to stand for an additional 60 minutes. The volume consumed by solids was estimated. The filter flux was determined for the 100-mL volume $BaSO_4$ slurry similarly to the $CaCO_3$ filter flux, except that the slurry was processed through 1 filter unit instead of 2 filter units. The $BaSO_4$ precipitate was washed three times with 10-mL 0.01M NaOH and percent solids determined. The 10-mL sample supernatant from the 0.8:1 $Ba:SO_4$ contact was filtered through a syringe filter. Both filtrates were analyzed by IC, ICP, GEA, and TIC methods.

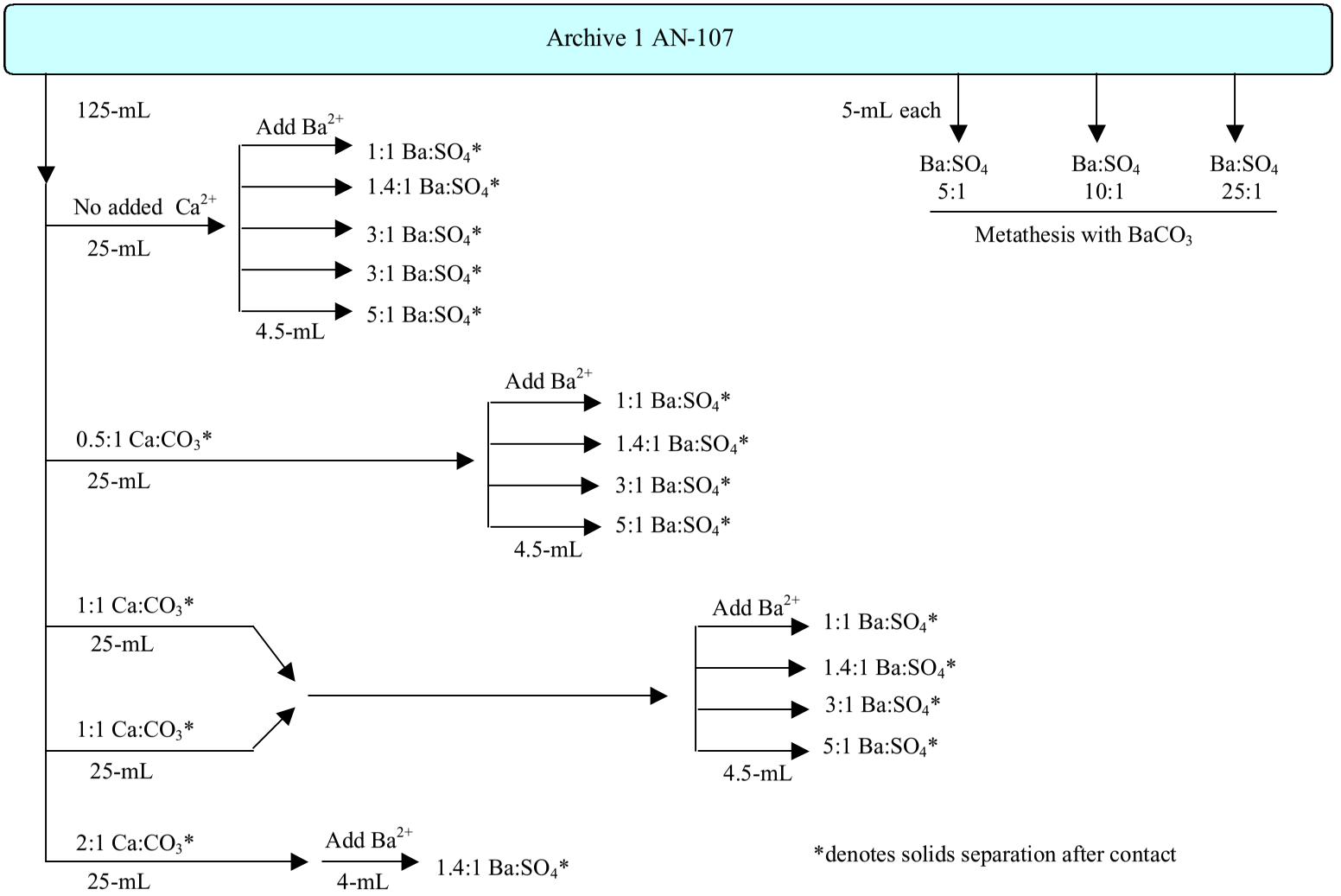


Figure 2.1. Experimental Outline of Phase 1 Testing

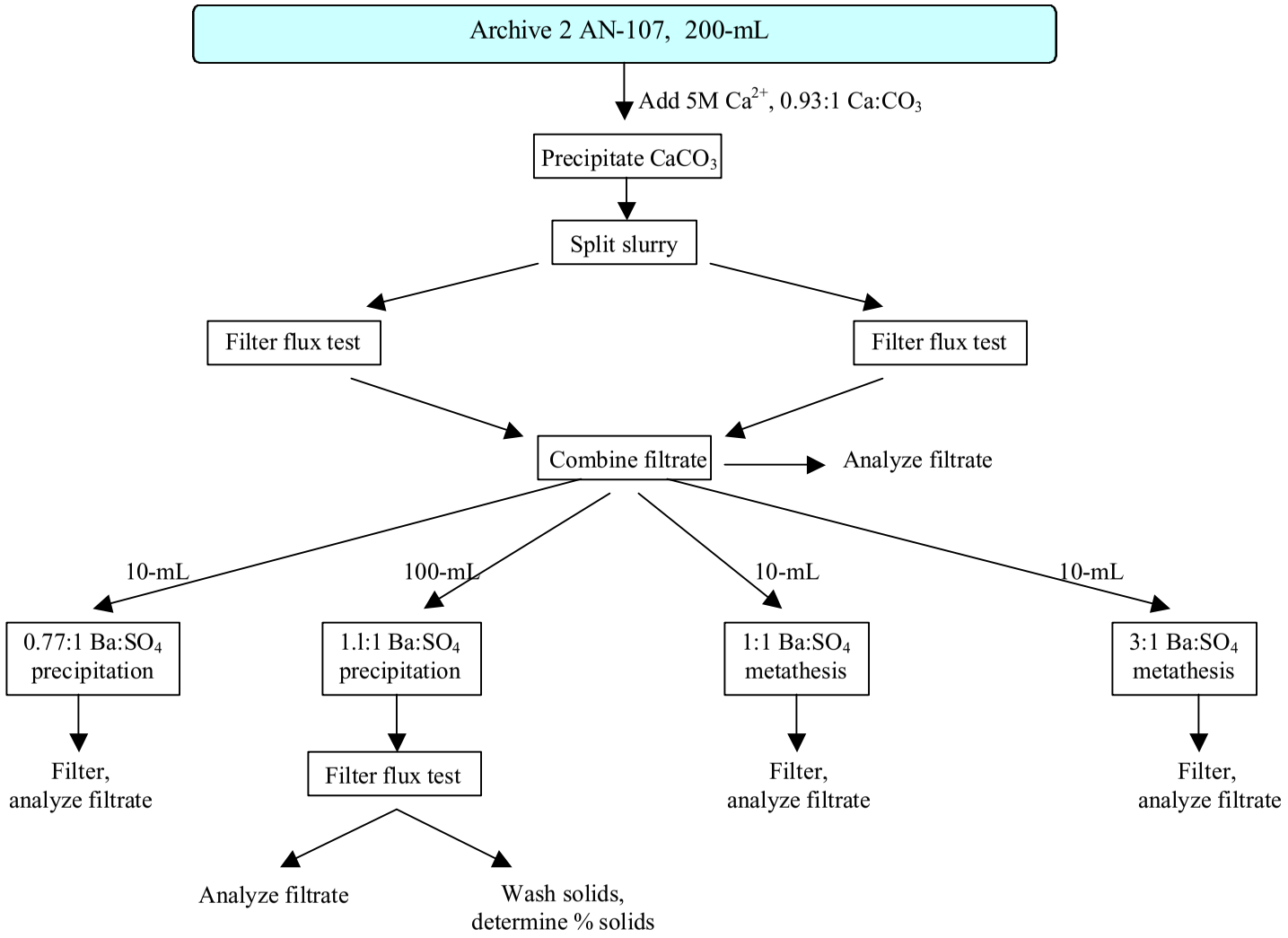


Figure 2.2. Experimental Outline of Phase 2 Testing

2.4.3 Scale-Up Precipitation Test, Phase 3

A scale-up test was conducted with 757mL of Actual AN-107 waste to more thoroughly evaluate the targeted 1.1:1 Ca:CO_3 prestrike and 1.3:1 Ba:SO_4 contact as well as generate enough low-sulfate AN-107 for vitrification tests. A process flow sheet, Figure 2.3, summarizes the general sample manipulation scheme as well as sampling points through the process. A 107-mL 5M Ca^{2+} solution, representing a 1.1:1 Ca:CO_3 mole ratio, was added slowly to a 757-mL aliquot of the Actual AN-107 sample while stirring over a 10-minute period. Stirring continued for 60 minutes after addition of the Ca to ensure complete mixing. The sample then was transferred to a graduated cylinder. The settling rate for the $CaCO_3$ solids was evaluated over a 2-hr period with the settle solids/liquid interface measurements taken every 10 minutes. After the settling test, the slurry was re-suspended by cylinder agitation. The slurry was sampled for viscosity testing, as well as density, insoluble solids, and particle size analysis. Half of the remaining slurry was then poured through a tarred filtration unit. Filtrate volume as a function of time was recorded, along with the total filtration time. Filtration was considered complete when the filtrate production was below 1 drop per 12 seconds. The filtration was repeated for the second half of the Ca^{2+} contacted Actual AN-107 slurry, using a second tared upper filtration reservoir. The filtrate was collected in the same lower reservoir used for the first half-filtrate collection, thus combining filtrates. Net wet precipitate masses were recorded along with the net filtrate mass. Samples of the wet precipitates (representative aliquots from each collection) and filtrate were taken for analysis.

The $CaCO_3$ precipitate was subjected to a series of washes; each wash was collected in a separate, clean, tarred, filtration receiver. Both $CaCO_3$ precipitate volumes (150-mL) were estimated based on the graduations of the upper reservoir and mixed with 300-mL 0.01M NaOH, a 2:1 volume ratio of 0.01M NaOH:precipitate. Mixing was done with a plastic stir rod until all chunks were dispersed. Vacuum was then applied and the total filtration time recorded. After a second 0.01M NaOH wash, samples of the wet precipitates were taken for analysis. Samples from the first and second filtrate washes were also taken for analyses. The $CaCO_3$ precipitate was then washed with a 2:1 volume ratio of 1M HNO_3:precipitate volume. A 200-mL volume of 1M HNO_3 was added slowly to each sample of $CaCO_3$ precipitate, with stirring. After reaction and thorough mixing, vacuum was applied and the total filtration time recorded. The wash was repeated after estimating the new precipitate volumes (about 50-mL each) at the same 2:1 ratio of 1M HNO_3:precipitate volume with 100-mL 1M HNO_3 added to each reservoir of $CaCO_3$. The $CaCO_3$ precipitates were then washed with 75-mL water each in a 3:1 volume ratio of water:precipitate. Samples of the washed precipitate were collected for analysis along with samples of the first and second 1M HNO_3 filtrate washes and the water filtrate wash. A fraction of the washed precipitate was dried to constant weight at 105°C to determine the percent of solids present in the washed precipitate.

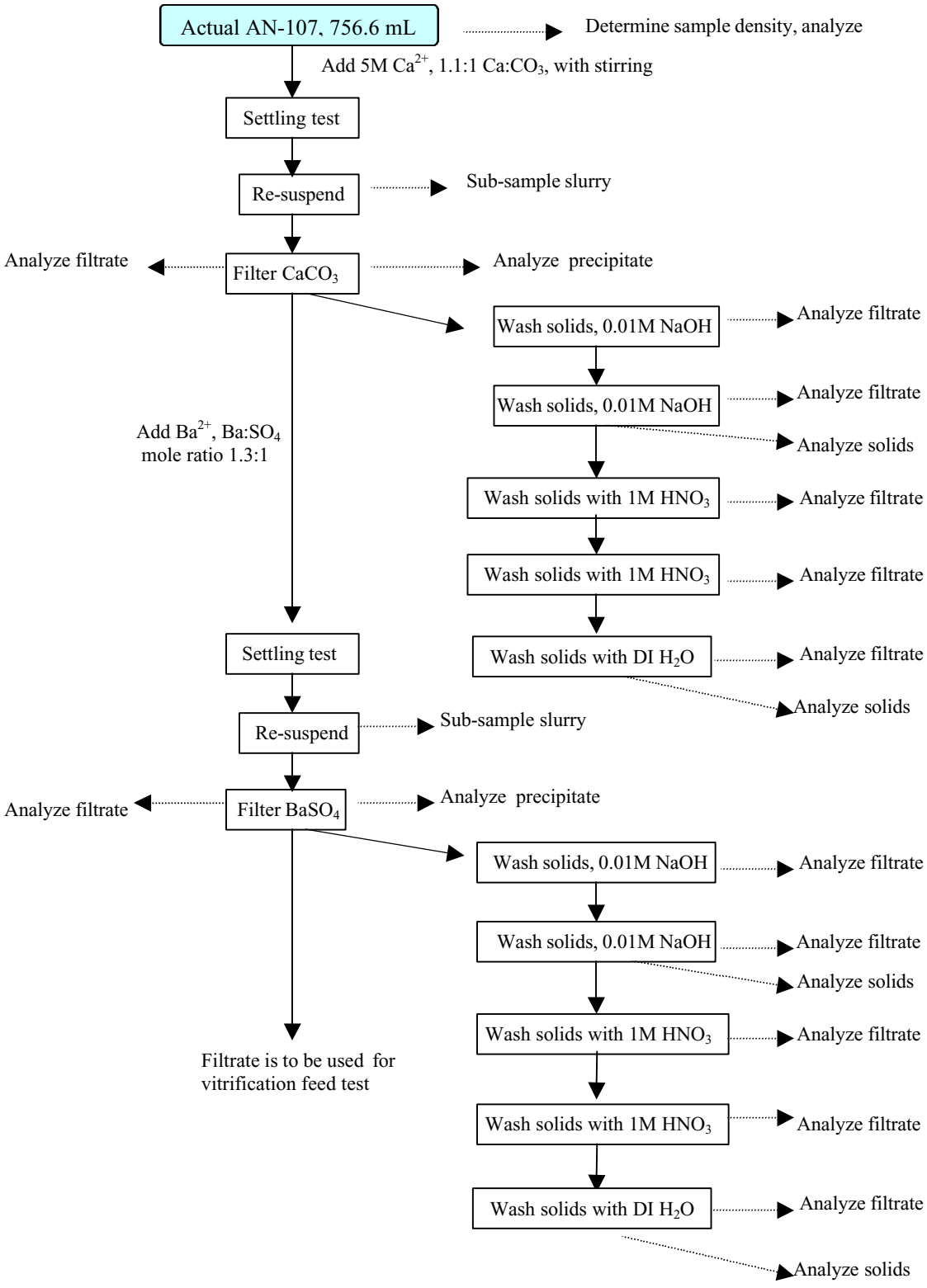


Figure 2.3. Experimental Outline of Phase 3 Testing

A 13.27-mL aliquot of 1.82M Ba^{2+} solution (as barium nitrite), representing a 1.3:1 $Ba:SO_4$ mole ratio, was added to the calcium pre-treated filtrate over a 6-min interval with stirring. The solution was stirred for an additional 60-min. to ensure complete mixing. The slurry was then poured into the graduated cylinder for a settling test. The settled solids volume was recorded every 10 minutes for 2 hours. The slurry was then allowed to continue settling overnight. The solid was re-suspended by mixing and aliquots taken for density, insoluble solids, viscosity, and particle size analyses. The remaining slurry was then poured into a filtration unit with tared upper and lower reservoirs. Total filtration time, gross wet precipitate mass, and filtrate mass were recorded. The wet precipitate and filtrate were sampled for analyses.

The barium precipitate was then subjected to a series of washes; each wash was collected in a clean, tared lower reservoir. The precipitate was washed with 40-mL 0.01M NaOH, representing a 2:1 volume ratio of wash:solids. The precipitate was stirred thoroughly in the wash reagent prior to applying vacuum. Total filtration time was recorded when vacuum was applied. After repeating the 0.01M NaOH wash, the solids and filtrates were sampled for analyses. Samples taken for particle size analysis were suspended in 0.01M NaOH. The solids were then washed with 40-mL 1M HNO_3, representing a 2:1 volume ratio of wash:solids. The solids were stirred thoroughly into the wash solution in the upper reservoir. After reaction was complete, vacuum was applied and the filtration time recorded. The solids were washed again with a 20-mL volume of 1M HNO_3, representing a 2:1 volume ratio of wash:solids, and the filtrate was collected in a clean reservoir. The solids were washed again with 30-mL deionized water, representing a 3:1 volume ratio of wash:solids. The filtrates and remaining solids were sampled for analyses and a fraction of the washed precipitate was dried to constant weight at 105°C to determine the percent solids present in the washed precipitate.

2.4.4 $BaCO_3$ to $BaSO_4$ Metathesis Study, Phase 1 and Phase 2

Six-gram aliquots of Archive 1 AN-107 were contacted with 0.2g, 0.4g and 1g aliquots of $BaCO_3$ (Figure 2.1). These aliquot sizes targeted 5:1, 10:1, and 25:1 ratios of $Ba:SO_4$, respectively. The materials were mixed in a shaker for nominally 21 hours, and then allowed to settle. The supernatants were filtered through 0.45-µm syringe filters and analyzed by IC.

Twelve-gram aliquots of the calcium-treated Archive 2 AN-107 were contacted with solid $BaCO_3$ in 1:1 and 3:1 $Ba:SO_4$ ratios and shaken for 90 hours (Figure 2.2). After settling, the supernatants were filtered, sampled, and analyzed by IC, ICP, GEA and TIC.

2.5 Analytical Methods

Specific analytical procedures used to support the solution and solid analyses are listed in Table 2.2. Solution anions were measured, after sample dilution, using a Dionex ion chromatograph similar to EPA Method 300.0. Large sample dilutions were generally necessary because of the high nitrate concentrations resulting in a sulfate detection limit of 2000 µg/mL. The $CaCO_3$ precipitate samples were leached with water that in turn was analyzed by ion chromatography; $BaSO_4$ solids were not analyzed by IC. The IC solids sample preparation utilizes a water leach. Barium sulfate solids are virtually insoluble in water, thus IC information on such a sample would be meaningless. Ion chromatography uncertainty was typically ±15% where the analyte concentrations were greater than ten times the detection limit. Total inorganic carbon, or carbonate, was measured directly on both liquids and solids by releasing CO_2 with hot 2M H_2SO_4 and sweeping the gas with an O_2 flow into CO_2 trapping solution. Carbon dioxide was measured coulometrically on a Coulometrics coulometer. Total organic carbon was measured

similarly except the entire sample was oxidized using sulfuric acid and potassium persulfate with silver nitrate catalyst for complete oxidation of organic species. Uncertainty was typically ±15% where the analyte concentration was greater than ten times the detection limit. Solutions and $BaSO_4$ solids were directly analyzed by gamma spectrometry to determine gamma-emitting radioisotopes. Calcium carbonate solids were first dissolved in HNO_3 prior to GEA analysis. Uncertainty was attributed primarily to counting statistics and ranged from ±2% for ^{137}Cs to ±25% for ^{241}Am. Filtrate and $CaCO_3$ sub-samples were acid digested; $BaSO_4$ samples were metathesized with ammonium carbonate and analyzed in 2 fractions: the sulfate-fraction and the Ba-fraction. The digested samples were processed through radiochemical separations to isolate ^{79}Se and ^{90}Sr followed by liquid scintillation and beta proportional counting, respectively. Inductively-coupled plasma mass spectrometry (ICP-MS) was used to measure ^{99}Tc, ^{237}Np, and ^{239}Pu after acid digestion and dilution; uncertainty was typically ±10%. Most cations were measured using inductively-coupled plasma atomic emission spectrometry with an uncertainty of nominally ±15% where analyte concentration was greater than ten times the detection limit. Uranium kinetic phosphorimetry was used to measure U concentration following matrix adjustment with typical uncertainties of ±4%. Ammonia was measured with an ion-selective electrode on aqueous samples directly or on water leachates of solids. Total alpha was measured by directly plating the acid-digested sample onto a planchet and counting using gas-flow proportional counting. Because of the high beta activity causing cross-talk into the alpha channel, alpha detection limits were generally high.

Table 2.2. Analytical Procedures

Analyte	**Procedure**
Anions	PNL-ALO-212, Rev. 1
Water leach	PNL-ALO-103, Rev. 1
TIC	PNL-ALO-381, Rev. 1
TOC	PNL-ALO-381, Rev. 1
NH_3	PNL-ALO-226
Acid digestion, solutions	PNL-ALO-128
Acid digestion, $CaCO_3$	PNL-ALO-128
Metathesis, $BaSO_4$	cognizant scientist direction
Sr-90	PNL-ALO-476 Rev. 1, -408, Rev. 1
GEA	PNL-ALO-450 Rev. 1
Am-241	PNNL-ALO-417 Rev. 2, -496, -422
Pu-239/240 and Pu-238	PNNL-ALO-417 Rev. 2, -496, -422
ICP-AES	PNL-ALO-211
ICP-MS	PNL-ALO-280 Rev. 1
U	PNNL-ALO-4014
Se-79	PNL-ALO-440, Rev. 7, -474
Total alpha	PNL-ALO-4001, -408, Rev. 1

Particle size, XRD, viscosity, TCLP, Hg, and Am (by AEA) were cancelled subsequent to sampling by customer request. The ICP-MS analyses of the $BaSO_4$ solids were also cancelled.

3.0 Results and Discussion

3.1 Survey of Calcium Carbonate and Barium Sulfate Precipitations, Phase 1

In all cases, the addition of calcium solution produced copious amounts of $CaCO_3$ precipitate. The Archive 1 AN-107/$CaCO_3$ mixtures had to be vigorously shaken by hand as the stir bar was largely ineffectual in mixing the slurried solids in the 35-mL vial. The estimated centrifuged solids volumes compared to the mole ratio of added $Ca:CO_3$ are summarized in Table 3.1. The sulfate concentration, where no Ca^{2+} was added, measured 0.034M and is consistent with simple dilution of the starting material (dilution factor = 1.28). Sulfate concentration dropped slightly with increasing Ca^{2+} addition indicating a small amount of $CaSO_4$ precipitated with the $CaCO_3$. The percent sulfate removal is shown in Table 3.1. The 1:1 $Ca:CO_3$ pre-strike supernatant resulted in nearly 68% carbonate removal from solution.

Table 3.1. Sulfate and Carbonate Removal from Calcium Contact, Phase 1 Testing

$Ca:CO_3$ mole ratio	% centrifuged solids	% SO_4 removed	% CO_3 removed
0.5:1	30	0	NA
1:1	55	6	68
2:1	70	12	NA

NA: not analyzed

The addition of 0.33 M Ba^{2+} resulted in a well-defined, heavy, precipitate that compacted more efficiently than $CaCO_3$. The dilution factor after Ba^{2+} addition varied slightly for each sample as water was not added as a make-up volume. Sulfate removal was calculated according to the equation $1-([SO_4]_f/[SO_4]_2^{'})$ where $[SO_4]_f$ is the final measured sulfate concentration and the $[SO_4]_2^{'}$ is the initial sulfate concentration corrected for the dilution factor. The results of the Ba^{2+} contact are summarized in Table 3.2. Where the Ca^{2+} pre-strike was less than 1:1 $Ca:CO_3$, the sulfate removed by Ba^{2+} addition was inconsequential, i.e., the measured sulfate concentration was virtually equal to the sulfate concentration corrected for dilution. For the case of a 1:1 $Ca:CO_3$ pre-strike contact, the Ba^{2+} contact was effective in removing sulfate. The four samples from this Ca^{2+} pre-strike underwent further analysis by ICP, GEA, TIC, and for ^{90}Sr. The 1.4:1 $Ba:SO_4$ contact, from this calcium pre-strike, resulted in 69% sulfate removal for a final 0.008 M SO_4 concentration. Higher $Ba:SO_4$ ratios resulted in diminishing returns of sulfate removal while increasing CO_3^{2-} removal. Dissolved Ba^{2+} remained in solution ranging from 51 to 164 µg/mL. The chromium concentration also dropped with Ba^{2+} addition, probably co-precipitating as $BaCrO_4$. For a 1:1 $Ba:SO_4$ contact, 81% of the chromium was removed. The ^{90}Sr and ^{154}Eu concentrations also dropped with increasing $BaSO_4$ precipitation; ^{137}Cs and ^{60}Co concentrations remained unaffected. Analytical details are provided in Appendix A. Based on these sulfate removal results, the 1:1 $Ca:CO_3$ pre-strike followed by 1:1 and 1.5:1 Ba^{2+} strike were selected for further study.

Table 3.2. Sulfate and Carbonate Removal After Ba^{2+} Contacts Following Ca^{2+} Pre-strike, Phase 1 Testing

$Ca:CO_3$ Mole Ratio	Targeted $Ba:SO_4$ Mole Ratio	Actual $Ba:SO_4$ Mole Ratio	% SO_4 Removed	% CO_3 Removed
0:1	1:1	0.92:1	6	NA
	1.4:1	1.3:1	3	NA
	3:1	2.8:1	9	NA
	3:1	2.8:1	1	NA
	5:1	4.6:1	5	NA
0.5:1	1:1	0.99:1	0	NA
	1.4:1	1.3:1	-3	NA
	3:1	3.0:1	-9	NA
	5:1	5.0:1	-1	NA
1:1	1:1	1.0:1	54	12
	1.4:1	1.4:1	69	16
	3:1	3.2:1	69	56
	5:1	5.3:1	>70	86
2:1	1.4:1	1.5:1	>76	NA

NA: not analyzed

3.2 Targeted 1:1 Calcium:Carbonate Precipitation Pre-Strike, Phase 2

Carbonate concentration of Archive 2 AN-107, determined by titration and combustion, resulted in 0.81M and 0.70M carbonate, respectively. Based on an average of these two results, 0.755M CO_3, the calcium:carbonate mole ratio was calculated to be 0.93:1, slightly below the targeted 1:1 mole ratio. As observed before, copious amounts of precipitate formed upon Ca^{2+} addition. The filtrate collection rate, or filter flux, of half the $CaCO_3$ slurry was determined in duplicate because the precipitate volume was relatively large compared to the holding capacity of one filter unit upper reservoir. The fluxes measured 12 g/min and 16 g/min through the 20-cm^2, 0.45-μm filter. The 169-mL filtrate volume represents 74% of the combined Archive 2 AN-107 and added calcium solution volumes (227-mL). The $CaCO_3$ precipitation resulted in a 69% carbonate removal, consistent with the results from phase 1 study. Virtually no $CaSO_4$ co-precipitated with the $CaCO_3$. These results are summarized in Table 3.3. Cobalt and cesium concentrations remained unaffected. Predictably, ^{90}Sr precipitated with the $CaCO_3$. Europium and americium also were nominally 60% co-precipitated. Analytical details are provided in Appendix B.

Table 3.3. Sulfate and Carbonate Removal of Targeted Calcium Pre-strike, Phase 2 Testing

	Volume	SO_4	CO_3
Ca Pre-Strike, 0.93:1 Ca:CO_3			
Archive 2 AN-107	200 mL	0.053 M	0.755 M
Added 5 M $Ca(NO_3)_2$-$4H_2O$	27.9 mL		
Dilution factor	1.14		
Filtrate	169 mL	0.048 M	0.204 M
% removed (dilution corrected)		-3	69
2 M $Ba(NO_2)_2$-H_2O contact, 1.1:1 Ba:SO_4			
Archive 2 AN-107 prestrike	100 mL	0.048 M	0.204 M
Added 2 M Ba^{2+}	2.68 mL		
Dilution factor	1.03		
Filtrate	98.7 mL	<0.02 M	0.248 M
% removed (dilution corrected)		>57	-25
2 M $Ba(NO_2)_2$-H_2O contact, 0.77:1 Ba:SO_4			
Archive 2 AN-107 prestrike	9.96 mL	0.048 M	0.204 M
Added 2 M Ba^{2+}	0.18 mL		
Dilution factor	1.02		
Filtrate	not measured	<0.02 M	0.222 M
% removed (dilution corrected)		>57	-11

The two ratios of Ba:SO_4 tested, 0.77:1 Ba:SO_4 and 1.1:1 Ba:SO_4, missed the target ratios of 1:1 and 1.5:1 because the sulfate concentration in the Archive 2 AN-107 was 30% higher than expected based on sample processing history. A filter flux of 21 g/min in through a 20-cm^2, 0.45-µm filter was obtained for the Archive 2 AN-107/$BaSO_4$ slurry. Sulfate concentration dropped to <0.02M in both filtrates corresponding to >57% sulfate removal. A more definitive sulfate removal could not be calculated because the absolute sulfate concentrations in both filtrates (1.1:1 and 0.77:1 Ba:SO_4 contacts) were less than the instrument detection limit of 0.02M sulfate. Carbonate measured in the Ba^{2+} contacted solutions measured higher than the CO_3^{2-} pre-strike AN-107 feed. There is no good explanation for this other than the samples may have absorbed CO_2 from the air while waiting analysis. Barium concentrations in the filtrates were 101 and 35 µg/mL. Chromium removal ranged from 72% to 80%, consistent with the phase 1 study. The washed $BaSO_4$ precipitate contained 35 weight percent solids. Results are summarized in Table 3.3, and analytical details are provided in Appendix B. Based on the successful sulfate removal and filterability of the $CaCO_3$ and $BaSO_4$, a scale-up test was defined to further evaluate processing characteristics.

3.3 Scale-up Precipitation Test, Phase 3

Upon addition of the 5M Ca^{2+} solution, solids immediately began to form and continued to form throughout the Ca^{2+} addition. Figure 3.1 shows the $CaCO_3$ settling rate as a function of time. After the 120-minute settling test, nearly 97% of the sample volume still contained slurried solids. The sub-samples of the $CaCO_3$ slurry taken for viscosity, particle size, and insoluble solids measurements were no longer fluid after setting aside for 2 days, thus obviating further analyses of the $CaCO_3$ slurry. After the 2-hr settling test, the $CaCO_3$ slurry was re-mixed and split in half for filtering through two filters. The split was necessary because the copious precipitate volume would nearly fill one Millipore filtration unit upper reservoir (500-mL). An approximate

filtration rate was determined based on the slurry volume change in the upper reservoir. The change in filtration rate as a function of time is presented in Figure 3.2. After about 150 mL filtrate was collected, the flow rate stabilized at nominally 8 mL/min. Each split produced about 280-mL filtrate. In both cases, the wet precipitate volume after filtration was nominally 150 mL (as measured from the graduations in the upper filter chamber). The combined net mass of the wet $CaCO_3$ precipitate mass was 361g. The individual filtrate masses were not measured; the filtrates were combined providing a total volume of 561 mL. From a starting volume of 846 mL, this corresponds to an aqueous volume recovery of 67%. The $CaCO_3$ co-precipitated 5.6% of the sulfate while removing 67.7% of the carbonate. These results are consistent with results from the previous 2 study phases and are summarized in Table 3.4. Each $CaCO_3$ precipitate was washed twice with 0.01M NaOH followed by two 1M HNO_3 washes and one water wash. The 1M HNO_3 wash resulted in the production of CO_2 as carbonate was decomposed. Predictably, each HNO_3 wash resulted in significant precipitate mass losses, 55% and 35%, respectively. A summary of the precipitate and filtrate masses, volumes, filtration times, etc., are provided in Appendix C. The $CaCO_3$ precipitation carried other species besides the $CaCO_3$. Co-precipitated with the $CaCO_3$ were Al, Fe, P [(probably as $Ca_3(PO_4)_2$], and Sr. Europium, Am, Pu, and Np also followed the $CaCO_3$. After the 1M HNO_3 washes, the Al, P, Eu, and Am remained disproportionately with the $CaCO_3$ precipitate, i.e., partial dissolution of the $CaCO_3$ did not cause partial dissolution of the Al, P, Eu, and Am precipitates. A detailed analytical comparison of the Actual AN-107 starting material and calcium pre-strike filtrate is provided in Tables 3.5 and 3.6. Wash filtrate and $CaCO_3$ analyses are provided in Table 3.7.

Barium precipitates formed immediately upon addition of Ba^{2+} to the Ca^{2+} pre-treated Actual AN-107 filtrate. After a 120-minute settling test, the slurried precipitate volume occupied 17% of the total solution volume. The $BaSO_4$ settling curve is shown in Figure 3.1. Filtration of the $BaSO_4$ slurry required 82 minutes with 94% of the $BaSO_4$ slurry volume recovered in the filtrate. The initial filtration rate was rapid (data not taken—observation only) settling down to nominally 4-mL/min after about 50% of the filtrate volume was collected (Figure 3.2). Consistent with previous work, 69.7% of the sulfate was removed and only 6.8% of the carbonate was removed during this $BaSO_4$ precipitation. The overall total sulfate removal for the combined Ca^{2+} and Ba^{2+} contact was 72% with a combined filtrate recovery of 63%. The sulfate and carbonate removal and filtrate recoveries are summarized in Table 3.4. Analytical details of the $BaSO_4$ filtrate are provided in Tables 3.5 and 3.6. The $BaSO_4$ co-precipitated Cr, while other cation and anion concentrations (except Ba and sulfate) remained virtually unchanged. Under the tested processing conditions, measurable Ba remained in solution at 110 μg/mL. The two 40-mL 0.01M NaOH washes were rapid, taking no more than 1 minute, and did not remove precipitate mass. The initial 40-mL 1M HNO_3 wash resulted in about a 50% reduction of precipitate mass as well as gas evolution resulting from the decomposition of carbonate. The second 10-mL 1M HNO_3 wash actually resulted in a mass increase from 8.7g to 13.2g. The filtrate wash and $BaSO_4$ compositions are provided in Table 3.8.

The mass and activity balances for selected cations and radionuclides and fractional recoveries are provided in Table 3.9. The sum of recovered fractions is nominally 1, within experimental error, except for ^{90}Sr, where it appears an analytical error could have occurred. The washed $BaSO_4$ material appears to be contaminated principally with Cr.

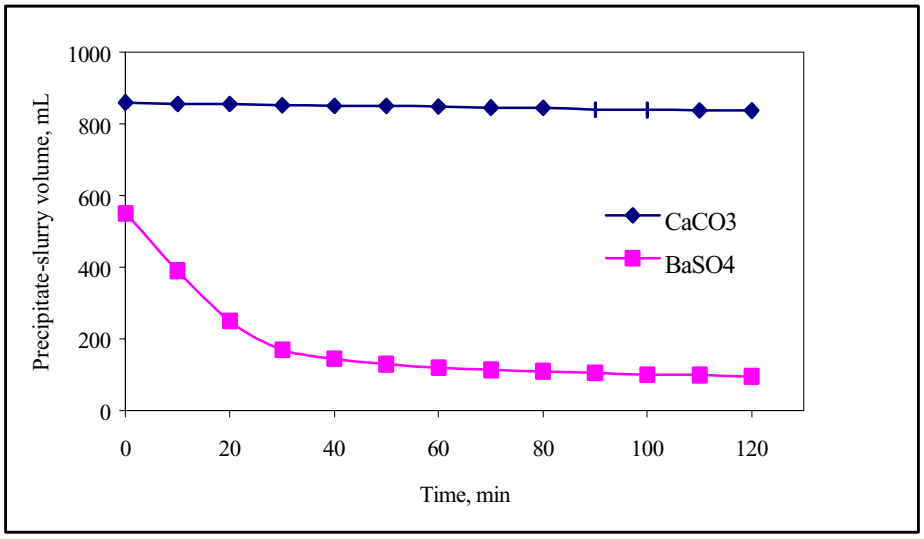


Figure 3.1. Settling Rates for Actual AN-107 $CaCO_3$ and $BaSO_4$ Slurries

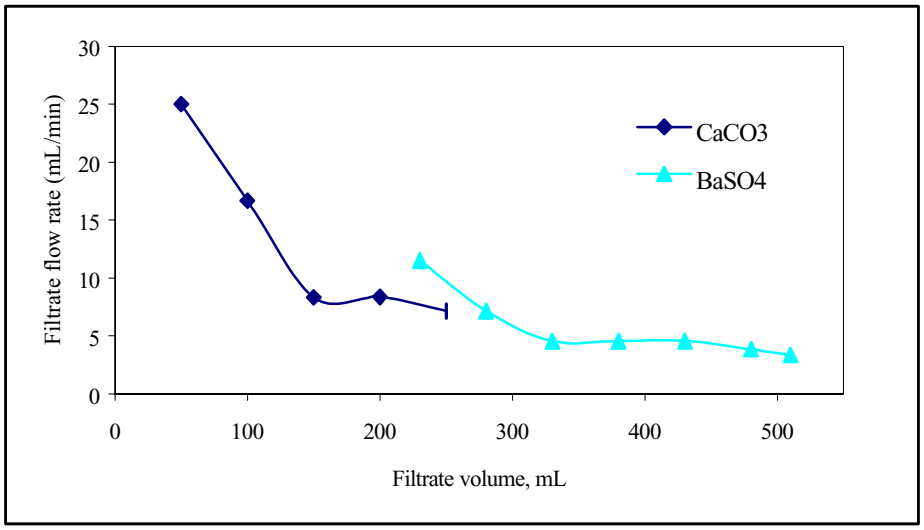


Figure 3.2. Actual AN-107 Filtration Rates as a Function of Filtrate Volume for the $CaCO_3$ and $BaSO_4$ Slurries

Table 3.4. Sulfate and Carbonate Removal from Actual AN-107 Scale-Up Test, Phase 3

	Solution Volume	SO_4 Removed	CO_3 Removed	Recovered Filtrate Volume
$CaCO_3$ Precipitation				
Actual AN-107	735 mL			
5M Ca^{2+}	107 mL			
Dilution factor	1.15			
Filtrate	561 mL	5.6%	67.7%	66.7%
$BaSO_4$ Precipitation				
Actual AN-107 filtrate from Ca prestrike	531 mL			
1.82M Ba^{2+}	13.7 mL			
Dilution factor	1.03			
Filtrate	513.mL	69.7%	6.8%	94.2%
Grand Total				
Actual AN-107	735 mL			
5M Ca^{2+} + 1.82M Ba^{2+}	121 mL			
Dilution factor	1.16			
Final filtrate	513 mL	72.0%	70.5%	62.8%

Table 3.5. Actual AN-107 Feed and Filtrate Compositions, Required Analytes

	Actual AN-107 Feed	Filtrate after $CaCO_3$ precipitation	Filtrate after $BaSO_4$ precipitation	BNFL-directed MRQ
Analyte/Units	µCi/mL	µCi/mL	µCi/mL	µCi/mL
^{137}Cs	8.22E-2	7.36E-2	7.33E-2	9.0E+0
^{154}Eu	3.26E-2	6.01E-3	3.10E-3	2.0E-3
^{155}Eu	2.31E-2	4.41E-3	2.17E-3	9.0E-2
^{241}Am	1.19E-2	2.08E-3	1.35E-3	7.75E-4
^{239}Pu	1.49E-3	6.82E-4	3.88E-4	7.75E-4
^{99}Tc	5.90E-2	5.51E-2	4.90E-2	1.50E-3
^{237}Np	4.17E-5	2.31E-5	1.22E-5	7.75E-4
Total Alpha	<3E-2	<2E-3	<2E-3	7.75E-4
^{79}Se	<1.6E-5	1.35E-5	<2E-6	1.E-04
^{90}Sr	2.46E-01	7.85E-03	2.43E-03	3.0E-1
Analyte/Units	µCi/mL	µCi/mL	µCi/mL	µCi/mL
Ag	<0.6	<0.4	<0.4	1
Al	2,340	850	768	75
As	<7	<5	<4	1
Ba	<0.3	<0.2	107	10
Ca	172	941	886	150
Cd	27	25	22	0.1
Co	2.1	[1.8]	[1.7]	30
Cr	43	36	6.3	1
Cu	13	12	11	17
Fe	8.6	[3.3]	2.8	150
K	711	646	755	200
La	<2	<0.9	<0.9	35
Mg	<3	<2	<2	150
Mn	[1.4]	<0.9	<0.9	150
Mo	16	15	13	90
Na	111,000	104,000	94,700	75
Ni	210	196	176	30
Pb	58	25	22	1
Se	<7	<5	<5	0.1
Si	[32]	<9	<9	170
U	<50	<35	<35	600
$U^{(1)}$	8.3	6.1	2.9	600
Zn	7.6	[4.7]	4.4	16.5
TOC	16,100	13,300	13,100	1,500
TIC	8,160	2,290	2,070	150
Cl	< 500	<500	< 500	25
$F^{(2)}$	3,500	3,000	3,000	150
NO_3	112,000	198,000	173,000	3,000
PO_4	1400	<1000	<1000	2,500
SO_4	4,020	3,300	970	2,300
NH_3	8.9	9.7	6.5	150

1) U determined by kinetic phosphorescence
2) F results are suspect due to peak distortion and retention time shift.
Radioisotope reference date is 11/1/99
Bracketed results represent values with errors exceeding 15%, 2-σ.

Table 3.6. Actual AN-107 Feed and Filtrate Compositions, Opportunistic Analytes

	Actual AN-107 Feed	Filtrate after $CaCO_3$ precipitation	Filtrate after $BaSO_4$ precipitation
Analyte/Units	µCi/mL	µCi/mL	µCi/mL
^{60}Co	4.95E-2	4.12E-2	3.96E-2
^{125}Sb	6.00E-4	<3.6E-4	<8.4E-5
$^{126}Sn/^{126}Sb$	3.80E-4	<2.4E-4	<3.6E-5
Analyte/Units	µg/mL	µg/mL	µg/mL
B	19	[1.8]	[1.2]
Nd	<3	<2	<2
P	302	48	43
Sb	<13	<9	<9
Sn	<38	<25	<25
Sr	130	[0.8]	7.0
W	[76]	[65]	[60]
Y	<2	<0.8	<0.8
Zr	[2.9]	<0.8	<0.8
NO_2	28,800	25,900	29,500
Oxalate	1400	<1000	<1000

Radioisotope reference date is 11/1/99

Bracketed results represent values with errors exceeding 15%, 2-σ.

Table 3.7. Composition of $CaCO_3$ Precipitate Wash Filtrates and $CaCO_3$ Precipitate

	$CaCO_3$ precipitate wash filtrates						**$CaCO_3$ precipitate**			
	0.01M NaOH		1M HNO_3		DI H_2O	BNFL	before	after NaOH	after DI	BNFL
Analyte	Wash 1	Wash 2	Wash 1	Wash 2	Wash 1	MRQ	wash	wash	H_2O wash	MRQ
Units:	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/g	µCi/g	µCi/g	µCi/g
^{137}Cs	2.26E-2	5.48E-3	2.63E-3	8.16E-4	2.21E-4	9.0E+0	1.96E-1	2.60E-2	9.77E-4	6.0E-2
^{60}Co	1.20E-2	2.58E-3	6.32E-4	8.41E-4	2.80E-4		1.39E-1	6.63E-2	1.31E-1	1.20E-2
^{154}Eu	6.92E-4	<3E-5	1.26E-4	1.84E-3	4.53E-4	2.0E-3	2.32E-1	3.42E-1	7.81E-1	6.0E-2
^{155}Eu	4.79E-4	3.94E-5	<7E-5	1.34E-3	3.11E-4	9.0E-2	1.70E-1	2.51E-1	5.54E-1	6.0E-2
^{241}Am	2.57E-4	3.09E-5	6.37E-5	5.93E-4	1.38E-4	7.75E-4	8.85E-2	1.28E-1	2.90E-1	1.20E-3
^{239}Pu	1.12E-4	2.05E-5	<1E-5	4.16E-5	1.99E-5	7.75E-4	8.50E-3	9.55E-3	2.21E-2	6 ug/g
^{99}Tc	1.45E-2	3.13E-3	8.40E-4	7.06E-4	2.30E-4	1.5E-3	1.43E-1	2.92E-2	4.85E-2	6 ug/g
^{237}Np	3.64E-6	<3E-7	<3E-7	4.37E-7	<2E-7	7.75E-4	1.64E-4	1.97E-4	4.39E-4	1.8ug/g
Total Alpha	<7E-4	<3E-4	<2E-2	<2E-2	<4E-3	7.75E-4	5.15E-2	<4E-1	3.50E-1	1.0E-3
^{90}Sr	3.75E-3	2.81E-3	6.78E-1	3.34E-1	3.39E-2	3.0E-1	8.93E+0	1.42E+1	8.94E+0	7.0E+1
Units:	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/g	µg/g	µg/g	µg/g
Ag	<0.4	<0.1	<0.3	<0.3	<0.3	1	<13	<7	<9	900
Al	[9.5]	3.14	19.8	9.7	[2.1]	75	12,100	21,700	53,100	330
As	<4	<1	<3	<3	<3	1	<130	<74	<100	3
B	[5.]	4.1	[1.9]	20.2	8.64		392	275	[140]	
Ba	<.2	<0.06	[0.74]	[0.45]	[0.14]	10	[17]	[25]	[38]	600
Ca	331	132	10,900	17,100	5,330	150	236,000	360,000	311,500	180
Cd	6.6	1.07	[0.36]	[0.95]	[0.34]	0.1	[75]	[24]	[30]	11
Co	<0.8	<0.1	<1	<1	<1	30	<26	<15	<20	3
Cr	10.2	2.7	[0.82]	[0.36]	[0.42]	1	110	[32]	100	120
Cu	3	0.67	<0.3	[0.4]	<0.3	17	[31]	[14]	[13]	18
Fe	<0.4	<.1	[0.69]	[1.7]	[0.97]	150	[51]	79	151	140
K	140	[36]	<25	<20	<20	200	<1100	<590	<800	1500
La	<0.8	<0.1	[3.9]	<0.5	[1.8]	35	[79]	<15	[130]	60
Mg	<2	<0.3	[11]	80.7	29	150	652	1,050	1,120	540
Mn	<0.8	<0.1	<0.6	<0.5	<0.5	150	<26	[16]	[29]	300
Mo	[4.1]	[0.94]	<0.6	<0.5	<0.5	90	[36]	<15	<20	6
Na	25,500	7,150	4,560	1,940	537	75	267,000	50,500	2,250	150
Nd	<2	<0.3	<1	<1	<1		<53	<29	[88]	77
Ni	54	12	3.9	[3.]	[1.2]	30	480	[82]	[72]	160
P	[11]	2.6	<1	<1	<1		2,040	2,940	7,210	
Pb	[2.3]	[0.37]	[6.1]	21	[8.]	1	[260]	423	549	600
Sb	<8	<1	<6	<5	<5		<260	<150	<200	12
Se	<4	<0.6	<3	<3	<3	0.1	<130	<74	<100	300
Si	[37]	32	<6	<5	<5	170	[1200]	[360]	[225]	3000
Sr	[0.54]	0.50	87	76	24		1,090	1,690	1,080	
U	<33	<5	<25	<20	<20	600	<1060	<590	<800	600
U(1)	0.87	0.16	0.07	0.67	0.17	600	31.6	38.5		600
W	<33	<5	<25	<20	<20		<1060	<590	<800	
Zn	[1.0]	[0.34]	<0.6	[1.8]	0.85	16.5	31	[35]	[45]	6
Zr	<0.8	<0.1	<0.6	<0.5	<0.5		<26	[24]	[62]	600
TOC(2)	4,050	980	390	260	91	1500	29,500	13,000	27,800	60
TIC(2)	430	150	26	37	13	150	43,200	73,700	77,400	30
F(3)	900	230	<250	<250	<125	150	5,600	1,100	480	7500
Cl	<250	<50	<250	<250	<125	25	<1700	<680	<240	230
NO_2	7,100	1,700	<500	<500	<250		40,100	4,050	<480	
NO_3	58,500	12,300	50,700	62,300	16,500	3000	334,000	28,250	23,600	450
PO_4	<500	<100	<500	<500	<250	2500	<3300	<1400	<480	600
SO_4	1,100	240	<500	760	<250	2300	8,000	4,050	1,400	1200
oxalate	<500	<100	<500	<500	<250		<3300	<1400	<480	
NH_3	2.0	0.5	0.2	<0.2	<0.2	150	3.8	<5	<0.4	60

1) Analyzed by kinetic phosphorescence. Bracketed results represent values with errors exceeding 15%, 2-σ.
2) Reported as micrograms C per mL or per g.
3) F results are suspect due to peak distortion and retention time shift.

Table 3.8. Composition of $BaSO_4$ Precipitate Wash Filtrates and $BaSO_4$ Precipitate

	$BaSO_4$ precipitate wash filtrates						**$BaSO_4$ precipitate**			
	0.01M NaOH		1M HNO_3		DI H_2O	BNFL	before	after NaOH	after DI	BNFL
Analyte	Wash 1	Wash 2	Wash 1	Wash 2	Wash 1	MRQ	wash	wash	H_2O wash	MRQ
Units:	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/mL	µCi/g	µCi/g	µCi/g	µCi/g
^{137}Cs	2.09E-2	5.24E-3	1.38E-3	3.58E-4	1.91E-4	9.0E+0	1.03E-1	1.06E-2	1.64E-3	6.0E-2
^{60}Co	9.89E-3	2.41E-3	4.87E-3	5.05E-3	1.92E-3		1.03E-1	1.02E-1	5.24E-2	1.20E-2
^{154}Eu	4.45E-4	6.04E-5	1.82E-2	1.15E-2	2.85E-3	2.0E-3	1.42E-1	2.45E-1	1.93E-2	6.0E-2
^{155}Eu	2.97E-4	3.65E-5	1.25E-2	7.86E-3	2.72E-3	9.0E-2	9.80E-2	1.63E-1	1.09E-2	6.0E-2
^{241}Am	2.02E-4	4.02E-5	4.19E-3	3.40E-3	1.30E-3	7.75E-4	3.94E-2	5.37E-2	4.38E-3	1.20E-3
^{239}Pu	7.44E-5	1.12E-5	1.72E-3	1.30E-3	3.97E-4	7.75E-4	NA	NA	NA	6 ug/g
^{99}Tc	1.31E-2	3.23E-3	2.40E-3	1.79E-3	6.05E-4	1.5E-3	NA	NA	NA	6 ug/g
^{237}Np	2.26E-6	3.53E-7	7.03E-5	2.68E-5	9.45E-6	7.75E-4	NA	NA	NA	1.8ug/g
Total Alpha	NA	NA	NA	NA	NA	7.75E-4	NA	NA	NA	1.0E-3
^{90}Sr	8.56E-4	4.84E-4	2.72E-2	2.75E-2	8.96E-3	3.0E-1	NA	NA	NA	7.0E+1
Units:	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/g	µg/g	µg/g	µg/g
Ag	<0.3	<0.1	<0.5	<1	<0.3	1	<32	<110	<110	900
Al	213	55	21	[5.8]	[1.3]	75	1560	137	35	330
As	<3	<0.5	<5	<13	<3	1	<170	<590	<590	3
B	[1.0]	<0.1	<1	<3	<0.6		580	89	35	
Ba	42	16	22,100	7,900	4,720	10	263,000	554,000	586,000	600
Ca	308	103	3,080	959	263	150	23,800	35,400	357	180
Cd	5.8	1.4	3.7	[1.4]	[0.49]	0.1	55	22	<110	11
Co	<0.5	<0.1	<1	<3	<0.6	30	<53	<190	<190	3
Cr	2.1	0.74	55	56	26	1	1250	2430	2630	120
Cu	2.7	0.70	[0.63]	<1.3	<0.3	17	72	36	34	18
Fe	[0.95]	[0.24]	[1.0]	[1.5]	[0.57]	150	452	125	222	140
K	212	50	<40	<100	<25	200	977	<14,800	<14,800	1,500
La	<0.5	<0.1	[2.2]	<2.5	<0.6	35	<53	<190	<190	60
Mg	<1	<0.2	<2	<5	<1	150	1110	903	745	540
Mn	<0.5	<0.1	<1	<3	<0.6	150	7	4	3	300
Mo	[3.9]	1.1	[1.1]	<3	<0.6	90	31	16	13	6
Na	27,700	7,530	2,630	592	162	75	154,000	20,700	1,300	150
Nd	<1	<0.2	[2.4]	<5	<1		<210	<740	<740	77
Ni	47	12	9.9	[4.8]	[2.1]	30	331	50	12	160
P	11	2.7	[9.3]	[6.1]	[2.1]		136	119	107	
Pb	[6.]	[1.4]	<2	<5	<1	1	<130	<440	<440	600
Sb	<5	<1	<10	<25	<6		<110	<370	<370	12
Se	<3	<0.5	<5	<13	<3	0.1	<110	<370	<370	300
Si	<5	<1	<10	<25	<6	170	8,180	2,180	1,540	3,000
Sr	3.2	1.5	356	110	33		2,490	4,100	56	
U	<20	<4	<40	<100	<25	600	<4200	<15,000	<15,000	600
U[1]	0.52	0.85	21	18	6.2	600	NA	NA	NA	600
W	<20	[4.8]	<40	<100	<25		125	<3700	<3700	
Zn	[1.2]	[0.45]	<1	<3	<0.6	16.5	191	97	78	6
Zr	<0.5	<0.1	<1	<3	<0.6		18	8	12	600
TOC[2]	3,640	1,010	923	461	156	1,500	24,000	20,700	17,100	60
TIC[2]	743	349	<33	<33	<7	150	24,500	30,200	660	30
F[3]	960	290	<250	<250	<125	150	NA	NA	NA	7,500
Cl	<500	<250	<250	<250	<125	25	NA	NA	NA	230
NO_2	8,100	2,000	<500	<500	<250		NA	NA	NA	
NO_3	54,500	13,700	44,600	51,800	20,900	3,000	NA	NA	NA	450
PO_4	<1000	<500	<500	<500	<250	2,500	NA	NA	NA	600
SO_4	<1000	<500	<500	<500	<250	2,300	NA	NA	NA	1,200
oxalate	<1000	<500	<500	<500	<250		NA	NA	NA	
NH_3	0.75	0.25	0.33	0.25	<0.2	150	NA	NA	NA	60

1) Analyzed by kinetic phosphorescence.
2) Reported in micrograms C per mL or per g.
3) Results are suspect due to peak distortion and retention time shift.

Bracketed results represent values with errors exceeding 15%, 2-σ.
NA: not analyzed

Table 3.9. Mass and Activity Balances for Calcium Carbonate and Barium Sulfate Precipitations, Phase 3

				$CaCO_3$ precipitate washes						$BaSO_4$ precipitate washes						Fractional recoveries				
	Actual	Filtrate from	Filtrate from	0.01M NaOH		1M HNO_3		DI H_2O		0.01M NaOH		1M HNO_3		DI H_2O						
	AN-107	$CaCO_3$	$BaSO_4$	Wash 1	Wash 2	Wash 1	Wash 2	Wash 1	$CaCO_3$	Wash 1	Wash 2	Wash 1	Wash 2	Wash 1	$BaSO_4$	Combined	Product			
ID	feed	ppt	ppt	00-466	00-467	00-469	00-470	00-471	00-472	00-476	00-477	00-479	00-480	00-481	00-482	wash	$CaCO_3$	($BaSO_4$)	$BaSO_4$	Total
Units	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	mg	solutions	precipitate	filtrate	precipitate[2]	recovered
Analyte																				
Al	1,700	477	394	6.0	1.7	9.9	2.4	0.3	1,530	7.9	2.2	1.0	0.09	0.04	0.21	0.02	0.90	0.23	0.0001	1.15
B	14	1.7	0.8	3.2	2.2	0.9	5.1	1.4	4.0	0	0	0.2	0.2	0.1	3.48	0.97	0.29	0.06	0.25	1.57
Ba[1]	<0.2	1	109			0.4	0.1		1.1	1.6	0.6	1,060	126	137	3460	0.39		0.03	1.01	1.43
Ca[1]	125	528	455	210	72	5,430	4,310	879	8,970	11	4.1	148	15	8	2	0.51	0.42	0.02	0.0001	0.95
Cd	20	14	11	4.2	0.6	0.2	0.2	0.1	0.9	0.2	0.06	0.2	0.02	0.01	0.70	0.29	0.04	0.57	0.04	0.94
Cr	31	20	3.3	6.5	1.4	0.4	0.1	0.1	2.9	0.08	0.03	2.6	0.89	0.76	15.5	0.42	0.09	0.11	0.50	1.12
Cu	9.3	6.5	5.4	1.9	0.4		0.1		0.4	0.1	0.03	0.03			0.200	0.27	0.04	0.59	0.02	0.92
Fe	6.4	1.8	1.4			0.3	0.4	0.2	4.3	0.04	0.01	0.05	0.02	0.02	1.30	0.17	0.68	0.22	0.20	1.27
K	520	363	386	89	20					7.8	2.0	1.9			87.1	0.23		0.74	0.17	1.14
Mo	12	8.3	6.8	2.6	0.5					0.1	0.04	0.05			0.1	0.28		0.58	0.01	0.87
Na	79,850	58,100	48,600	16,200	3,880	2,270	489	89	65	1,020	301	126	9.5	4.7	7.7	0.31	0.001	0.61	0.0001	0.91
Ni	151	110	90	35	6.4	1.9	0.8	0.2	2.1	1.7	0.47	0.5	0.08	0.06	0.10	0.31	0.014	0.60	0.001	0.92
P	219	27	22	7.0	1.4				208	0.4	0.11	0.4	0.10	0.06	0.60	0.04	0.95	0.10	0.003	1.09
Pb	42	14	11	1.5	0.2	3.0	5.4	1.3	15.8	0.2	0.06				2.6	0.28	0.38	0.27	0.06	0.99
Sr	94	0.6	4	0.3	0.3	43	19	3.9	31	0.1	0.06	17	1.8	0.96	0.30	0.93	0.33	0.04	0.003	1.30
U	6.0	3.4	1.11	0.55	0.08	0.03	0.17	0.03	NA	0.02	0.03	1.00	0.3	0.2	NA	0.40		0.18		0.58
Zn	5.0	2.6	2.2	0.6	0.2	0.3	0.5	0.1	1.3	0.04	0.02				0.5	0.35	0.26	0.44	0.10	1.15
SO_4	2,918	1,850	498	699	130		192		37						2425[3]	0.35	0.01	0.17	0.83	1.36
	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi	mCi					
^{60}Co	3.6E-2	2.3E-2	2.0E-2	7.6E-3	1.4E-3	3.1E-4	2.1E-4	4.6E-5	3.8E-3	3.7E-4	9.7E-5	2.3E-4	8.1E-5	5.6E-5	3.1E-4	0.29	0.10	0.56	0.009	0.96
^{137}Cs	6.0E-2	4.1E-2	3.8E-2	1.4E-2	3.0E-3	1.3E-3	2.1E-4	3.6E-5	2.8E-5	7.7E-4	2.1E-4	6.6E-5	5.7E-6	5.5E-6	9.7E-6	0.33	0.00	0.62	0.0002	0.96
^{154}Eu	2.4E-2	3.4E-3	1.6E-3	4.4E-4		6.3E-5	4.6E-4	7.5E-5	2.3E-2	1.6E-5	2.4E-5	8.7E-4	1.8E-4	8.3E-5	1.1E-4	0.09	0.94	0.07	0.005	1.10
^{155}Eu	1.7E-2	2.5E-3	1.1E-3	3.0E-4	2.1E-5		3.4E-4	5.1E-5	1.6E-2	1.1E-5	1.5E-6	6.0E-4	1.3E-4	7.9E-5	6.4E-5	0.09	0.94	0.07	0.004	1.09
^{241}Am	8.7E-3	1.2E-3	6.9E-4	1.6E-4	1.7E-5	3.2E-5	1.5E-4	2.3E-5	8.4E-3	7.5E-6	1.6E-6	2.0E-4	5.4E-5	3.8E-5	2.6E-5	0.08	0.96	0.08	0.003	1.12
^{99}Tc	4.3E-2	3.1E-2	2.5E-2	9.2E-3	1.7E-3	4.2E-4	1.8E-4	3.8E-5	1.4E-3	4.9E-4	1.3E-4	1.1E-4	2.8E-5	1.8E-5	NA	0.28	0.03	0.58	0.10	0.90
^{237}Np	3.1E-5	1.3E-5	6.3E-6	2.3E-6			1.1E-7		1.3E-5	8.4E-8	1.4E-8	3.4E-6	4.3E-7	2.7E-7	NA	0.22	0.41	0.21	0.17	0.83
^{239}Pu	1.1E-3	3.8E-4	2.2E-4	7.1E-5	1.1E-5		1.1E-5	3.3E-6	6.4E-4	2.8E-6	4.4E-7	8.2E-5	2.1E-5	1.2E-5	NA	0.20	0.58	0.20	0.02	0.98
^{90}Sr	1.8E-1	4.4E-3	1.2E-3	2.4E-3	1.5E-3	3.4E-1	8.4E-2	5.6E-3	2.6E-1	3.2E-5	1.9E-5	1.3E-5	4.4E-4	2.6E-4		2.39	1.42	0.01		3.82

1) Fractional recoveries for Ca and Ba include the added Ca and Ba as well as that originally present in the Actual AN-107 feed.
2) Analyte composition in $BaSO_4$ was by difference for ^{99}Tc, ^{237}Np, and ^{239}Pu.
3) The given sulfate value was not measured but is provided based on the Ba value and stoichiometric $BaSO_4$ composition and could be biased high.

Note: NA indicates not analyzed empty spaces indicate non-detected analytes.

3.4 Metathesis with $BaCO_3$

Batch contact of the Archive 1 AN-107 material directly with the $BaCO_3$ resulted in no detectable change from the starting sulfate concentration of 0.041M over the wide range of $BaCO_3$:SO_4 molar ratios. Contact with Ca-pretreated Archive 2 AN-107 (Ca:CO_3 equal to 0.93:1) also resulted in no measurable sulfate concentration change, despite the drop in carbonate concentration from 0.7M to 0.25M. Under these processing conditions, metathesis did not occur to any measurable extent. Contact at elevated temperature might yield more favorable results.

4.0 Conclusions

Direct addition of Ba^{2+} to AN-107 tank waste solutions overwhelmingly favors $BaCO_3$ over $BaSO_4$ formation. Even a 5:1 $Ba:SO_4$ mole ratio addition resulted in inconsequential sulfate removal in this matrix. It was found that by dropping the carbonate concentration from nominally 0.7M to 0.2M, Ba^{2+} addition did result in significant sulfate removal. This gross carbonate removal was effected by an initial Ca^{2+} pre-strike of 1:1 mole ratio of $Ca:CO_3$. This corresponds to a carbonate concentration reduction from 16 times to 6 times that of the sulfate concentration (0.2M CO_3^{2-} and 0.032M SO_4^{2-}) and is enough to favor the reaction forming $BaSO_4$ over $BaCO_3$ upon addition of Ba^{2+}. Using an initial 1:1 $Ca:CO_3$ mole ratio addition in conjunction with the follow-on 1:1 mole ratio $Ba:SO_4$ addition, 70% sulfate can be removed from AN-107.

Calcium addition, to effect carbonate removal, has significant drawbacks. The volume associated with the 1:1 $Ca:CO_3$ precipitate is large, consuming nominally 35% by volume (including interstitial liquids) after filtering. The settling rate for $CaCO_3$ in the Actual AN-107 matrix is extremely slow. Upon standing for approximately 2 days, the slurry virtually solidifies. However, while still fluid, the $CaCO_3$ slurry can be filtered at reasonable rates.

Both $Ba(NO_3)_2$ and $Ba(NO_2)_2$ were tested and are equally effective for sulfate removal. Because the latter has a higher solubility (2.9M as opposed to 0.33M), the use of $Ba(NO_2)_2$ results in a smaller volume increase, i.e., less waste production, than the $Ba(NO_3)_2$. There is no indication that the source of soluble Ba^{2+} impacts the precipitation behavior.

The $BaSO_4/BaCO_3$ precipitate has relatively favorable processing characteristics. The precipitate volume is much smaller and less flocculent than the $CaCO_3$, and it settles rapidly. Upon standing, the slurry does not solidify, but remains fluid. The precipitate can be filtered at reasonable rates and does not appear to cause filter clogging.

Metathesis of $BaCO_3$ to $BaSO_4$ at room temperature in the AN-107 matrices tested is not measurably successful. Initial gross carbonate removal from the AN-107 matrix also has no effect on sulfate removal by metathesis.

5.0 References

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APPENDIX A

Appendix A: Test Instruction BNFL-TI-29953-061 and Analytical Reports for Sample Analysis

APPENDIX B

Appendix B: Test Instruction BNFL-TI-29953-064 and Supporting Analytical Data

APPENDIX C

Appendix C: Test Instruction BNFL-TI-29953-071 and Supporting Analytical Data

Table C.1. Actual AN-107 Scale-Up Test Detail, Phase 3

	Solutions								Solids					
Description	AN107 mass (g)	Reactant and filtrate mass (g)	Density (g/mL, T~20C)	Solution vol (mL)	Total filtration time (min)	Sampled mass (g)	Net mass remaining (g)	Wt. % Recovered Filtrate	Wet solid in filter (g)	Approx. volume (mL)	Wet solids sampled (g)	Wet solids remaining (g)	Wt.% solids	Wt. % change on wash
$CaCO_3$ Precipitation														
AN107 Starting material	926.4		1.224	756.6		26.9	899.5		na				na	
Added 5M $Ca(NO_3)_2$		163.4	1.527	107.0			1062.9							
AN-107+$CaCO_3$, suspended solids		1062.9	1.241	856.3		12.413	1050.5							
Filter	678.6		1.209	561.3	77	36.2	642.4	64.6%	361.1	300	2.9	358.2	39.0	
Wash 0.01M NaOH		677.7	1.068	635 (filtrate)	62	24			291.4	300				-19%
Wash 0.01M NaOH, 2nd		551.1	1.014	543 (filtrate)	60	24			254.4	300	7.1	247.3	24.0	-13%
Wash 1.0M HNO_3		517.5	1.039	498 (filtrate)	20	24			140	200	0	140		-45%
Wash 1.0M HNO_3, 2nd		264.2	1.049	252 (filtrate)	9	24			91.2	100	0	91.2		-35%
DI water wash		166	1.009	165 (filtrate)	11	24			83.9	50	est. 5g	nm	34.3	-8%
$BaSO_4$ Precipitation														
AN-107 Filtrate from Ca prestrike	642.4		1.209	531.4			642.4							
Added 1.82M $Ba(NO_2)_2$		18.35	1.344	13.7										
AN-107+$BaSO_4$ suspended solids			1.210			12.099	648.65							
Filter	619.4		1.206	513.4	82	35	584.4	95.5%	22.1	20	0.8728	21	46.4	
Wash 0.01M NaOH		39.6	1.061	37.3 (filtrate)	1	24			nm					
Wash 0.01M NaOH, 2nd		40.4	1.014	39.8 (filtrate)	<1	24			20.2	20	1.63	18.6	34.5	-3.8%
Wash 1.0M HNO_3		50.1	1.051	47.7 (filtrate)	1	24			8.7	10		8.7		-57%
Wash 1.0M HNO_3, 2nd		16.5	1.041	15.9 (filtrate)	1	24			13.2	10		13.2		+52%
DI water wash		29.5	1.012	29.2 (filtrate)	4	24			13.3	10	all		44.4	+1%

Notes: na = not applicable, nm = not measured

Figure C.1. $CaCO_3$ Slurry Settling Test at T=2 Hours

Figure C.2. $BaSO_4$ Slurry Settling Test at T=2 Hours

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APPENDIX A

Appendix A: Test Instruction BNFL-TI-29953-061 and Analytical Reports for Sample Analysis

PNNL Test Instruction	Document No.: BNFL-TI-29953-061 Rev. No.: 0
Title: Sulfate Removal Studies from AN107 Archived Waste	
Work Location: Building 325, various laboratories	**Page** 1 of 7
Author: SK Fiskum	**Effective Date: New** **Supersedes Date: New**
Use Category Identification: Reference	
Identified Hazards: _X_ Radiological _X_ Hazardous Materials ___ Physical Hazards ___ Hazardous Environment ___ Other:	**Required Reviewers:** _x_ Author _x_ Technical Reviewer __ RPL Manager __ Project Manager __ RPG Quality Engineer __ BNFL (not required)

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

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

On-The Job Training Required? _____ Yes or __x__ No

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

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

Approval	Signature	Date
Author	Sandra K. Fiskum	10/13/99
Technical Reviewer	Richard T. Hallen	10/13/99

~~Controlled~~ Uncontrolled Document

Applicability

This test instruction outlines 3 tests for sulfate removal from AN-107 Archive material. The first test incorporates an initial carbonate removal using Ca followed by a series of experiments using various ratios of [Ba(NO3)2] : [SO4]. The second test explores the metathesis of BaCO3 solid to BaSO4 as it is contacted with sulfate from the supernatant material. The third test examines the applicability of ion exchange material to remove sulfate from solution.

The AN107 Archive material is composed of the following: 0.041M sulfate, 0.7M carbonate, 0.01M EDTA, 0.0039M HEDTA (the last 3 molarities are assumed based on known initial concentrations and an estimated dilution factor of 2x).

DRD Reference: 12.2

Schedule Reference: BNFL desires completion of these tests and anion analyses by October 29, 1999.

Justification

Work was requested by BNFL to provide information for conceptual design of sulfate removal system.

Objective

These experiments will help define an optimal method for sulfate removal from tank waste solutions prior to vitrification.

Work Instructions

I. Calcium nitrate prestrike

1. Obtain AN107 Archive tank waste with Cs and Sr removed from lab 410. The bottle is contaminated externally so caution (draping/extra gloves) is necessary when handling this solution.

2. Aliquot 25 mL of AN107 Archive into each of 5 tared vials (40-mL vials containing a stir bar), labeled as CA1, CA2, CA3, CA4 and CA5. Record the net weight of AN107 Archive in each vial.

Sample ID	Tare mass * (g)	Gross mass (g)	Net mass (g)	Tare w/ stir
CA1	29.8714	60.4509 g	30.4214	30.0295
CA2	29.4336	59.9812	30.3826	29.5986
CA3	29.7408	60.3101	30.4134	29.8967
CA4	29.7821	60.351~~9~~ DKT 10/14	30.4083	29.9436
CA5	29.9233	60.4763	30.3875	30.088~
Balance ID	360-06-01-033	360-06-01-026		360-06-~ J

* No stir bar

A 25-mL aliquot of AN107 Archive material is expected to contain 1 millimole sulfate, 17.5 mmoles carbonate, 0.25 millimoles EDTA, and 0.0975 millimoles HEDTA.

3. Prepare a Ca(NO3)2-4H2O solution by dissolving 118 g Ca(NO3)2-4H2O into ~~50~~ 100 mL water (near maximum solubility) in a 50-mL volumetric flask. Final solution is ~~10~~ 5 M Ca.
   Tare out volumetric flask on balance, add Ca(NO3)2, weigh, add water, weigh, stir until dissolution is complete, transfer to plastic bottle.
   Vol flask tare 62.2538 g
   Mass of Ca(NO3)2-4H2O 118.00 g Aldrich, ACS grade
   Final mass ~~50~~ 100-mL dilution 215.37 g final net mass = 153.12 g
   Calculated density 1.531 g/mL
   T = 20 °C

4. To CA1 add ~~0.875~~ 1.75-mL ~~10.0~~ 5 M Ca solution, also add ~~2.62~~ 5.25-mL DI water (8.75 millimoles Ca providing a 0.5:1 Ca:CO3 ratio)

5. To CA2 add ~~1.75~~ 3.50-mL ~~10.0~~ 5 M Ca solution and ~~1.75~~ 3.50 mL DI water (17.5 millimoles Ca providing a 1:1 Ca:CO3 ratio)

6. To CA3 add ~~3.5~~ 7.0-mL ~~10.0~~ 5 M Ca solution, no DI water (35 millimoles Ca providing a 2:1 Ca:CO3 ratio)

7. To CA4 add ~~3.5~~ 7.0-mL DI water

8. To CA5 add ~~1.75~~ 3.5-mL ~~10.0~~ 5 M Ca solution and ~~1.75~~ 3.5 mL DI water (17.5 millimoles Ca providing a 1:1 Ca:CO3 ratio)

Lots steps 3-7 SK 10/14/99 see Note section.

Sample ID	Vial + sample mass	Mass after adding Ca solution (g)	Mass Ca solution added (g)	Mass after adding DI water	Mass DI water added (g)
CA1	60.4509	63.0939	2.6430	68.3419 g	5.2480
CA2	59.9812	65.3054	5.3242	68.8156	3.5102
CA3	60.3101	71.0026	10.6925	71.0025	-0.0001
CA4	60.3519	60.3518	-0.0001	67.3657	7.0139
CA5	60.4763	65.7796	5.3033	69.2814	3.5018

9. Mix solutions well for about 5 minutes on a stir plate and allow to sit for 60 minutes. Centrifuge samples for about 3 minutes at about 1500 rpm.

10. Separate the supernate from the precipitate by siphoning with a transfer pipet, loading into a syringe/filter apparatus (0.45µm filter) and filter into a clean vial (35-mL) containing a stir bar. The precipitate is to be retained for possible future analyses.

11. Remove 3-mL from each sample and place in a 2-dram or smaller vial for TIC and IC analysis. Label as CA1-a, CA2-a, CA3-a, CA4-a, and CA5-a.

12. Split the remaining solution (nominally 25-mL) from the 4 vials as follows:
use 4.5 mL inoked (except CA3).

CA1 ~~4~~mL to CA1-b, ~~4~~-mL to CA1-c, ~~4~~-mL to CA1-d, ~~4~~-mL to CA1-dd, ~~4~~-mL to CA1-e
CA2 ~~4~~mL to CA2-b, ~~4~~-mL to CA2-c, 4-mL to CA2-d, 4-mL to CA2-dd, ~~4~~-mL to CA2-e
CA3 4mL to CA3-b, ~~4-mL to CA3-c, 4-mL to CA3-d~~, 4-mL to CA2-dd, ~~4-mL to CA3-e~~
CA4 ~~4~~mL to CA4-b, ~~4~~-mL to CA4-c, ~~4~~-mL to CA4-d, 4-mL to CA2-dd, ~~4~~-mL to CA4-e
CA5 will be used with BaCO3 and IX experiments in sections II and III.

A ~~4~~ 4.5-mL aliquot of the diluted AN107 Archive sample (25mL to ~~28.5~~ 32mL from Ca prestrike) is expected to contain 0.14 millimoles sulfate, 0.035 millimoles EDTA and 0.014 millimoles HEDTA.

12. Dissolve 8.7g BaNO3 in 100 mL DI water (this gives a 0.33M Ba solution).
Vol. flask tare 62.2223 g
Mass of BaNO3 8.7103 g
Final mass 100-mL dilution 168.5327 (+ vol flask) Net mass: 106.3104 g
Calculated density 1.0631 g/mL

14. To each of the –b vials, add 0.424 mL 0.33M Ba solution (0.14 millimoles Ba providing a 1:1 Ba:SO4 ratio)

15. To each of the –c vials, add ~~0.688~~ 0.585mL 0.33M Ba solution (~~0.189~~ 0.193 millimoles Ba providing a 1:1 Ba: [SO4 + EDTA + HEDTA] ratio)

16. To each of the –d and –dd vials, add 1.27 mL 0.33M Ba solution (0.42 millimoles Ba, providing a 3:1 Ba:SO4 ratio)

17. To each of the –e vials, add 2.12 mL of 0.33M Ba solution (0.70 millimoles Ba).

Sample ID	Vial Tare + Stir bar (g)	Vial + added sample mass	4-mL AN107 Archive prestrike mass (g)	Vial + sample + Ba	0.33M Ba mass added
CA1-b	10.1800	15.5044 g	5.3244	15.9620 g	0.4576 g
CA2-b	10.0755	15.4020	5.3265	15.8574	0.4554
CA3-b	10.1403	14.9260	4.7857	15.4793	0.5533
CA4-b	10.1412	15.4379	5.2967	15.8933	0.4554
CA1-c	10.3471	15.6697	5.3199	16.2920	0.6243
CA2-c	10.1701	15.5024	5.3323	16.1225	0.6201
CA3-c	10.2466				
CA4-c	10.8258	15.5121	5.2863	16.1331	0.6210
CA1-d	10.2115	15.5474	5.3359	16.9362	1.3888
CA2-d	10.1562	15.4803	5.3241	16.8641	1.3838
CA3-d					
CA4-d	10.2855	15.5826	5.2971	16.9671	1.3845

Balance ID 360-06-01-038 360-06-01-026

CA1-dd					
CA2-dd					
CA3-dd			JK 10/15/99		
CA4-dd	10.1919	15.4809	~~16.8606~~ 5.2890	16.8606	1.3797
CA1-e	10.3761	15.6994	5.3233	18.0024	2.3030
CA2-e	10.2787	15.6075	5.3288	17.8962	2.2888
CA3-e					
CA4-e	10.1903	15.4775	5.2872	17.7688	2.2913

18. Stir each solution for about 5 minutes and allow to sit for about 60 minutes. Centrifuge samples for about 3 minutes at about 1500 rpm.

✓19. Separate the supernatant from the precipitate by siphoning with a transfer pipet, loading into a syringe/filter apparatus (0.45-µm filter) and filter into a clean 2-dram vial. Retain the precipitate for possible future analyses.

20. Aliquot 3-mL of the solution for IC and TIC analyses.

21. Send the IC and TIC sample aliquots in for analyses. Keep remaining supernatants for possible future testing.

II. Barium carbonate Batch contact

1. Measure BaCO3 (shelf supply, powdery material) into 20-mL vials as follows:

ID	BaCO3 mass	mole ratio
BA1:	0.039g	1:1 Ba:SO4
BA2:	0.197g	5:1 Ba:SO4
BA3:	0.395g	10:1 Ba:SO4
BA4:	0.986g	25:1 Ba:SO4
BA5:	0.0355g	1:1 Ba:SO4 after Ca prestrike 1:1 ratio of Ca:CO3
BA6:	0.178g	5:1 Ba:SO4 after Ca prestrike 1:1 ratio of Ca:CO3

2. Add 5-mL AN107 Archive waste to the first 4 vials (this correlates to about 0.2 millimoles sulfate). Add 5-mL of CA5 (AN107 Archive Ca prestike material) to vials BA5 and BA6 (this correlates to about 0.18 mmoles sulfate).

Sample ID	Vial tare (g)	BaCO3 mass + vial mass (g)	Net BaCO3 mass (g)	BaCO3 + AN107 Archive + vial mass	Net AN107 Archive mass
BA1	16.8826	16.9223	0.0397		
BA2	16.9189	17.1151	0.1962	23.1398	6.0247
BA3	17.1386	17.5302	0.3916	23.5489	6.0187

Balance ID 360-06-01-038

BA4	16.8843g	17.8806 g	0.9963 g	23.8517 g	5.9711 g
				BaCO3+ AN107 Ca prestrike +vial	Net AN107 Arc. Prestrike mass
BA5	17.2888	17.3266	0.0378		
BA6	16.7950	16.9893	0.1943		

3. Seal each sample and transfer to room 507. Place samples in the shaker along with a thermometer to monitor temperature and shake on low setting for nominally 24 hours.

4. Remove samples from the shaker. Filter each sample through a 0.45-μm filter into a clean 2-dram vial.

5. Aliquot 3-mL and transfer to Marilyn Steele for IC analysis. Save remaining supernatant fraction for possible additional analyses.

III. Ion exchange material study

1. Measure the mass of IX material (provided by Rich Hallen) into 20-mL vials as follows:

ID	IX mass	Ratio IX capacity to SO4
IX1	0.05g	0.5:1
IX2	0.1g	1:1
IX3	0.5g	5:1
IX4	1.0g	10:1
IX5	0.05g	0.5:1 after Ca prestrike 1:1 ratio of Ca:CO3
IX6	0.09g	1:1 after Ca prestrike 1:1 ratio of Ca:CO3

Sample ID	Vial tare mass	Tare + IX mass	Net IX mass	Tare + IX mass + AN107 Archive	Net AN107 Archive mass
IX1	16.9956	17.0516	0.0560	23.0761	6.0245
IX2	16.9628	17.0705	0.1077	23.0884	6.0179
IX3	JK 10/14/99 ~~17.2402~~ 17.0532	17.5462	0.4930	23.5845	6.0383
IX4	16.9885	18.0019	1.0134		
			JK 10/14/99 1X	~~BaCO3~~+ AN107 Ca prestrike +vial	Net AN107 Arc. Prestrike mass
IX5	16.9072	16.9631	0.0559		
IX6	17.0549	17.1484	0.0935		

2. Add 5-mL AN107 Archive waste to the first 4 vials. Add 5-mL of CA5 (AN107 Archive Ca prestike) to IX5 and IX6.

3. Seal each sample and transfer to room 507. Place samples in the shaker along with a thermometer and shake on low setting for nominally 24 hours.

4. Remove samples from the shaker. Filter each sample through a 0.45-μm filter into a clean 2-dram vial.

5. Aliquot 3-mL and transfer to Marilyn Steele for IC analysis.

6. Measure 0.5g resin into a tared vial. Heat the resin at nominally 95°C for 3 days. Check the mass of resin (after cooling) at 1-day intervals until constant mass is achieved.

Vial tare mass	_____
Initial mass (vial + resin)	_____
Calculated resin mass	_____
Mass 1-day heating vial + resin	_____
Mass day-2 heating vial + resin	_____
Mass day-3 heating vial + resin	_____
Calculated final resin mass	_____.

Shaker time on: 4:20 p.m. 10/14/99
off: 1:45 p.m. 10/15/99.
for both the K + $BaCO_3$ contacts.

Battelle
Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	TI: BNFL-TI-29953-001 Rev. No. 0
CLIENT	BNFL Sulfate Removal

Observations I

Step 1 Upon transfer of the AN107 Archive material to a clean vial, I noticed some particulate material which transferred toward the end of the pour. ~170-mL volume total

Step 3. Preparation of 10 M $Ca(NO_3)_2 \cdot 4H_2O$ solution proved to be difficult. Solution was syruppy ~ almost like Karo syrup. I decided to reduce molarity to 1/2 (5M) and add 2X originally specified volume. Thus the total number of millimoles added remain the same. However final solution volume increases, decreasing anticipated molarity of analytes of interest. To compensate, I will take a proportionately larger volume of Ca-prestrike solution for additional testing.

Original plan

$$\frac{25 \text{ ml waste}}{28.5 \text{ ml final vol}} \times 0.041 \text{ M } SO_4 \times 4 \text{ mL} = 0.144 \text{ mmoles } SO_4$$

New plan:

$$\frac{25 \text{ ml waste}}{32 \text{ ml final vol}} \times 0.041 \text{ M } SO_4 \times 4.5 \text{ mL} = 0.144 \text{ mmole } SO_4$$

Note: All masses are taken with caps ON vessel. All vials & caps have been water-leached.

Step 9: Lots of precipitate formed. So much ppt mass caused difficulty with stirring using the stir bar / stir plate. For the most part I mixed these by shaking them by hand. CA3 copious ppt.

analyst D.K. Fiskum date 10/14/99 reviewer _____ date ____

Battelle
Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	
CLIENT	BNFL Sulfate Removal Removal

Pipet Checks 10/14/99 T= 21°C Balance = 360-06-01-026

DK 10/14/99

1.7591g, 1.7481, ~~1.7481~~, 1.7547, 1.7642

3.5035g, 3.5002, 3.5036, 3.4999

6.9616g, 6.9865, 6.9871, 6.9740

5.2595, 5.2405, 5.2445, 5.2583

analyst D.K. Fiskum date 10/14/99 reviewer _____ date _____

Battelle
Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	BNFL-TI-29953-061 Rev #0
CLIENT	BNFL Sulfate Removal

Step 9: After ~1.5 hour settling time, CA1 is about 3/4 vol. supernate ~ 1/4 volume ppt.
CA2 is about 1/8 vol supernate ~ 7/8 vol. ppt.
CA3 is ~~about~~ SK# 10/14/99 is all tan shade ppt. (no phase separation)
CA5 is similar to CA2
After centrifuging ~3 min at 1700 rpm CA1 is ~ 1/5 by vol. ppt., and 4/5 supernate
CA2 is ~ 1/2 supernate ~ 1/2 ppt.
CA5 is ~ 1/2 " ~ 1/2 ppt
CA3 is ~ 1/3 supernate, ~ 2/3 ppt. Also the supernatant color is much paler yellow compared to the others.
All ppts are light tan.

Step 10 CA3 ~ 9 mL CA2, CA5 ~ 14 mL CA4 ~ 32 mL CA1 ~ 22 mL
Combined CA2 + CA5 supernatants so we could run a larger suite of tests.
The IX + $BaCO_3$ contacts with prestrike material were eliminated.

analy J.K. Hokan date 10/15/99 reviewer _____ dat ____

Batte
Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	
CLIENT	BNFL Sulfate Removal

Pipet checks 10/15/99 T= 19°C

Aliquot Ca pre-strike solution to vials 4.5 mL: Balance = 1360-06-01-026

DI H_2O 4.4999g, 4.4926, 4.5063

4.0 mL:

4.0080g, 3.9894, 3.9992

Aliquot $BaNO_3$ (0.33M) into CA# -b:

0.4308g, 0.4344, 0.4300, 0.4304

Aliquot $BaNO_3$ (0.33M) into CA# -c

0.5833g, 0.5860, 0.5839, 0.5842

Aliquot $BaNO_3$ into CA# -d, -dd

1.3096g, 1.3073, 1.3080, 1.3086

Aliquot 0.33M $BaNO_3$ into CA# -e

2.1511g, 2.1644, 2.1649, 2.1661

Aliquot 0.33M $BaNO_3$ into CA3-b (0.52 mL)

0.5179g, 0.5190, 0.5203, 0.5202

analyst V.K. Fiskum date 10/15/99 reviewer _____ date _____

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: D. Blanchard

RECORD COPY

29953
Task 2.12
T 2.12.5.2
TI 61
SO_2 removal
Screening Tests
1st test
AN-107
archive.

ACL Number(s): 00-0157 through 00-0172

Client ID: "CA2-a" through "CA2-e"

ASR Number: 5552

Total Samples: 5

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

Analyst: D.R. Sanders

Analysis Date (Filename): 12-01-99 (A0562)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry Wagner 12-28-99
Reviewed by

M W Lin 1-4-00
Concur

12/28/99

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Five radioactive liquid samples, CA2-a through CA2-e (ACL# 00-0157 through 00-0172), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 1 ml of sample (weighed) was digested and diluted to a final volume of approximately 10ml. The final volume was weighed. Concentration is adjusted for dilution from processing using the weight of final solution divided by sample weight.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in µg/ml for liquid samples. Weights have been recorded on bench sheets and included with this report.

Liquid samples contained high concentrations of sodium (approximately 5 to 7%). All other analytes measured were much lower in concentration.

Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

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

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

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

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

Blank Spike:
(Aqueous samples) None required or prepared.

Matrix Spiked Sample:
(Aqueous samples) None required or prepared.

Quality Control Check Standards (aqueous samples):

Concentration of all analytes was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except as follows. Tin and thorium were low, up to 18% in QC_MCVB check standard measurements. Single element standards of tin at 2 µg/ml and thorium at 10 µg/ml measured were well within the tolerance limit thus confirming calibration check for these two analytes. One measurement out of five for silicon in calibration blank ICP98.0 was slightly greater than 2 * IDL (up to 2.2 times IDL). The single high silicon reading in the calibration blank is likely due to carry-over from a previous sample containing high concentration of silicon. The four other measurements for silicon in the blank is within tolerance.

High Calibration Standard Check (aqueous samples):

Verification of the high-end calibration concentration for all analytes is within tolerance of ± 5% accuracy.

Process Blank:

(Aqueous samples) All analytes are within tolerance limit of ≤ EQL or < 5% of sample concentration except Ba and Zn (greater than 17% to 91% of sample concentration). The blank appears to be contaminated with barium and zinc. Barium and zinc concentration in the blank is about the same as that found in the samples. The source of contamination is not known. Since both barium and zinc are very high in the process blanks, sample measurements for these two analytes should be considered suspect.

Laboratory Control Standard (LCS):

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

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

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

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

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= (Analyte)	9.4 00-157-PB Process Blank 12/1/99 ug/mL	43.5 00-157 @5 CA2-a 12/1/99 ug/mL	42.9 00-157-DUP @5 CA2-a 12/1/99 ug/mL	16.9 00-162 @2 CA2-b 12/1/99 ug/mL	16.8 00-166 @2 CA2-c 12/1/99 ug/mL
0.025	Ag	–	–	–	–	–
0.060	Al	–	47.1	47.6	45.2	44.3
0.250	As	–	–	–	–	–
0.050	B	–	[5.3]	[5.6]	12.6	12.2
0.010	Ba	3.70	[0.71]	–	56.1	86.8
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	[2.4]	736	742	781	691
0.015	Cd	–	16.6	16.7	14.7	14.3
0.200	Ce	–	–	–	–	–
0.050	Co	–	–	–	[1.1]	[1.0]
0.020	Cr	–	9.36	9.50	[1.6]	[1.3]
0.025	Cu	–	11.7	11.4	10.8	10.4
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	–	–	–	[0.73]	[0.67]
2.000	K	–	[450]	[450]	433	419
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	–	–	–	–
0.050	Mo	–	[10]	[10]	8.83	8.54
0.150	Na	–	74,500	73,100	67,200	66,000
0.100	Nd	–	–	–	–	–
0.030	Ni	[0.49]	162	165	140	136
0.100	P	–	[36]	[36]	31.4	30.0
0.100	Pb	–	[24]	[24]	20.7	20.1
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	–	–	[42]	[44]
1.500	Sn	–	–	–	–	–
0.015	Sr	–	[0.68]	[0.67]	[1.7]	[0.95]
1.500	Te	–	–	–	–	–
1.000	Th	–	–	–	–	–
0.025	Ti	–	–	–	–	–
0.500	Tl	–	–	–	–	–
2.000	U	–	–	–	–	–
0.050	V	–	–	–	–	–
2.000	W	–	–	–	[40]	[38]
0.050	Y	–	–	–	–	–
0.050	Zn	11.7	[14]	[6.1]	10.1	13.0
0.050	Zr	–	–	–	–	–

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).

5/6

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	17.4 / 00-169 @2 / CA2-d / 12/1/99 / ug/mL	17.1 / 00-172 @2 / CA2-e / 12/1/99 / ug/mL			
0.025	Ag	–	–	–	–	–
0.060	Al	40.6	35.4	–	–	–
0.250	As	–	–	–	–	–
0.050	B	10.6	9.30	–	–	–
0.010	Ba	51.1	164	–	–	–
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	504	423	–	–	–
0.015	Cd	12.9	11.1	–	–	–
0.200	Ce	–	–	–	–	–
0.050	Co	[0.92]	–	–	–	–
0.020	Cr	[1.1]	[0.59]	–	–	–
0.025	Cu	9.43	8.00	–	–	–
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[0.63]	[0.66]	–	–	–
2.000	K	379	[320]	–	–	–
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	–	–	–	–
0.050	Mo	[7.8]	[6.7]	–	–	–
0.150	Na	59,300	51,700	–	–	–
0.100	Nd	–	–	–	–	–
0.030	Ni	125	107	–	–	–
0.100	P	27.6	23.4	–	–	–
0.100	Pb	18.2	[16]	–	–	–
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	[39]	[35]	–	–	–
1.500	Sn	–	–	–	–	–
0.015	Sr	[0.35]	[0.71]	–	–	–
1.500	Te	–	–	–	–	–
1.000	Th	–	–	–	–	–
0.025	Ti	–	–	–	–	–
0.500	Tl	–	–	–	–	–
2.000	U	–	–	–	–	–
0.050	V	–	–	–	–	–
2.000	W	[35]	–	–	–	–
0.050	Y	–	–	–	–	–
0.050	Zn	[3.6]	[6.3]	–	–	–
0.050	Zr	–	–	–	–	–

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).

6/6

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

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WO/Project: **W53400 / 29953**
Client: **D. Blanchard**

proj 29953
Task 2.12
T 2.12.5.2
TI-61

ACL Numbers: 00-00156 through 00-00180
ASR Number 5552

SO4 removal, 1st test AN-107 ordering

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Analyst: MJ Steele **Analysis Date:** October 21-23, 1999

M&TE: IC system (WD25214); Mettler AT400 Balance (360-06-01-031) See Chemical Measurement Center 98620 RIDS for IC File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: MJ Steele

Approval: MW Thun **Date** 10-28-99

Notes:

1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) 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.
3) Routine precision and bias is typically ± 15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Final Results:

The 25 liquid samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5520. The samples were diluted at the IC workstation up to 2,000-fold to obtain all anions were within the calibration range. However, at 2,000-fold the nitrate results exceed the calibration range. Per client request, no further nitrate dilutions were performed, since sulfate is the primary anion of interest. The anion results are presented in the table below.

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

Lab ID	Sample ID	F (1) ug/ml	Cl ug/ml	NO_2 ug/ml	Br ug/ml	NO_3 (2) ug/ml	PO_4 ug/ml	SO_4 ug/ml	C_2O_4 ug/ml
00-0156	CA1	2,900	760	21,700	< 250	102,000	< 500	3,100	1,400
00-0157	CA2	< 250	790	22,000	< 250	111,000	< 500	3,000	800
00-0158	CA3	2,500	810	22,600	< 250	81,300	< 500	2,700	< 500
00-0159	CA4	3,000	780	22,800	< 250	83,600	1,400	3,300	1,600
00-0160	CA5	2,700	720	20,400	< 250	111,000	< 500	2,800	< 500
00-0161	CA1-B	2,800	750	20,500	< 250	100,000	< 500	2,800	1,300
00-0162	CA2-B	2,800	790	21,300	< 250	111,000	< 500	1,200	700
00-0163	CA3-B	2,100	670	19,300	< 250	93,100	< 500	< 500	< 500
00-0164	CA4-B	2,600	670	19,600	< 250	80,200	< 500	2,800	1,400
00-0165	CA1-C	2,600	690	19,200	< 250	97,500	< 500	2,800	1,300
00-0166	CA2-C	2,500	660	18,800	< 250	110,000	< 500	800	700
00-0167	CA4-C	2,700	700	20,000	< 250	79,000	1,200	2,800	1,400
00-0168	CA1-D	2,300	610	17,000	< 250	92,900	< 500	2,600	1,100
00-0169	CA2-D	2,200	800	16,500	< 250	107,000	< 500	700	600
00-0169 Dup	CA2-D Dup	2,200	600	16,700	< 250	108,000	< 500	< 500	600
	RPD	0	29	1	n/a	1%	n/a	n/a	n/a
00-0169 MS Rec	CA2-D MS Rec	81%	97%	71%	99%	OvrRng	0% (3)	102%	104%
Blank Spike Rec	Blank Spike Rec	104%	101%	104%	104%	105%	102%	105%	103%
00-0170	CA4-D	2,200	570	16,300	< 250	76,000	< 500	2,300	1,200
00-0171	CA1-E	2,000	530	14,800	< 250	88,700	< 500	2,100	900
00-0172	CA2-E	1,900	520	14,500	< 250	104,000	< 500	< 500	< 500
00-0173	CA4-E	1,900	510	14,200	< 250	71,700	< 500	2,100	1,000
00-0174	CA4-DD	2,200	580	16,500	< 250	75,800	< 500	2,500	1,200
00-0175	BA2	3,800	1,000	28,600	< 250	97,900	1,500	3,900	2,100
00-0176	BA3	3,900	1,100	29,000	< 250	98,000	1,600	4,000	2,000
00-0177	BA4	< 250	1,000	27,800	< 250	98,000	1,600	3,900	2,300
00-0178	IX1	3,600	1,000	27,300	< 250	98,700	1,300	3,600	2,000
00-0179	IX2	3,600	930	27,400	< 250	98,700	1,300	3,900	2,000
00-0180	IX3	3,900	1,100	29,000	< 250	97,400	1,600	4,100	2,100

RPD = Relative Percent Difference (between sample and replicate); results not calculated if sample or replicate less than two times the EQL. The high RPD for Cl is due primarily to the very low measurement concentrations (i.e., 2.5x EQL).

(1) Areas quantified by IC system as fluoride; however, slight retention time peak shift and peak shape suggest significant organic anion interference. High probability that little or no fluoride is actually present in the samples. Although not verified, formate is the most probable interfering anion.

(2) Nitrite and nitrate results are over range (i.e., exceed highest calibration standard) by factors of 1.2 to 1.8; however, reasonable linearity has been demonstrated for concentrations at two times the highest calibration standard. Results exceeding the nitrate calibration range by less than 2x are typically bias low by 15 to 25%.

(3) The phosphate matrix spike is 0%; however, this is not unexpected due to the addition of barium. The phosphate blank standard spike demonstrated good recovery at 102%.

Analytical Q.C. Comments

Besides the duplicate, matrix spike, and blank spike QC, the following are results of analytical quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan.

System Blank/Processing Blanks: About 1 system blanks were process during the analysis of the samples. No anions were detected above reportable concentrations in the system blanks.

Quality Control Calibration Verification Check Standards: Eight mid-range verification standards were analyzed throughout the analysis runs. Two of the eight analyses were discarded since the volume of the standard injected for analysis was low due to insufficient sample in the sampling vial. Of the remaining verification standards, the reported results for all analytes of interest were recovered within the acceptance criteria of ±10%, except for a single fluoride value.

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

Client: D. Blanchard
ACL Numbers: 00-0157,162,166,169,172
Analyst: MJ Steele
Charge Code/Project: W53400 / 29953
ASR Number: 5552
Analysis Date: November 09-10, 1999

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)

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proj 29953
Tank 2.12
T2.12.5.2
TF-61
SO_4 removal
screening test
1st test
AN-107
archive

Final Results:

Lab Number	Sample ID	Sample Vol (ml)	TIC ugC/ml	TIC RPD (%)	Sample Wt (g)	TIC ugC/g
00-0157	CA2-a	0.10	2000		0.1190	1680
00-0157 Dup	CA2-a Dup	0.30	1860	7	0.3532	1580
00-0162	CA2-b	0.20	1600		0.2369	1350
00-0162 Dup	CA2-b Dup	0.30	1500	6	0.3520	1280
00-0166	CA2-c	0.30	1440		0.3506	1230
00-0166 Dup	CA2-c Dup	0.20	1440	0	0.2325	1240
00-0169	CA2-d	0.30	650		0.3418	570
00-0169 Dup	CA2-d Dup	0.40	680	5	0.4542	600
00-0172	CA2-e	0.40	180		0.4508	160
00-0172 Dup	CA2-e Dup	0.60	180	n/a	0.6709	160
00-0380 MS	MS % Recovery	0.10	93%		0.1235	93%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
n/a = Not applicable; either sample or duplicate result is <5 times MDL

The analysis of the subject samples submitted under ASR 5552 were performed by 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. Per the ASR, only TIC analyses were performed on these samples.

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

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC) in either solid or liquid form.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

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

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being 101.4% for TIC (no TOC standards were analyzed).

Calibration Blanks: The five calibration blanks run at the beginning and middle of the analysis run were acceptable, averaging 15.5 µgC TIC. However, the calibration blank analyzed at the end of the analysis run was 55 µgC TIC. This high blank appears to result from a memory effect from the high carbon samples analyzed at the end of the run. The end of run calibration blank has not been used to calculate the blank concentration. All samples under this ASR were analyzed at the start or middle of the analysis run and should not be affected by the high end of run blank.

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all values measured above the EQL.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. None of the aqueous samples in this series of sample was prepared as a spike. However, sample 00-0380 analyzed in the same batch had a TIC spike recovery within the 75% to 125% recovery acceptance criteria.

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.
- 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: MW Thomas 11-12-99

Review/Approval by: D R Baldwin **Date** 11-16-99

Archive Information:

Files: ASR 5552 Blanchard.doc	ASR 5552 5568 5582.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

CA1-a

11/11/99

Client : ~~Blanchard~~ Kenneth

Cognizant Scientist: L R Greenwood Date : 11/11/99

Concur : T Trang-Le Date : 11/11/99

Procedure: PNL-ALO-450

RECORDS COPY

proj # 29953
Task 12
T 2.12.5.2
P 1/1
TI-61

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

ALO ID Client ID	Co-60 Error %	SnSb-126 Error %	Sb-125 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Ce-141 Error %	Am-241 Error %
00-0156 CA1-A	4.56E-2 2%	<3.E-4	3.12E-4 55%	2.12E-2 2%	5.02E-3 3%	4.15E-3 5%	<2.E-4	1.84E-3 14%
00-0160 CA5-A	4.23E-2 2%	<3.E-4	<6.E-4	2.08E-2 2%	1.71E-3 6%	1.14E-3 13%	<3.E-4	7.88E-4 42%
00-0162 CA2B	3.83E-2 2%	<3.E-4	<6.E-4	1.90E-2 2%	1.21E-3 9%	9.91E-4 13%	<2.E-4	3.20E-4 57%
00-0165 CA1-C	4.01E-2 2%	<3.E-4	<6.E-4	1.95E-2 2%	1.88E-3 5%	1.54E-3 9%	<2.E-4	9.59E-4 21%
00-0166 CA2-C	3.70E-2 2%	<3.E-4	<6.E-4	1.80E-2 2%	8.85E-4 11%	9.09E-4 16%	<2.E-4	4.09E-4 52%
00-0168 CA1-D	3.47E-2 2%	<3.E-4	<5.E-4	1.63E-2 2%	1.76E-3 6%	1.22E-3 10%	<2.E-4	6.67E-4 24%
00-0169 CA2-D	3.19E-2 2%	<2.E-4	<3.E-4	1.63E-2 2%	6.38E-4 8%	4.12E-4 15%	<1.E-4	4.59E-4 24%
00-0170 CA4-D	3.46E-2 2%	<3.E-4	<6.E-4	1.62E-2 2%	3.43E-3 4%	2.53E-3 6%	<2.E-4	1.50E-3 17%
00-0172 CA2-e	2.69E-2 2%	<4.E-5	<9.E-5	1.43E-2 2%	4.97E-4 4%	3.63E-4 7%	<4.E-5	2.35E-4 16%
00-0176 BA3	5.68E-2 1%	2.40E-4 15%	<5.E-4	2.63E-2 2%	9.50E-3 2%	6.94E-3 4%	<2.E-4	4.45E-3 8%
00-0179 IX2	5.85E-2 1%	2.89E-4 12%	<5.E-4	2.63E-2 2%	1.07E-2 2%	7.73E-3 4%	1.52E-4 42%	4.27E-3 8%

Battelle Pacific Northwest Laboratory CA1-a
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team 3/13/00

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 3/13/00

Concur : T Trang-Le Date : 3/13/00

Procedure: PNL-ALO-450

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

ALO ID Client ID	Co-60 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0157 CA2-A	3.39E-2 2%	1.78E-2 2%	1.29E-3 7%	8.06E-4 12%	4.11E-4 34%

Battelle Pacific Northwest Laboratory CA1-a
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team 3/23/00

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 3/23/00

Concur : T Trang-Le Date : 3/23/00

Procedure: PNL-ALO-476

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

ALO ID Client ID	Sr-90 Error +/-
00-0157 PB CA2-A	<5.E-5
00-0157 CA2-A	4.16E-3 3%
00-0157 DUP CA2-A	4.48E-3 3%
RPD	7%
00-0162 CA2B	3.40E-3 3%
00-0166 CA2-C	1.51E-3 4%
00-0169 CA2-D	2.53E-4 14%
00-0172 CA2-E	3.55E-4 10%
Blank Spike	104%
Matrix Spike	104%
Blank	<5.E-5

APPENDIX B

Appendix B: Test Instruction BNFL-TI-29953-064 and Supporting Analytical Data

PNNL Test Instruction

Document No.: BNFL-TI-29953-64
Rev. No.: 0

Title: Sulfate Removal Studies from AN107 Archived Waste

Work Location: Building 325, various laboratories

Author: SK Fiskum

Use Category Identification: Reference

Effective Date: New
Supersedes Date: New

Identified Hazards:
_X_ Radiological
_X_ Hazardous Materials
___ Physical Hazards
___ Hazardous Environment
___ Other:

Required Reviewers:
_x_ Author
_x_ Technical Reviewer
__ RPL Manager
__ Project Manager
__ RPG Quality Engineer
__ BNFL (not required)

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

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

On-The Job Training Required? ____ Yes or __x__ No

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

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

Approval	Signature	Date
Author	Sandra K. Fiskum Sandra K. Fiskum	10/27/99
Technical Reviewer	DE Kurath Dean E. Kurath	10/27/99

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Applicability

This test instruction outlines 2 tests for sulfate removal from AN-107 Archive material. The first test incorporates an initial carbonate removal using Ca followed by a series of experiments using various ratios of [Ba(NO2)2-H2O] : [SO4]. The second test explores the metathesis of BaCO3 solid to BaSO4 as it is contacted with sulfate from the AN107 Archive material treated with Ca for initial carbonate removal.

The AN107 Archive material is assumed to be composed of the following: 0.041M sulfate, 0.7M carbonate, 0.01M EDTA, 0.0039M HEDTA (the last 3 molarities are assumed based on known initial concentrations and an estimated dilution factor of 2x). Density is assumed to equal 1.22

DRD Reference: 12.2

Schedule Reference: BNFL desires completion of these tests and anion analyses by November 19, 1999.

Justification

Work was requested by BNFL to provide information for conceptual design of sulfate removal system.

Objective

These experiments will help define an optimal method for sulfate removal from tank waste solutions prior to vitrification.

Work Instructions

1. Calcium prestrike 1:1 Ca:CO3

1.1 Determine solution density. Tare a 10-mL volumetric flask. Add AN107 Archive to the 10-mL mark and weigh the solution.

Volumetric flask mass	13.1870 g
Flask + AN107 Archive mass	25.7759
Net AN107 Archive mass	12.5889
Temperature	at 18 °C

Calculated solution density of AN107 Archive
1.25889 g/mL

1.2 Calculate the mass of AN107 required to obtain a 200-mL volume.

200 mL x 1.2589 g/mL = 251.778 g AN107 Archive

1.3 Weigh a 200-mL aliquot of AN107 Archive into a tared 250-mL polyethylene bottle containing a stir bar. 282 SK 10/28/99

Bottle tare mass	41.1853 g.	
AN107 Archive + bottle mass	292.3517 g	
AN107 Archive mass	251.2235 g	199.56 mL.

(A 200-mL sample of AN107 Archive is expected to contain 140 millimoles CO3.)

1.4 Add 28 mL 5M Ca solution with stirring (made previously for TI BNFL-TI-29953-061) to the AN107 Archive sample. (This will provide 140 millimoles Ca.)

AN107 mass + bottle + 5M Ca mass 335.0650 g
Net 5M Ca mass 42.7133 g
Density of 5M Ca solution 1.531 g/mL
Calculated volume of 5M Ca added 27.899 mL.

1.5 Stir the AN107/Ca mixture for nominally 15 minutes and allow to stand for nominally 60 minutes.

1.6 Label and weigh both the lower reservoir and upper reservoir of a filtration apparatus containing a 0.45-µm nylon filter. Have on reserve a second upper reservoir, labeled and weighed.

Lower reservoir mass 50.840 g Reserve lower: 50.6553
Upper reservoir mass 39.1402 g
Reserve upper reservoir 39.1596 g

1.7 Obtain a vacuum gauge meter to measure the house vacuum line and a stopwatch. Measure the house vacuum. 19.8 mm Hg

1.8 **NOTE: This step will require 2 analysts.** Place the filter apparatus into secondary containment. Hook the filter apparatus to the house vacuum. Pour the AN107 Archive/Ca mixture into the upper reservoir of the filter apparatus. Simultaneously start the stopwatch and vacuum, measuring the time it takes to pull about 90% of the filtrate through the filter. Shut off the vacuum and disconnect the hose while shutting off the timer.

Time for 90% volume passage 5 min 23 sec seconds or minutes. Repeat 2 min 38 sec

1.9 Disconnect the lower reservoir from the upper reservoir and measure the mass of filtrate.

Gross filtrate mass 114.9500 g
Net filtrate mass 64.11 g
Calculated mass filtered per second: 0.198 g/sec = 11.9 g/min.

Reserve:
92.7590 g
42.1037 g
0.266 g/sec = 16 g/

1.10 Finish filtering the sample, pulling as much volume of AN107 Archive as practical.

1.11 Disconnect the lower filter chamber apparatus. Save the solids for now in the upper reservoir stored in a plastic bag.

1.12 Weigh the final mass of the filtrate. (It is anticipated 25% of the original sample volume will be consumed by the CaCO3 mass/solids, and that nominally 171 mL filtrate will be obtained.) Combine filtrates into initial reservoir.

Gross filtrate mass 259.9585 g
Net filtrate mass 209.118 g at 1.237 g/ml = 169.05 mL.

1.13 Remove 3-mL filtrate for analysis by IC and TIC, and another 10-mL for analysis by GEA followed by ICP—Sample ID = "CaPS-64" (calcium pre-strike TI –64).

10 mL = 12.3201 g measured into LSC vial

1.14 Prepare 2M Ba(NO2)2-H2O solution. The Ba(NO2)2-xH20 is technical grade and is listed as containing 90% Ba(NO2)2-xH2O. The material also contains 7-8% KClO4. We are assuming there is 1 water of hydration.

Weigh 5.497g Ba(NO2)2-xH2O (247.37 g/mole) -Actual mass 5.4966 g. 2.7411
Tare 10-mL volumetric flask: 16.1053 g 15.9090
Dissolve salt in 10-mL DI water in vol. Flask - Material did not completely dissolve; filter into new 10-ml vol flask
Weigh vol. Flask + $Ba(NO_2)_2$ 25.6498 g.
Net mass Ba solution 13.7163 g
Calculated density 1.3716 g/mL
Jar have: 11.9335g
$KClO_4$ solubility = 0.75g/100cc
Submit 2-mL of the Ba solution for ICP analysis; call samples BaC-64 (barium carrier TI-64).

1.15 Determine the density of the AN107 Archive Ca pre-strike filtrate (PS filtrate). Tare a 10-mL volumetric flask. Add AN107 Archive Ca pre-strike filtrate to the 10-mL mark and weigh the solution.

Volumetric flask mass 11.8337 g
Flask + AN107 Archive pre-strike filtrate 24.2040 g
Net AN107 Archive pre-strike filtrate 12.3703 g
Temperature 19.5 °C

Calculated solution density of AN107 Archive Ca pre-strike filtrate 1.237 g/mL

2 Barium Contacts *The sulfate concentration is expected to be 0.036M in the filtrate.*

2.7 Tare 3 LSC vials with caps on labeled Ba-64-1 (with a stir bar), BaCO3-64-1, BaCO3-64-2. Tare a 200-mL polyethylene bottle, with stir bar, labeled "Ba-64-2."

2.8 Measure BaCO3 into vials BaCO3-64-1 and BaCO3-64-2

			g measured	gross vial mass
BaCO3-64-1	1:1 contact	71 mg BaCO3 (0.36 millimoles BaCO3)	70.6 mg	17.0071 g
BaCO3-64-2	3:1 contact	213 mg BaCO3 (1.08 millimoles BaCO3)	213.8 mg	17.4856 g

Table 1

Sample ID	Description	Vessel tare (g)	Tare + sample (g)	Sample (g)	Gross + Ba(NO2)2	Net Ba(NO2)2 added
Ba-64-1	1:1 Ba:SO4	17.9510	30.2725	12.3215	30.5257g	0.2532 g
Ba-64-2	1.5:1 Ba:SO4	42.5035	166.2357	123.7272	169.9061	3.6704
BaCO3-64-1	1:1 BaCO3:SO4	16.9361	29.3447	12.3376	----------	----------
BaCO3-64-2	3:1 BaCO3:SO4	17.2712	29.8288	12.3432	----------	----------

2.3 Add 10-mL PS filtrate solution into samples Ba-64-1, BaCO3-64-1, and BaCO3-64-2. Record mass.

2.9 Add 100-mL PS filtrate solution into sample Ba-64-2. Record mass.

2.10 Seal samples BaCO3-64-1 and BaCO3-64-2, place into the shaker, and allow to shake for >12 hours on low speed. ON 10/28/99 2:00 p.m.
OFF 11/1/99 8:10 a.m T= 28 °C

2.11 Add 0.18 mL 2M Ba(NO2)2-H2O to sample Ba-64-1. Add ~~1.8~~ 2.7 mL 2M Ba(NO2)2-H2O to Ba-64-2. Stir each solution for nominally 15 minutes and allow to stand for nominally 60 minutes. Record mass. ~ 1.5 mL : 10 mL = solids to total volume or ~15% vol. consumed by settled solids

2.12 Filter Flux of BaSO4

2.7.1 Label and weigh both the lower reservoir and upper reservoir of a filtration apparatus containing a 0.45-µm nylon filter.

Lower reservoir mass 50.6660 g
Upper reservoir mass 39.0323 g.

2.7.2 Obtain a vacuum gauge meter to measure the house vacuum line and a stopwatch. Measure the house vacuum. 19.5 mm Hg

2.7.3 **This step will require 2 analysts**. Hook the filter apparatus to the house vacuum. Pour the filtrate/BaSO4 mixture Ba-64-2 (90-mL volume) into the upper reservoir of the filter apparatus. Simultaneously start the stopwatch and vacuum, measuring the time it takes to pull about 90% of the filtrate through the filter. Shut off the vacuum and disconnect the hose while shutting off the timer.

Time for 90% volume passage 3min, 13 sec seconds or minutes

2.7.4 Disconnect the lower reservoir from the upper reservoir and measure the mass of filtrate.

Gross filtrate mass 117.6448 g
Net filtrate mass 66.9788 g
Calculated mass filtered per second: 0.347 g/sec = ~20.82 g/min.

2.7.5 Finish filtering the sample, pulling as much volume of filtrate through the filter as practical.
final gross mass - 172.4260 Net mass (final) 12 [illegible] = 93.70 [illegible]
2.7.6 Connect a clean lower reservoir to the BaSO4 upper reservoir.

2.7.7 Wash the solids 3 times each with 10-mL 0.01M NaOH. Continue to pull vacuum until the solids appear dry.

2.7.8 Transfer the solids to a tared glass vial, weigh. Dry the solids at 105°C overnight, weigh.

Vial tare 17.0707
Wet solids + tare 19.9367 ~ 90% transference from filter apparatus
Dry solids + tare 18.0826

2.7.9 Sample the solids for ICP and GEA analyses.

2.7 Allow the 10-mL volume contact sample solutions to settle overnight, or centrifuge at 1500 rpm for 3 minutes. Estimate the solids content in each vial.

Ba-64-1 ~ 10 % or less
BaCO3-64-1 ~ 10 % or less same vol. as starting
BaCO3-64-2 ~ 10 % or less same vol. as starting

2.8 Filter the supernatant solutions through a 0.45-µm filter/syringe apparatus, collecting the filtrate in clean LSC vials labeled Ba64-1 supernatant, BaCO3-64-1 supernatant, BaCO3-64-2 supernatant.

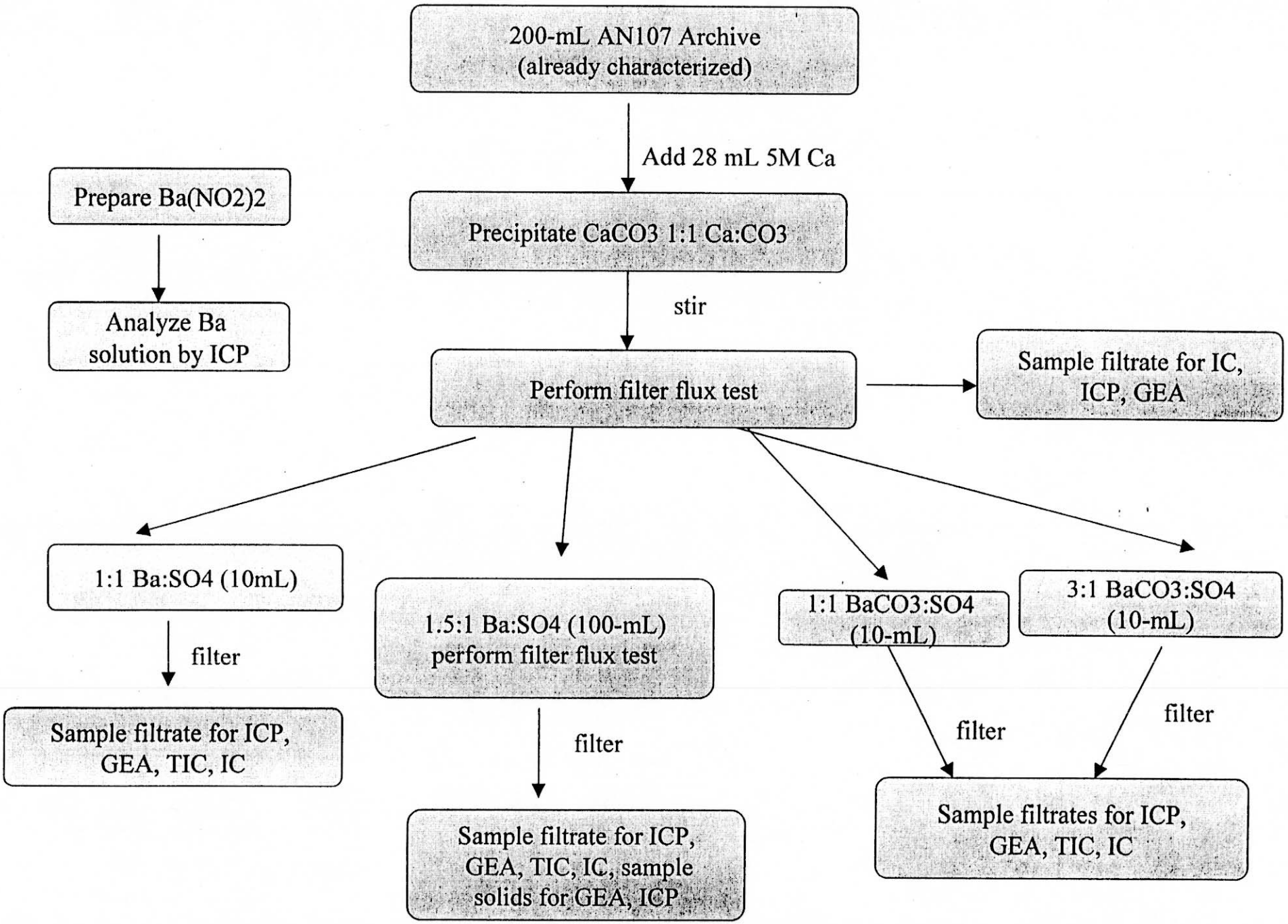
2.9 Sample all the filtrates for IC, ICP, GEA, and TIC and submit to the laboratory for analysis.

BA-64-2 density check

10-ml vial tare	12.1476g
gross	24.4836g
Net	12.3360g

δ = 1.234 g/mL

12.2407g → GEA analysis


200-mL AN107 Archive
(already characterized)
Add 28 mL 5M Ca
Prepare Ba(NO2)2
Precipitate CaCO3 1:1 Ca:CO3
Analyze Ba
solution by ICP
stir
Perform filter flux test
Sample filtrate for IC,
ICP, GEA
1:1 Ba:SO4 (10mL)
1.5:1 Ba:SO4 (100-mL)
perform filter flux test
1:1 BaCO3:SO4
(10-mL)
3:1 BaCO3:SO4
(10-mL)
filter
filter
filter
filter
Sample filtrate for ICP,
GEA, TIC, IC
Sample filtrate for ICP,
GEA, TIC, IC, sample
solids for GEA, ICP
Sample filtrates for ICP,
GEA, TIC, IC

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: S. Fiskum

ACL Number(s): 00-0267 through 00-0273

Client ID: "2M Ba" through "BaCO3-64-2"

ASR Number: 5568

Total Samples: 7

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

Analyst: D.R. Sanders

Analysis Date (Filename): 11-05-99 (A0551) & 11-19-99 (A0558)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry W. Wagner 1-13-00
Reviewed by

M W Thur 1-14-00
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

One radioactive solid sample, **Ba Sulfate Solids** (ACL# 00-0274) is missing from this report. Approximately 0.12g of the solid was processed using PNL-ALO-129 acid digestion procedure but failed to completely dissolve. An attempt to dissolve the sample using a fusion procedure is scheduled but has not been completed at this time.

One radioactive aqueous sample, **2M Ba** (ACL# 00-0267) was analyzed by ICPAES after the sample was dilute 10201 fold using 0.32N nitric acid because of the high concentration of barium present in the sample. The sample was first diluted 101-fold by adding 0.100ml of sample to 10.0 ml of 0.32N nitric acid, then 0.100 ml of the 101-fold diluted sample was added to 10.0 ml 0.32N nitric acid and analyzed by ICPAES. Final analytical dilution of the sample is 10201 fold. Barium concentration results are calculated as shown below and reported as molarity of barium (mols per liter). Preliminary results were sent by e-mail to the client earlier indicating a barium concentration of 1.86M. The final results in this report are the same. Concentration uncertainty is approximately ± 10%. Barium concentration was corrected for calibration drift by multiplying the ratio of a single element barium standard ("true" value) to the concentration of the standard as measured by ICP. The single-element barium standard concentration of 20 µg/ml was similar in concentration to the 10201-fold diluted sample.

Barium molarity is calculated as follows:

$[Ba] = (\mu g/ml)_{ICP} * \{(\mu g/ml\ std)_{TRUE} / (\mu g/ml\ std)_{MEASURED}\} *$
1.0E-03 (conversion factor to convert µg/ml to g/liter) /
gram formula wt of barium (137.34 g/L) =
mols Ba

[Ba] = 276000 µg/ml * [(20 µg/ml "True")/(21.6 µg/ml "measured")] * 1.0E-03) / 137.34 g/L = 1.86M Ba

Six additional radioactive aqueous samples, **Ca PS-64** through **BaCO3-64-2** (ACL# 00-0268 through 00-0273), prepared by SRPL using PNL-ALO-128 acid digestion procedure were analyzed by ICPAES. Approximately 1ml-sample aliquots (weighed) were digested and brought to a final volume of about 10ml (weighed). Processing factor was determined using weight ratio of final volume weight divided by sample aliquot weight. Concentration units reported are in µg/ml. Analytes of interest are listed in Table 6.1 Analytical Requirements ("Filtrate, Wash Solutions ... ") TSP-W375-99-00016 Rev. 2 include: Al, Ag, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Se, Si, U, and Zn.

Measurement results reported have been corrected for preparation and analytical dilution. Weights and volumes used have been recorded on bench sheets and are included with this report.

1/13/00

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Aqueous sample) All results are within tolerance limit of ≤ 10% after correcting for dilution except calcium in sample **Ba-64-1** and **BaCO3-64-1**. Calcium varied by 30% and 22% respectively, between the 25-fold and 5-fold dilutions. Calcium in three other samples diluted similarly were well within tolerance limit. Possibly the two samples that were outside the tolerance limit contained a small amount of particulate material high in calcium.

Duplicate RPD (Relative Percent Difference):

(Aqueous sample) None prepared.

Post-Spiked Samples (Group A):

(Aqueous sample) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Aqueous sample) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Aqueous sample) None.

Matrix Spiked Sample:

(Aqueous sample) None.

Quality Control Check Standards (aqueous sample):

Concentration for all analytes of interest is within tolerance limit of ± 10% accuracy except for a slightly high recovery for potassium in the standard QC_SSTMCV. One of two measurement results for potassium was 18% too high. The other measurement was within tolerance. A single element standard of potassium at 100 µg/ml was measured twice during the ICP run and the measurement results (102, 102 µg/ml) were both within acceptable tolerance limits.

1/13/00

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

High Calibration Standard Check (aqueous sample):
Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy except for potassium (18% too high). A single element standard of potassium at 100 µg/ml was measured twice during the ICP run and the measurement results (102, 102 µg/ml) were both within acceptable tolerance limits.

Process Blank:
(Aqueous sample) None.

Laboratory Control Standard (LCS):
(Aqueous sample) None.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

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

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

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	38.8 00-0268 @5 CA PS-64 11/19/99 ug/mL	40.0 00-0269 @5 AN107 Archive 11/19/99 ug/mL	39.6 00-0270 @5 Ba-64-1 11/19/99 ug/mL	39.9 00-0271 @5 Ba-64-2 11/19/99 ug/mL	39.9 00-0272 @5 $BaCO_3$-64-1 11/19/99 ug/mL
0.025	Ag	–	–	–	–	–
0.060	Al	64.8	127	64.0	62.6	65.6
0.250	As	–	–	–	–	–
0.050	B	[6.8]	[9.8]	[6.9]	[6.7]	[7.9]
0.010	Ba	–	–	35.2	101	7.92
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	789	163	973	1,000	788
0.015	Cd	22.1	28.4	21.8	21.2	21.7
0.200	Ce	–	–	–	–	–
0.050	Co	–	–	–	–	–
0.020	Cr	29.8	35.3	8.06	[5.9]	29.7
0.025	Cu	10.3	12.2	9.98	[9.5]	[9.8]
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[4.8]	[9.3]	[3.7]	[3.2]	[3.8]
2.000	K	[620]	[720]	[690]	[680]	[590]
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	[2.1]	[3.5]	–	–	–
0.050	Mo	[12]	[15]	[12]	[12]	[13]
0.150	Na	83,500	102,000	86,600	87,300	90,500
0.100	Nd	–	–	–	–	–
0.030	Ni	195	228	193	189	196
0.100	P	43.6	201	44.1	42.1	44.2
0.100	Pb	43.0	91.7	42.5	41.2	42.7
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	–	–	–	–
1.500	Sn	–	–	–	–	–
0.015	Sr	[1.4]	123	[1.8]	[1.6]	[1.0]
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	23.2	[9.0]	[5.9]	[4.6]	[5.4]
0.050	Zr	–	[2.3]	–	–	–

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= 39.9 ALO#= 00-0273 @5 Client ID= BaCO3-64-2 11/19/99 ug/mL				
0.025	Ag	–	–	–	–	–
0.060	Al	64.7	–	–	–	–
0.250	As	–	–	–	–	–
0.050	B	[8.2]	–	–	–	–
0.010	Ba	8.30	–	–	–	–
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	750	–	–	–	–
0.015	Cd	21.6	–	–	–	–
0.200	Ce	–	–	–	–	–
0.050	Co	–	–	–	–	–
0.020	Cr	29.2	–	–	–	–
0.025	Cu	[9.8]	–	–	–	–
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[3.7]	–	–	–	–
2.000	K	[580]	–	–	–	–
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	–	–	–	–
0.050	Mo	[13]	–	–	–	–
0.150	Na	89,800	–	–	–	–
0.100	Nd	–	–	–	–	–
0.030	Ni	195	–	–	–	–
0.100	P	44.6	–	–	–	–
0.100	Pb	42.5	–	–	–	–
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	–	–	–	–
1.500	Sn	–	–	–	–	–
0.015	Sr	[0.96]	–	–	–	–
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	[3.9]	–	–	–	–
0.050	Zr	–	–	–	–	–

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	10201.0 / 00-0267 @10201 / ~2M Ba / 11/5/99 / ug/mL	1.0 / Ba $20 PM / 11/5/99 / ug/mL		
0.025	Ag	--	--	--	--
0.060	Al	--	--	--	--
0.250	As	--	--	--	--
0.050	B	[3,700]	--	--	--
0.010	Ba	276,000	21.6	--	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.250	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	--	--	--
0.025	Cu	--	0.615	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	--	--	--	--
2.000	K	--	--	--	--
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	--	--	--
0.150	Na	[5,000]	--	--	--
0.100	Nd	--	--	--	--
0.030	Ni	--	--	--	--
0.100	P	--	--	--	--
0.100	Pb	--	--	--	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	--	--	--	--
1.500	Sn	--	--	--	--
0.015	Sr	[300]	--	--	--
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	--	--	--	--
0.050	Zr	--	--	--	--

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

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

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

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

Client: S. Fiskum
ACL Numbers: 00-0268 to 00-0273
Analyst: MJ Steele

Charge Code/Project: W53400 / 29953
ASR Number: 5568
Analysis Date: November 01-03, 1999

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.

Final Results:

Lab ID	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
00-0268	Ca PS-64	1,300	< 1000	29,400	< 1000	205,000	< 2000	4,600	< 2000
00-0268 Rep	Ca PS-64 Rep	< 1000	< 1000	29,000	< 1000	203,000	< 2000	4,700	< 2000
	RPD	n/a	n/a	1%	n/a	1%	n/a	0%	n/a
00-0269	AN1017 Archive	1,200	< 1000	33,700	< 1000	136,000	< 2000	5,100	2,000
00-0270	Ba-64-1	< 1000	< 1000	32,800	< 1000	204,000	< 2000	< 2000	< 2000
00-0271	Ba-64-2	< 1000	< 1000	34,400	< 1000	200,000	< 2000	< 2000	< 2000
00-0272	$BaCO_3$-64-1	< 1000	< 1000	29,400	< 1000	203,000	< 2000	4,600	< 2000
00-0273	$BaCO_3$-64-2	< 1000	< 1000	29,500	< 1000	206,000	< 2000	4,600	< 2000
	00-0273 MS Rec	102%	120%	107%	110%	115%	113%	112%	116%
	Blank Spike Rec	100%	96%	101%	105%	104%	102%	104%	103%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS Rec = Matrix Spike Standard % recovery
Blank Spike Rec = Blank Spike Standard % recovery

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation at 4,000-fold to ensure that all anions were within the calibration range. Due to the very high nitrate concentrations, lower dilutions could not be analyzed. Due to this high dilution, the sulfate results are all near the Estimated Quantitation Level (EQL).

Q.C. Comments:

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the relative percent differences (RPD) between replicates are well within the acceptance criteria of 20% for all anions measured above the EQL.

Matrix Spike: A matrix spike was prepared and measured for sample $BaCO_3$-64-2. The spike recoveries for all anions are within the 75% to 125% recovery acceptance criteria.

Blank Spike: The blank spike is used as the laboratory control sample and recovered within the acceptance criteria of 80% to 120%.

System Blank/Processing Blanks: Approximately ten system blanks were process during the analysis of the samples. With the exception of only single nitrate value, no anions were detected above reportable concentrations in the system blanks. Since the nitrate results are very high, this single QC failure does not affect the reported nitrate results.

Quality Control Calibration Verification Check Standards: Approximately ten mid-range verification standards were analyzed throughout the analysis runs. Except for a single phosphate value, the reported results for all analytes of interest were recovered within the acceptance criteria of ±10% for the verification standard. The one phosphate result recovered at +11% above the true value. This single phosphate failure has no impact on the reported results.

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.

Analyst: M J Steele

Approval: M W Thim **Date** 11-12-99

Archive Information:

Files: ASR 5568 Fiskum.doc	ASR 5463 5533 -36 -68 -71.xls

RECORD COPY

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

Client: S. Fiskum
Charge Code/Project: W53400 / 29953
ACL Numbers: 00-0268 to 00-0273
ASR Number: 5568
Analyst: MJ Steele
Analysis Date: November 09-10, 1999

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).

Final Results:

Lab Number	Sample ID	Sample Vol (ml)	TIC ugC/ml	TIC RPD (%)	Sample Wt (g)	TIC ugC/g
00-0268	CA PS-64	0.50	2800		0.6160	2280
00-0268 Dup	CA PS-64 Dup	0.50	2100	29	0.6146	1710
00-0269	AN107 Archive	0.50	8200		0.6212	6600
00-0269 Dup	AN107 Archive	0.10	8670	6	0.1314	6600
00-0270	Ba-64-1	0.20	2680		0.2451	2190
00-0270 Dup	Ba-64-1 Dup	0.50	2650	1	0.6080	2180
00-0271	Ba-64-2	0.50	2570		0.6101	2100
00-0271 Dup	Ba-64-2 Dup	0.50	2630	3	0.6127	2150
00-0272	BaCO3 64-1	0.50	2980		0.6123	2430
00-0272 Dup	BaCO3 64-1 Dup	0.50	2990	1	0.6120	2450
00-0273	BaCO3 64-2	0.50	2990		0.6093	2450
00-0273 Dup	BaCO3 64-2 Dup	0.50	2960	1	0.6023	2450
00-0380 MS	MS % Recovery	0.10	**93%**		0.1235	**93%**

RPD = Relative Percent Difference (between sample and duplicate/replicate)

The analysis of the subject samples submitted under ASR 5568 were performed by 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. Per the ASR, only TIC analyses were performed on these samples.

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

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC) in either solid or liquid form.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

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

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being 101.4% for TIC (no TOC standards were analyzed).

Calibration Blanks: The five calibration blanks run at the beginning and middle of the analysis run were acceptable, averaging 15.5 µgC TIC. However, the calibration blank analyzed at the end of the analysis run was 55 µgC TIC. This high blank appears to result from a memory effect from the high carbon samples analyzed at the end of the run. The end of run calibration blank has not been used to calculate the blank concentration. Based on the system run, this high blank appears to affect Samples 00-0271 through 00-0273. Using the higher blank value for the carbon concentration calculation lowers the reported results about 3%; the reported results have not been adjusted for the higher blank.

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all values measured above the EQL, except CA2-a, which has a 29% RPD.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. None of the aqueous samples in this series of sample was prepared as a spike. However, sample 00-0380 analyzed in the same batch had a TIC spike recovery within the 75% to 125% recovery acceptance criteria.

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.
- 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: MO Thurston 12-99

Review/Approval by: [signature] **Date** 11-16-99

Archive Information:

Files: ASR 5568 Fiskum.doc	ASR 5552 5568 5582.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # **5568.01**

WP# **W53400**

File: L:\radchem\hydroxide\asr5568-rr
Analysis Date: **12/3/1999**
Print Date: 12/6/99

Hydroxide and Alkalinity Determination
Governing Procedures: **PNL-ALO-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843**
Lab Loc. 525

Analyst: [signature]
Reviewer: [signature] 12-6-99

Titrant	Molarity
HCl	**0.2034**
pH 7.0 reading =	6.98

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-00269	**AN107 Archive**		0.300	0.3779	1.260	3	12.124	1.271	10.914	0.259	0.86	
00-00269	**AN107 Archive**	Replicate 1	0.300	0.3812	1.271	4	12.189	1.294	10.962	0.263	0.88	1.79%
00-00269	**AN107 Archive**	Replicate 2	0.300	0.3802	1.267	5	12.135	1.279	10.940	0.260	0.87	1.17%
QC Data:												
Reag. Blk.			5.00			1	4.164				**OH % Recovery**	
Standard 1	0.1018 N NaOH		5.000	5.0081	1.002	2	12.213	2.236	10.629	0.4548	**89.4%**	Std 1
00-0269MS	An-107 + 2mL 0.1N NaOH		0.100	0.1268	1.268	6	11.993	1.366	10.738	0.278	**93.8%**	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Performance checks

Balance # 360--01-06-037

		Vol.	Wt.
Pipet #	H30762	5.00	5.0049
Pipet #	223382	0.300	0.2998
Pipet #	223382	0.300	0.2997
Pipet #	223382	0.300	0.2983

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proj # 29953
Task 2.12
T2.12.5.2
T+-64

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5568.01
WP# W53400

File: L:\radchem\hydroxide\asr5568-rr
Analysis Date: 12/3/1999
Print Date: 12/6/1999

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: RG Swanson 12/6/99
Reviewer: JR Greenwood 12-6-99

Titrant	Molarity
HCl	0.2034

				CO3						HCO3				
				2nd Equivalence						3rd Equivalence				
RPG #	Sample ID		Sample Vol. (mL)	Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD		Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-00269	AN107 Archive	0	0.300	2.447	7.844	0.239	0.797			3.665	4.927	0.248	0.83	
00-00269	Sample ID	Replicate	0.300	2.481	7.914	0.241	0.805	0.93%		3.696	4.899	0.247	0.82	0.2%
00-00269	AN107 Archive	Replicate	0.300	2.483	7.769	0.245	0.816	1.42%		3.701	4.866	0.248	0.83	0.2%
							CO3 % Recovered							
Standard 1	0.1018 N NaOH		5.000	2.461	8.056			sample		2.664	3.86			
00-0269MS	An-107 + 2mL 0.1N NaOH		0.100	1.838	8.068	0.096	119.0%	sample		2.334	4.82	0.1009	122.2%	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0..100-mL of sample for each matrix spilke.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Chem Rec_51a

Prep date: 04/18/1999

Preparation of Standardized 0.2 M HCl

WP# K51300

Standardized O.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 --prep.date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole -- Barcode # 52232 --- (see below verification check).
The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.
Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H2O.
20 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalien endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity veification

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

Titration Id.	aliquot of sample	Vol. of **0.1018M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	20.00	39.88	0.2030	
2	20.00	39.92	0.2032	
3	20.00	40.04	0.2038	
		Ave Molarity HCl =	**0.2034**	0.00042

Analyst/Date 12/3/99

Battelle Pacific Northwest Laboratory 00-0268
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team 11/9/1999

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 11/9/99

Concur : T Trang-Le Date : 11/9/99

Procedure: PNL-ALO-450

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

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0268 Ca Ps-64	5.48E-2 2%	<3.E-4	<1.E-4	1.81E-2 2%	9.62E-3 2%	7.12E-3 3%	2.90E-3 10%
00-0269 An107 Archive	6.44E-2 2%	4.12E-4 28%	3.12E-4 8%	2.02E-2 2%	2.66E-2 2%	1.89E-2 3%	9.72E-3 9%
00-0270 Ba-64-1	5.57E-2 2%	<4.E-4	<2.E-4	1.82E-2 2%	8.30E-3 2%	5.64E-3 4%	3.16E-3 12%
00-0271 Ba-64-2	5,30E-2 2%	<2.E-4	<7.E-5	1.78E-2 2%	6.70E-3 2%	4.89E-3 3%	2.31E-3 10%
00-0272 BaCO3-64-1	5.66E-2 2%	<3.E-4	<2.E-4	1.86E-2 2%	8.98E-3 2%	6.66E-3 3%	2.90E-3 7%
00-0273 BaCO3-64-2	5.73E-2 2%	<3.E-4	<2.E-4	1.88E-2 2%	9.10E-3 2%	6.38E-3 3%	3.31E-3 6%



proj # 29953
task 2.12
T2.12.5.2
TI-64

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

00-0268

12/20/1999

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 12/20/99

Concur : C Soderquist Date : 12-20-99

Measured Activities (uCi/ml) with 1-σ error

ALO ID Client ID	Sr-90 Error %
00-0268 Ca Ps-64	1.05E-2 6%
00-0269 An107 Archive	1.07E+0 4%
00-0270 Ba-64-1	6.82E-3 8%
00-0271 Ba-64-2	4.55E-3 10%
00-0271 DUP Ba-64-2	4.92E-3 10%
RPD	8%
Blank	<7.E-4
Blank Spike	114%
Sample Spike	117%

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TI-64

APPENDIX C

Appendix C: Test Instruction BNFL-TI-29953-071 and Supporting Analytical Data

PNNL Test Instruction

Document No.: BNFL-TI-29953-071
Rev. No.: 0

Title: Sulfate Removal from 650-mL of Previously Treated AN107 DF

Work Location: Building 325, various laboratories

Page 1 of 11

Author: SK Fiskum

Use Category Identification: Reference

Effective Date: New
Supersedes Date: New

Identified Hazards:
_X_ Radiological
_X_ Hazardous Materials
__ Physical Hazards
__ Hazardous Environment
__ Other:

Required Reviewers:
_x_ Author
_x_ Technical Reviewer
__ RPL Manager
__ Project Manager
__ RPG Quality Engineer
__ BNFL (not required)

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

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

On-The Job Training Required? ____ Yes or _x_ No

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

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

Approval	Signature	Date
Author	Sandra K. Fiskum Sandra K. Fiskum	11/15/99
Technical Reviewer	Richard T. Hallen Richard T. Hallen	11/15/99

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Applicability

This test instruction delineates the sulfate removal from a large aliquot of AN107DF for subsequent testing in glass vitrification. The material will be received from the Tc removal ion exchange run. Characterization of the composite material resulted in 0.66M CO3, 0.041M SO4, and 4.97M Na.

Additional testing will be conducted on the product materials to verify processing acceptablitiy. These include such parameters as filter flux testing, TCLP analysis on the precipitates, particle size analysis, as well as chemical and radionuclide compositions.

DRD Reference: 12.2.6

Schedule Reference: BNFL desires completion of this test and analyses by November 30, 1999. The product needs to be delivered to the vitrification personnel by December 3.

Justification

Work was requested by BNFL to provide information for conceptual design of sulfate removal system.

Work Instructions

1.0 Pre-strike

1.1 Measure the density of the AN107 post Tc IX composite material.

Volumetric flask tare	11.7613 g	volume 10	mL
Gross mass	24.0056 g		
Calculated density	1.2244 g/mL	(1.232 g/mL expected)	
Temperature	21°C		
Balance ID	360-06-01-026		

1.2 Aliquot 10-mL sample for radiochemical analysis (GEA, Sr-90, Tc-99, Pu, Am/Cm total alpha, Np-237), 4-mL for ICP digestion and prep, 3-mL for Hg, and 3-mL for TIC/TOC, and IC, 2-mL for NH3. (Composite was already submitted for IC, ICP, TIC, however, I want a duplicate submission for highest accuracy.) These values will comprise baseline concentrations. These samples are labeled TI-71-AN107.
TI-71-AN107(GEA) tare 16.7889 g gross mass 28.9640 g net mass 12.1751 g

1.3 Prepare the following reagents:

100-mL 5M Ca(NO3)2-4H2O (FW 236.15 g/mole)
Ca(NO3)2-4H2O mass 118.0759 g (target mass 118.075g Ca(NO3)2-4H2O) Aldrich, 99%, ACS reagent grade
100-mL volumetric tare 62.5304 g
Gross mass tare + 5M Ca(NO3)2 215.2130 g
Net mass solution 152.6826 g
Calculated density 1.5268 g/mL
Balance ID 360-06-01-038

More vol. was needed, I added more Ca from previously made 5M Ca solution.

25-mL 2M Ba(NO2)2-H2O (FW 247.37 g/mole)
Ba(NO2)2-H2O mass 12.3602 g (target mass is 12.3685g Ba(NO2)2-H2O)
Aija Acars

25-mL volumetric tare 21.8942 g
Gross mass tare + 2M Ba(NO2)2-H2O 55.5730 g
Net mass solution 33.6788 g
Calculated density 1.3472 g/mL
Temperature 20 °C
Balance ID 360-06-01-038

Not all solids dissolved. V filtered solution and analyzed. Ba = [illegible] M s

See additional analysis comment sheet

1.3 Clean a 1-L polyethylene bottle, dry and tare the bottle with a 1.5-inch magnetic stir bar.

Tare mass of bottle 114.6 g Balance: Acculab V-4800

1.5 Calculate the mass of a 630-mL volume of AN107 waste
630 mL * 1.2317 g/mL = 775.97 g

1.6 Weigh AN107 into the flask until the appropriate volume is reached.
Final mass (gross) 1014.1 g
Net AN107 mass 899.5 g = 734.6 mL

While stirring, add ~~91.5~~ 107 mL 5M Ca(NO3)2-4H2O to the waste to achieve a 1.1:1 Ca:CO3 ratio. Record the start time and end time for Ca addition. Continue stirring for 10 minutes. Measure the final mass.
Start time 10:10 End time 10:20
Gross solution mass 1177.5 g
Net 5M Ca added 163.4 g Calculated volume 107.0 mL

1.7 Allow the solution to ~~sit~~ stir for at least 60 minutes.

1.8 Re-suspend solids and pour entire contents into a 1-L graduated cylinder. Record the total volume. At 10-min intervals, record the volume of the settled precipitate, also measure centimeters above precipitate of clear supernatant.

T = 19.9 °C

Time	Precipitate volume (mL)	Supernatant (cm above ppt)
T_0		
11:34:30	860	0
11:44:30	855	2 mm
11:54:30	855	
12:04:30	852.5	3
12:14:30	~~860~~ 850	3.5
12:24:30	850	
12:34:30	848	4
12:44:30	846	5 mm
12:54:30	845	5 first picture
1:04:30	840	7
1:14:30	839	7
1:24:30	838	
1:34:30	337	7

1.9 Mix the solution, re-suspending the solids.

1.10 Sub-sample 10-mL into a tared 10-mL volumetric flask and determine the solution density. From this material sub-sample 8-mL into a vial and submit for viscosity testing.

Use the remaining 2-mL to determine insoluble solids (see section 7.0). Aliquot an additional 2-mL for particle size analysis.

Volumetric flask tare 12.0497 g
Gross mass 24.4627 g
Net solution mass 12.4130 g
Calculated density 1.2413 g/mL
Temperature 20 °C

2.0 CaCO3 Filter Flux

2.1 Label and weigh both the lower reservoir and upper reservoir of a 1-L filtration apparatus containing a 0.45-µm nylon filter. Have on reserve a second upper and lower reservoir, labeled and weighed.

Lower reservoir mass 125.2 g (AN107 CA1)
Upper reservoir mass 88.4 g
Reserve lower reservoir 124.6 g (AN107 CA2)
Reserve upper reservoir 89.0 g

2.2 Obtain a vacuum gauge meter to measure the house vacuum line and a stopwatch. Measure the house vacuum.

19.5 mm Hg

2.3 **NOTE: This step will require 2 analysts**. Place the filter apparatus into secondary containment. Hook the filter apparatus to the house vacuum. Pour the AN107 /Ca mixture into the upper reservoir of the filter apparatus. Record the start time while starting vacuum, measuring the time it takes to pull all of the filtrate through the filter. Shut off the vacuum and disconnect the hose while noting the end time. Due to the large sample volume the sample was processed through 2 filter units.

AN107CA1 Start time 13:54 End time 14:31

AN107CA2 gross mass 597.4g Net mass ~~482.8g~~ 480.2g Start 2:37 End 3:17

Hose 117.2 → 2.4 Disconnect the lower reservoir from the upper reservoir and measure the mass of filtrate.

Gross filtrate mass 803.8 g
Net filtrate mass 678.6 g
Calculated mass filtered per second: _____ g/sec

2.5 Remeasure the house vacuum.

19.5 mm Hg

3.0 Final filtration

3.1 Finish filtering the sample, pulling as much volume of AN107 as practical. Use AN107 filtrate to rinse the 1-L graduated cylinder to quantitatively (as much as possible) remove residual precipitate.

3.2 Disconnect the lower filter chamber apparatus.

3.3 Weigh the final mass of the filtrate. (It is anticipated 25% of the original sample volume will be consumed by the CaCO3 mass/solids, and that nominally 580-mL filtrate will be obtained.)

Gross filtrate mass 803.8 g
Net filtrate mass 678.6 g = 561.3 mL (see § step 3.8)

3.4 Weigh the gross filter chamber mass, determine the net wet CaCO3 precipitate weight.

AN107 CA1 (1)
Gross filter mass 277.4 g
Net wet ppt mass 189.0 g
After sampling 275.9 g Net: 187.5g

AN107 CA2
~~250.1~~ g 261.1
(BR) ~~161.1~~ g 172.1
11-16-99

After sampling
~~260.5~~
259.7
Net: 170.7

* 3.5 Determine the approximate volume of precipitate. Calculate the quantity of wash needed (2:1 ratio wash volume to precipitate volume). Place a CLEAN, tared lower reservoir below the filter. Shut off the vacuum and add the wash solution to the upper reservoir. Allow the wash solution to contact the ppt. for 10 minutes, then pull the vacuum (note vacuum start time and end time). Save the wash solution and label it as TIxx-CaPS-NaOH-wash1.

stirred solids to break them up.

Precipitate volume: 150 @ mL Added wash volume 300 @ mL
Reservoir tare 124.6 g gross mass 802.3 g net solution 677.7 g
Start time 4:17 pm End time 4:52
AN107 CA1 upper 247.9 g gross 159.5 g Net
AN107 CA2 8:13 a.m. 8:40 stop

Gross 220.9
AN107 131.9

3.6 Wash the solids again with 2:1 volume of 0.01M NaOH:ppt volume collecting the wash in another CLEAN lower reservoir (note start and end time). Save the wash solution and label it as TIxx-CaPS-NaOH wash2.

Added wash volume ~~300~~ mL to 1st (estimated solids volume ~150 mL) 200 mL to 2nd (estimated solids volume ~100 mL)
Reservoir tare 124.6 g gross mass 675.7 g net solution 551.1 g
Start time 0855 (#1) / 0923 (#2) End time 0925 (#1) / 0953
Reweigh upper reservoir AN107 CA1 = 219.9 g net wet ppt. mass CA1 131.5 g
AN107 CA2 = 211.9 CA2 122.9

3.7 Remove a portion of wet precipitate (CaCO3) and cap tightly in a tared 2-dram vial. Suspend in 2-mL of 0.01M NaOH and submit for particle size analysis. Remove another portion of the precipitate and submit for XRD analysis. Remove 0.1g in triplicate into tared 40-mL vials, determine net mass, and submit for Hg analysis. Remove another nominal 2-g aliquot into a tared glass vial, dry at 105°C to constant weight and determine percent solids. Submit dried solids for digestion, ICP, TIC/TOC, and radchem analysis. Submit wet solids for NH3 analysis, in duplicate. Label all samples TI-71-CaCO3-1.

Particle size: vial tare 16.8337 g gross mass 17.6843 g net mass 0.8506 g

TI-71-CaCO3-1 Hg1 tare 25.6751 g gross mass 25.8012 g net mass 0.1261 g
TI-71-CaCO3-1 Hg2 tare 25.4961 g gross mass 25.7064 g net mass 0.2103 g
TI-71-CaCO3-1 Hg tare 25.6746 g gross mass 25.7834 g net mass 0.1088 g

Reweigh upper reservoir AN107 CA1 216.4 g net wet ppt mass AN107 CA1 128.0 g
AN107 CA2 208.3 AN107 CA2 119.3

CaCO3-1
Vial tare mass 16.8389 g
Gross mass before drying 19.9411 g Net wet mass 3.1022 g
Mass 1 after drying 19.5924 g Net dry mass 0.7535 g
Mass 2 after drying 17.5886 g Net dry mass 0.7497 g
Calculated % solids 24.00 % g/g 0.7444
17.5833
Mass 3 2.5 days drying

* $CaCO_3$ ppt:
tare 16.7331
gross 19.6669
Net 2.9338g

NH_3 tare 16.8648g gross 17.3630 Net 0.4982

XRD tare 16.8160g gross 17.6146 Net 0.7986

3.8 Remove 10-mL AN107 Ca precipitated filtrate solution into a tared volumetric flask. Determine the density and submit the 10-mL for radchem analysis (GEA, Sr-90, Tc-99, total alpha, Np-237, Pu, Am/Cm); remove 4-mL for acid digestion and ICP analysis; remove 3-mL for TIC/TOC, and IC analysis; remove 3-mL for NH3, and remove 3-mL for Hg analysis. Label samples as TIxx-CaPS-FILTRATE.

Volumetric flask tare 12.0449 g
Gross mass 24.1344 g
Net solution mass 12.0895 g
Calculated density 1.2089 g/mL
Temperature 20 °C

after sampling — Radchem Vial tare 16.9226 g gross mass 28.9493 g net mass 12.0267 g
New gross mass AN107 CaPS filtrate 767.6g net = 642.4 g = 531.4 mL

3.9 Remove aliquots of each of the 0.01M NaOH wash solutions (10-mL for radchem, 5-mL ICP, 3-mL IC/TIC/TOC, 3-mL for Hg, 3-mL for NH3—prorate as necessary). Determine density.

	First wash		Second wash
Volumetric flask tare	11.6804 g	(10-mL)	12.0564 g
Gross mass	22.3567 g		22.1965 g
Net solution mass	10.6763 g		10.1401 g
Calculated density	1.0676 g/mL		1.0140 g/mL
Temperature	18.5 °C	360·06-01-026 balance M+TE	

Radchem: 10.6178g — Radchem: 10.0861g

3.10 Wash the CaCO3 precipitate with 2:1 volume ratio solution:ppt with 1M HNO3 collecting the filtrate in a CLEAN, tared reservoir as defined in step 3.5. Weigh the upper reservoir to determine net ppt loss.

Volume added 1M HNO3 200 (each) mL — Estimate after 1st 1M HNO3 wash 50mL in each

cloudy solution → Lower reservoir tare 125.3 g gross mass 642.8 g net solution 517.5 g
Upper reservoir gross mass AN107 CA-1 = 161.1 g net ppt mass AN107 CA-1 72.7 g
AN107 CA-2 = 136.3 AN107 CA-2 67.3 g
Start time 11:03 End time 11:13
AN107 CA1 x 50mL 11:35 Start for AN107CA2 end 11:45

3.11 Determine remaining ppt volume. Wash the CaCO3 precipitate again with a 2:1 volume ratio of 1M HNO3 collecting the filtrate into another CLEAN tared reservoir as defined in step 3.5. Weigh the upper reservoir and determine ppt. loss.

Volume ppt. 50mL (each) mL 1M HNO3 wash 100mL (ea) mL
Lower reservoir tare 124.4 g gross mass 388.6 g net solution 264.2 g
Upper reservoir gross mass CA1 137.6 CA2 134.0 g net ppt mass CA1 49.2 CA2 42.0 g
Start time AN107 CA1 = 12:13 AN107 CA2 = 12:34 End time AN107 CA1 = 12:18 AN107 CA2 = 12:38

3.12 Determine the remaining ppt. volume. Wash the CaCO3 precipitate again with a 3:1 volume DIW:ppt collecting the filtrate into a CLEAN tared reservoir as defined in step 3.5. Weigh the upper reservoir to determine ppt. loss.

Volume ppt. ~25mL (ea) mL DIW wash 75 mL@
Lower reservoir tare 125.1 g gross mass 291.1 g net solution 166.0 g
Upper reservoir gross mass CA2 127.8 CA1: 133.5 g net ppt mass CA1 45.1 g CA2 38.8g
Start time AN107 CA1 = 12:52 AN107 CA2 = 13:08 End time AN107 CA1 = 10:58 AN107 CA2 = 1313
12:58 SK 2/7/00

3.13 If any solids remain, sub-sample the solids and dry to constant weight at 105°C. Pull an aliquot for TIC/TOC analysis, dissolve the remainder and analyze for Radchem and ICP.

Vial tare mass 16.7800 g

Gross mass before drying	20.8553 g	Net wet mass	4.0733 g
Mass 1 after drying	18.1915 g	Net dry mass	1.4115 g
Mass 2 after drying	18.1829 g	Net dry mass	1.4029 g
Calculated % solids	18.1750 g/g		1.3950
	34.25%		

Mass 3 2.3 days drying

3.14 If any further solids are available, sub-sample and analyze for TCLP.

3.15 If any further solids remain, sub-sample for particle size distribution and XRD.

3.16 Sample the 1M HNO3 filtrates and the water filtrate for ICP (5-mL), TIC/TOC/IC (3-mL), Hg (3mL), NH3 (3mL) and Radchem (10-mL) analyses. Label as per the analytical table. Determine solution densities.

	1st 1M HNO3 wash	2nd 1M HNO3 wash	H2O wash
10-mL vol. Flask tare	12.0813 g	11.7098 g	12.0121
gross mass	22.4708 g	22.1997	22.1011
net mass	~~10.3895~~	10.4899	10.089
calculated density	1.03895 g/mL	1.048998 g/mL	1.0089

Temperature 19.5 °C

Balance ID 360-06-01-026

Radchem GEA: 10.3276g 10.4344g 10.0260g

4.0 Barium Sulfate Precipitation

4.1 Tare a 1-L plastic bottle with a stir bar. Add the remaining AN107 Ca treated filtrate material and weigh again. The sulfate concentration should be nominally 0.036 M including the dilutions thus far.

Note, we add AN107 Ca bottom

Bottle tare mass 134.7g g

Gross mass with sample 777.1 g

Net sample mass 642.4 g

Sample volume 531.4 mL (see density step 3.8)

Moles sulfate 0.5314 L x 0.041 ~~M/DF~~ 0.036 sulfate= 0.019 moles SO4

Calculate a 1.3:1 barium:sulfate composition 0.0247 moles Ba

Calculate volume of ~~2M~~ 1.82M Ba addition necessary 13.57 mL ~~2M~~ 1.82M Ba = 18.3466g = 5.24g

4.2 Add _____ ~~mL 2M~~ 1.82M Ba(NO2)2-H2O to the AN107 Ca pre-strike sample with stirring. Continue stirring for 10 minutes. Record the start and end time for Ba addition. Stirred 60 min.

Start time 13:52 End time 13:58

4.3 Weigh the sample and determine the mass and volume of added 2M Ba solution.

Gross mass 795.8 g

1.82 Net ~~2M~~ Ba added 18.3466 g

Net volume ~~2M~~ Ba added 13.57 mL

Rinsed 1.82M Ba vial with ~20x0.5 H_2O & added this - not added

$Ba(NO_2)_2$ = 18.3466 g

d = 1.344 g/mL = 13.65 mL

stir

4.4 Allow the sample to ~~sit~~ for at least 60 minutes.

4.5 Re-suspend solids and pour entire contents into a 1-L graduated cylinder. Record the total volume. At 10-min intervals, record the volume of the settled precipitate, also measure centimeters above precipitate of clear supernatant.

	Time	Precipitate volume (mL)	Supernatant (cm above ppt)
11/17/99	T_0 15:10	550	0
	15:20	390	5.6
	15:30	250	10.5
	15:40	170	13.4
	15:50	145	14.2
	16:00	130	14.8
	16:10	120	15.1
	16:20	113	15.3
	16:30	110	15.5
	16:40	105	15.6
	16:50	100	15.8
	17:00	99	15.9
	17:10	95	15.9
11/18/99	8:00 am	60	17.1

19.2 ~~FCm~~ total ht.

picture 5

picture

4.6 Mix the solution, re-suspending the solids.

4.7 Sub-sample 10-mL into a tared 10-mL volumetric flask and determine the solution density. From this material sub-sample 8-mL into a vial and submit for viscosity testing. Use the remaining 2-mL to determine insoluble solids (section 7.0). Aliquot an additional 5-mL for particle size analysis. Label samples TI-71-BaSO4-suspension.

Volumetric flask tare	12.1202	g
Gross mass	24.2194	g
Net solution mass	12.0992	g
Calculated density	1.2099	g/mL
Temperature	21	°C

Balance MettE 360-06-01-026

5.0 Determination of BaSO4 filter flux

5.1 Label and weigh both the lower reservoir and upper reservoir of a 1-L filtration apparatus containing a 0.45-µm nylon filter. Have on reserve a second upper and lower reservoir, labeled and weighed.

Lower reservoir mass	AN107 Ba1	124.5	g
Upper reservoir mass	AN107 Ba1	88.6	g
Reserve lower reservoir	AN107 Ba2	124.5	g
Reserve upper reservoir	AN107 Ba2	89.0	g

5.2 Obtain a vacuum gauge meter to measure the house vacuum line and a stopwatch. Measure the house vacuum.

20 mm Hg

5.2 **NOTE: This step will require 2 analysts**. Place the filter apparatus into secondary containment. Hook the filter apparatus to the house vacuum. Pour the AN107 /Ba mixture into the upper reservoir of the filter apparatus. Simultaneously record the start time and start the vacuum, measuring the time it takes to pull all of the filtrate through the filter. Shut off the vacuum and disconnect the hose while shutting off the timer.

11/13/99 Start time 8:25 a.m. End time 9:47 a.m

5.3 Disconnect the lower reservoir from the upper reservoir and measure the mass of filtrate. Measure the vacuum reading

(R2) 11-18-99

Gross filtrate mass ~~110.7~~ 743.9 g
Net filtrate mass 619.4 g
Calculated mass filtered per second: _____ g/sec
20 mm Hg

5.4 Finish filtering the sample, pulling as much volume of AN107 as practical, using the Ba treated filtrate to rinse the graduated cylinder as necessary.

5.5 Disconnect the lower filter chamber apparatus.

5.6 Weigh the final mass of the filtrate.
Gross filtrate mass _____ g
Net filtrate mass _____ g

5.7 Weigh the gross filter chamber mass, determine the net wet BaSO4 precipitate weight.
Gross filter mass 110.7 g
Net wet ppt mass 22.1 g

Sample codes for Table 6.1 tare wt = 17.0843 wt vial + sample = 17.9571

wt upper reservoir after sep
gross = 109.6 g
Net = 21.0g

6.0 BaSO4 Solids Washing

6.1 Determine the ppt volume. Wash the solids with about a 2:1 volume 0.01M NaOH:ppt volume collecting the wash in a CLEAN tared lower reservoir as defined in step 3.5. Save the wash solution and label it as TIxx-BaSO4-NaOH wash1.
Precipitate volume: 20 mL Added wash volume 40 mL
Reservoir tare (R02) 124.5 g gross mass 164.1 g net solution 39.6 g
Start time 10:06 End time 10:07

6.2 Wash the solids again with the same ratio collecting the solids in another clean lower reservoir. Save the wash solution and label it as TIxx-BaSO4-NaOH wash2.
Added wash volume 40 mL
Reservoir tare 124.6 g gross mass 165.0 g net solution 40.4 g
Start time 10:16 End time 10:16

upper reservoir mass 108.8g 20.2g net wet BaSO4 ppt mass

6.3 Remove 10-mL AN107 Ba treated filtrate solution into a tared volumetric flask. Determine the density and submit for radchem, ICP (4-mL), TIC/TOC, IC (3mL), NH3 (3-mL) and Hg (3mL) analysis. Label sample as TIxx-BaSO4-FILTRATE.
Volumetric flask tare 12.0147 g
Gross mass 24.0786 g
Net solution mass 12.0639 g
Calculated density 1.20639 g/mL
Temperature 21.5 °C
Balance 340-06-01-026

6.4 Remove aliquots of each wash solution and submit 10-mL for radchem, 5-mL for ICP, and 3-mL for TIC/TOC/IC, 3-mL for NH3, and 3-mL for Hg (analytical labs can sub-aliquot for Hg). Pro-rate volumes as necessary.
11.9985g - GEA/Radchem

filtrate mass after sampling
tare 124.5 gross 708.9g Net 584.4g = 484.4 mL

6.5 Remove a portion of wet precipitate (BaSO4) and cap tightly in a tared 2-dram vial. Determine net sample mass, suspend in 2-mL 0.01M NaOH and submit for particle size analysis. Remove another portion of the BaSO4 ppt and submit for XRD analysis. Remove another portion of the BaSO4 precipitate into a tared glass vial. Measure the wet weight. Dry at 105°C to constant weight. Determine the % solids. Remove 3 aliquots of the wet solids for Hg analysis and 2 aliquots for NH3 determination.

Particle size analysis tare 9.7605 g gross mass 9.7685 g net mass 0.0080 g
25.9648

TI-71-BaSO4-1 Hg1 tare 25.8835 g gross mass ~~25.9217~~ g net mass 0.0813 g
TI-71-BaSO4-1 Hg2 tare 25.7928 g gross mass 25.9145 g net mass 0.1217 g
TI-71-BaSO4-1 Hg tare 25.6940 g gross mass 25.8011 g net mass 0.1071 g

TI-71-BaSO4-1 NH31 tare 9.7618 g gross mass 9.8491 g net mass 0.0873 g
~~TI-71-BaSO4-1 NH3 2 tare g gross mass g net mass g~~

Vial tare mass	17.2852 g	18.2458 = gross wet mass	
Gross mass before drying	18.2458 g	Net wet mass	0.9606 g
Mass 1 after drying	17.6210 g	Net dry mass	0.3358 g
Mass 2 after drying	17.6190 g	Net dry mass	0.3338 g
Calculated % solids	17.6141 34.45%g/g		0.3309

Mass 3 17.5244

tare XRD 16.9547 gross mass = 17.2206 Net = 0.2659

6.6 Remove a portion of the dried solids for TIC/TOC analysis. Submit another sub-sample for ICP and Radchem analysis.

6.7 Wash the remaining solids with 2:1 1M HNO3 solution: ppt volume, collecting the rinse into a CLEAN, tared reservoir as per step 3.5. Weigh the upper reservoir and determine the net ppt. mass.
OR 11-18-99
Added wash volume ~~40~~ ~~20~~ 40 mL
Reservoir tare 125.0 g gross mass 175.1 g net solution 50.1 g
Upper reservoir gross mass 97.3 g net mass 8.7 g
Start time 10:49 End time 10:50

6.8 Determine the remaining ppt vol. Repeat the wash using the 2:1 1M HNO3, collecting the rinse into another CLEAN, tared reservoir.
Precipitate vol. ~~40~~ 20 10 @a mL 11-18-99 Added wash volume 20 mL
Reservoir tare 124.1 g gross mass 140.6 g net solution 16.5 g
Start time 11:11 End time 11:12
Upper reservoir gross mass = 101.8 Net mass = 13.2 g

6.9 Wash the solids with 3:1 DIW: ppt volume, collecting the rinse into another CLEAN, tared reservoir.
Added wash volume 30 mL
Reservoir tare 125.0 g gross mass 154.3 g net solution 29.3 g
Start time 11:26 End time 11:30
Upper reservoir gross mass = 101.9 13.3g net BaSO4 wet weight

6.10 Sample each rinse above for ICP analysis (5-mL), Radchem analysis (10-mL), Hg analysis (3-mL), TIC/TOC/IC analyses (3-mL), and NH3 (3-mL). Pro-rate sample volumes as necessary.

6.11 Sub-sample the solids and place in a tared glass vial. Dry at 105°C to constant weight. Determine the percent solids.

Vial tare mass	17.1447 g			
Gross mass before drying	19.0857 g	Net wet mass	1.9410	g
Mass 1 after drying	18.0124 g	Net dry mass	0.8677	g
Mass 2 after drying	18.0118 g	Net dry mass	0.8671	g
Calculated % solids	44.35% g/g		0.8608	

mass 3 2.3 days drying

6.12 Sample the dried solids and submit for digestion/fusion, ICP and radchem analysis.

6.13 Sub-sample the wet solids for TCLP leach/analysis. tare 17.0879 gross 18.5081

6.14 Sub-sample the wet solids for particle size analysis (in water) and XRD analysis.

particle size gross tare 9.5318

XRD gross 18.1528 tare 17.3161

7.0 Insoluble Solids (Reference Method 2420D, Standard Methods for the Examination of Water and Wastewater)

7.1 Tare 4 25-mm diameter 0.045-µm nylon filters.

7.2 Suspend solids from samples retained from steps 1.9 and 4.6 .

7.3 Pass a known volume (e.g. 1-mL, depending on visual solids) of a sample through a filter.

7.4 Wash the precipitate with 5-mL DIW.

7.5 Dry the filter/precipitate at 105°C to constant weight.

7.5 Repeat steps 7.3--7.5 for successive samples on successive filters, taking care not to mix up the samples and filters.

This sample was filled virtually completely

	Filter Tare	Volume	Weight 1	Weight 2	Weight 3	
CaCO3—1						
CaCO3—2						
BaSO4—1	0.0232	{0.200 mL 0.2425 g	0.0256	0.0250	0.0250	0.018g/0.2 mL
BaSO4—2	0.0238	0.500 mL {0.6058 g	0.0308	0.0299	0.0300	0.062g/0.5mL

8.0 Volume Reduction

8.1 Tare a 600-mL beaker. Add the sample with sulfate removed to the beaker. Measure the transferred mass.

Beaker tare _____ g

Gross mass _____ g

Net mass _____ g

8.2 The [Na] is expected to equal 4.28M. Calculate the expected Na concentration based on actual volumes used.

4.97M Na/ DF (Ca addition) / DF(Ba addition)

8.3 Evaporate the AN107 Ba treated filtrate such that the final Na concentration equals 8M (nominally this will be a 47% reduction by volume). Cover, and allow the solution to cool.

8.4 Transfer the reduced volume with precipitates to a polyethlyene bottle.

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Radiochemical Processing Group
Radioanalytical Applications Team

COMMENTS

ASR#	
CLIENT	BNFL

make new 1M $Ba(NO_2)_2$ solution

Balance : 360-06-01-038

6.1893 g $Ba(NO_2)_2 \cdot H_2O$ → 25 ml vol flask

measured 6.1899 g $Ba(NO_2)_2 \cdot H_2O$.

dissolved in warm water = still incomplete dissolution. A white powdery material remains

V filtered original 2M solution; provide a 1000x dilution to ICP

0.2 mL → 200 mL vol flask

pipet 33032 0.1982 g
0.1978
~~0.1990~~ 0.1973
0.1971 g

$\bar{x}$ = 0.1976 +/- 0.0005 g
= +/- 0.25%

vol flask tare 106.6379 g
+ sample 200ml 106.8944
0.2565 g $Ba(NO_2)_2 \cdot H_2O$ 2M

final mass 305.9163 g δ = 0.9964 g/ml

New density determination: tare 10-ml vol. flask: 11.8369 g
gross 25.2811
Net 13.4442
δ = 1.3444 g/mL at 20°C

analyst D. K. Fiskum date

reviewer date

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Radiochemical Processing Group
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COMMENTS

ASR#	BNFL-TI-24993-071
CLIENT	BNFL

Balance Calibration Confirmation 11/16/99

CA balance Acculab V-4800 Calibrated balance 510-06-01-022

1 kg:	1000.0	1000.05
500 g	500.0	500.04
200	200.0	200.01
100	100.0	100.02
3 Kg	2999.6	2999.90
20 g	20.0*	

(1) Balance V-4800 - Tare 1 L plastic bottle + 2" magnetic stirring bar. Placed on balance & zeroed. Remove & replace with stir bar & bottle in different positions. No change (to tenth of mg) if bottle is centered. Repeat 4-5 times - same result. BR 11/16/99

* from Mettler Newegh Set NN2106 = M+TE

(2) $CaCO_3$ settling total height = 30.1 cm.
6.5 cm o.d. calculated 6 cm i.d.
100 ml vol in 1-L graduate = 3 cm height ∴ r = 3.0 cm, diam = 6.0 cm

(3) After 1st DI wash of CA salts cake of solids ~ 150 & 100 ml, respectively. BR 11-17-99

analyst S.K. Fiske date 11/17/99

reviewer _____ date

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COMMENTS

ASR#	
CLIENT	

11/17/99 Balance check

	V - 4800	570-06-01-022
1 kg	1000.1	1000.04
500 g	500.0	500.03
200 g	200.0	200.00
100 g	100.0	100.02
3 kg	3000.3	2999.89

Step 3.13 sample $CaCO_3$ solids after water wash

	analysis	tare	gross (wet wt)
T1-71-$CaCO_3$-Hg-1		25.7618 gr	27.4743
	Hg-2	25.9458	27.8779
	Hg-MS	25.7243	27.5993
	XRD	16.8253	20.5183
	particle size	16.8845	
	TCLP	16.8206	20.7811
	NH_3	9.6318 g	10.9309

Use AN 107 CA 1 bottom for Ba pot. Gross wt = 767.6, + stirbar & reweigh = 777.1 So new tare wt = old tare wt + (777.1 - 767.6) = 125.2 + 9.5 = 134.7 g (see 2.1)

analyst U. K. Stoker date 11/17/99 reviewer _____ date _____

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ASR#	
CLIENT	

Balance checks

11/18/99

	Acculab V-4800	570-06-01-022
1 kg	1000.1	1000.04
500 g	500.0	500.04
200 g	200.0	199.99
100 g	100.1	100.02
3 kg	3000.2	2999.90

analyst VK Fiske date 11/18/99

reviewer _____ date _____

Chart for Recording Sample Filtration Rates Procedure TI-29953-071, Rev. _____

Step #: _____

Start time: _____ End time: _____

	Time	Filtrate Volume
AN107CA1	13:54	0
	13:56	50 mL
	13:59	100mL
	14:05	150 mL
	14:11	200 mL
	14:18	250 mL
AN107CA2	14~~13~~:37	0
	14~~13~~:38	50 mL
	14:46	150mL

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ASR#	BNFL
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Ca ppt showed gas evolution through the 1st & 2nd 1M HNO_3 washes.

Note: 1st 1.0 M HNO_3 to Ba ppt gave much gas evolution.

Step 3.5 $CaCO_3$ % solids prior to any washing

vial tare mass	16.7331 g		
gross mass before drying	19.6669	Net wet mass	2.9338 g
Mass 1 after drying	17.8931	Net dry mass	1.1600 g
Mass 2 after drying	17.8809	Net dry mass	1.1478
Calculated % solids	1.1443 / 2.9338 × 100 = 39.00%		
Mass 3 after weekend drying	17.8774	Net dry mass	1.1443

Step 5.7 $Ba(IO_3)_2$ % solids prior to any washing

vial tare mass	17.0843		
gross mass before drying	17.9571	Net wet mass	0.8728 g
Mass 1 after drying	17.4985	Net dry mass	0.4142 g
Mass 2 after drying	17.4922	Net dry mass	0.4079
Calculated % Solids.	0.4052 / 0.8728 × 100 =	46.43%	
Mass 3 after weekend drying	17.4895	Net dry mass	0.4052

analyst B. R. 4 date 11-17-99

reviewer date

Filtration following
~~[redacted]~~ BA ppt.

8:25	530mL total / unfiltered
8:45	300mL total / unfiltered.
8:52	250 "
9:03	200 "
9:14	150 "
925	100 "
938	50 "
947	20 mL

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ASR#	
CLIENT	

11/18/99

Steps 6.1 + 6.2 Determine solution density

	$BaSO_4$ NaOH first wash	$BaSO_4$ NaOH second wash
tare (10-mL vol)	11.9348g	11.6003
gross	22.5492	21.7393
net mass	10.6144	10.1390
calculated density	1.06144	1.0139
T:	21.5°C	
Balance ID	360.06.01-026	
GEA Radchem:	10.5711g	10.0936g

Steps 6.7, 6.8, 6.9

	$BaSO_4$ - HNO_3 - wash 1	$BaSO_4$ - HNO_3 - wash 2	$BaSO_4$ - H_2O - wash 1
tare (10-mL)	11.8103	11.7587	11.9184
gross	22.3157	22.1685	22.0346
Net mass	10.5054	10.4098	10.1162
calculated density	1.05054	1.04098	1.01162
GEA Radchem:	10.4263g	10.3743	10.0791g

analyst K.K. Anderson date 11/18/99 reviewer date

Battelle
Radiochemical Processing Group
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COMMENTS

ASR#	
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11/18/99

Step 6.11 Sample $BaSO_4$ washed solids for Hg & NH_3

TI-71-$BaSO_4$-2	tare	gross wt	Net wt.
Hg-1	25.9734	26.0289	
Hg-2	25.7817	25.9637	
Hg-3(MS)	25.5706	25.8232	
NH_3	9.7688	9.8503	
IC/TIC/TOC	9.6170	9.6893	

analyst D. K. Fisk date 11/18/99 reviewer _____ date _____

Table C.1. Actual AN-107 Scale-Up Test Detail, Phase 3

	Solutions								Solids					
Description	AN107 mass (g)	Reactant and filtrate mass (g)	Density (g/mL, T~20C)	Solution vol (mL)	Total filtration time (min)	Sampled mass (g)	Net mass remaining (g)	Wt. % Recovered Filtrate	Wet solid in filter (g)	Approx. volume (mL)	Wet solids sampled (g)	Wet solids remaining (g)	Wt.% solids	Wt. % change on wash
$CaCO_3$ Precipitation														
AN107 Starting material	926.4		1.224	756.6		26.9	899.5		na				na	
Added 5M $Ca(NO_3)_2$		163.4	1.527	107.0			1062.9							
AN-107+$CaCO_3$, suspended solids		1062.9	1.241	856.3		12.413	1050.5							
Filter	678.6		1.209	561.3	77	36.2	642.4	64.6%	361.1	300	2.9	358.2	39.0	
Wash 0.01M NaOH		677.7	1.068	635 (filtrate)	62	24			291.4	300				-19%
Wash 0.01M NaOH, 2nd		551.1	1.014	543 (filtrate)	60	24			254.4	300	7.1	247.3	24.0	-13%
Wash 1.0M HNO_3		517.5	1.039	498 (filtrate)	20	24			140	200	0	140		-45%
Wash 1.0M HNO_3, 2nd		264.2	1.049	252 (filtrate)	9	24			91.2	100	0	91.2		-35%
DI water wash		166	1.009	165 (filtrate)	11	24			83.9	50	est. 5g	nm	34.3	-8%
$BaSO_4$ Precipitation														
AN-107 Filtrate from Ca prestrike	642.4		1.209	531.4			642.4							
Added 1.82M $Ba(NO_2)_2$		18.35	1.344	13.7										
AN-107+$BaSO_4$ suspended solids			1.210			12.099	648.65							
Filter	619.4		1.206	513.4	82	35	584.4	95.5%	22.1	20	0.8728	21	46.4	
Wash 0.01M NaOH		39.6	1.061	37.3 (filtrate)	1	24			nm					
Wash 0.01M NaOH, 2nd		40.4	1.014	39.8 (filtrate)	<1	24			20.2	20	1.63	18.6	34.5	-3.8%
Wash 1.0M HNO_3		50.1	1.051	47.7 (filtrate)	1	24			8.7	10		8.7		-57%
Wash 1.0M HNO_3, 2nd		16.5	1.041	15.9 (filtrate)	1	24			13.2	10		13.2		+52%
DI water wash		29.5	1.012	29.2 (filtrate)	4	24			13.3	10	all		44.4	+1%

Notes: na = not applicable, nm = not measured

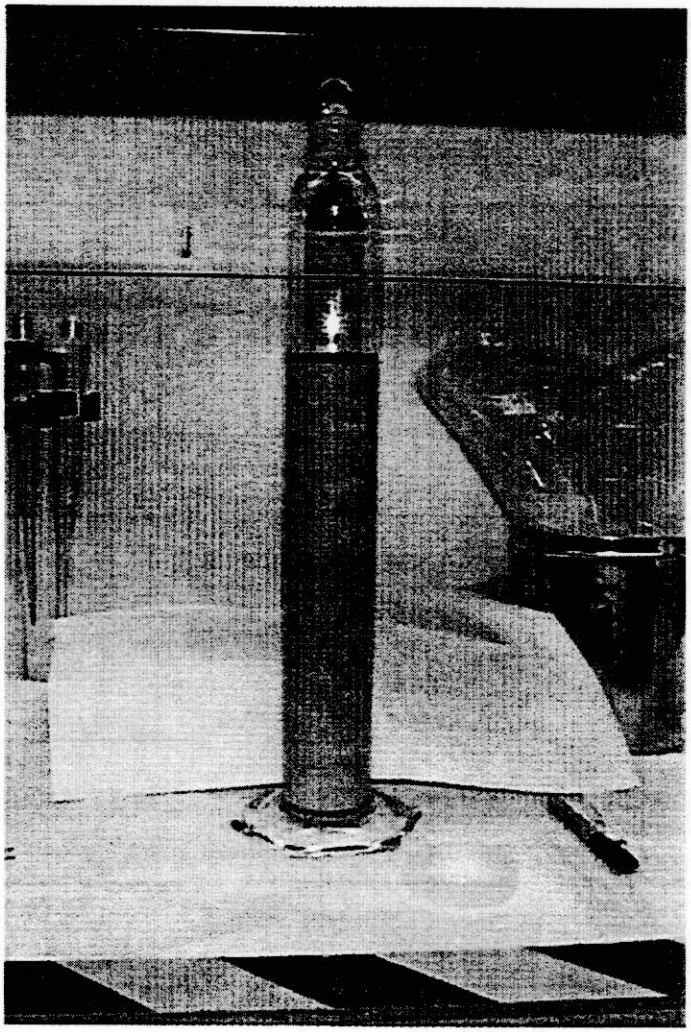


Figure C.1. $CaCO_3$ Slurry Settling Test at T=2 Hours

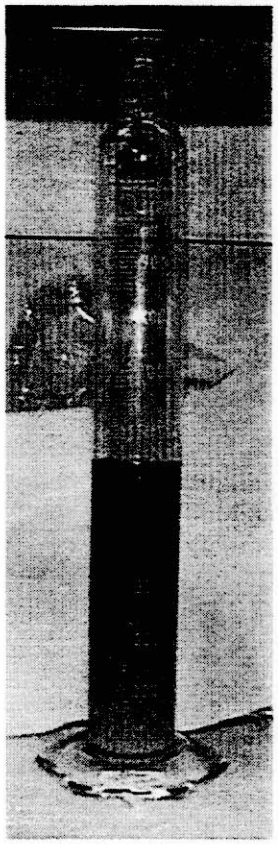


Figure C.2. $BaSO_4$ Slurry Settling Test at T=2 Hours

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: S. Fiskum

Task 12
T2.12.5.2

RECORD COPY

ACL Number(s): 00-0461 through 00-0474, 00-0476, 00-0477, 00-0479 through 00-0481

Client ID: "TI-71-AN107" through "TI-71-BaSO4-H2O wash 1"

ASR Number: 5600

Total Samples: 19

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

Analyst: D.R. Sanders

Analysis Date (Filename): 12-03-99 (A0564) & 12-07-99 (A0566)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry Wagner 01-04-00
Reviewed by

MW Ulm 1-5-00
Concur

1/4/00

Four radioactive solid samples and one duplicate, **TI-71-CaPS ppt prior to wash** through **TI-71-CaCO3-2** (ACL# 00-0465, 00-0468, 00-0472 and 00-0472 dup), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were digested using 1ml of nitric acid, 1ml of hydrochloric acid and diluted to 20ml with water. Approximately 0.25-gram sample aliquots were prepared.

Fifteen radioactive aqueous samples and one duplicate, **TI-71-AN107** through **TI-71-BaSO4-HNO3 wash 2** (ACL# 00-0461 through 00-0464, 00-0466, 00-0467, 00-0469 through 00-0471, 00-0473, 00-0474, 00-0476, 00-0477, 00-0479 through 00-0480), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were digested using PNNL-ALO-128 procedure. One to 5ml sample aliquots were prepared and diluted to a final volume of 10ml with water except sample 00-0479 required additional acid followed by dilution to 20ml with water. Sample 00-0473 was prepared in duplicate.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in µg/ml for aqueous samples and µg/g for solid samples. Weights and volumes used have been recorded on bench sheets and included with this report.

Various samples contained high concentrations of sodium, calcium, barium, aluminum, or strontium.

Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous and solid samples)

All results for analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution except calcium in two samples that were slightly low (13% and 11%). The two samples affected were ALO# 00-0463 and 00-0464 (aqueous samples). All other samples tested were within tolerance limits. The reason for the low result is not apparent.

Duplicate RPD (Relative Percent Difference):
(Aqueous and solid samples)

All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):
(Aqueous and solid samples)

All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):
(Aqueous and solid samples)

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous and solid samples)

None required or prepared.

Matrix Spiked Sample:
(Aqueous and solid samples)

None required or prepared.

Quality Control Check Standards (aqueous and solid samples):

Concentration for all analytes of interest is within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV. ICP98.0 (calibration blank) was within two times IDL.

High Calibration Standard Check (aqueous and solid samples):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy.

Process Blank:
(Aqueous and solid samples)

All analytes are within tolerance limit of ≤ EQL or < 5% of sample concentration.

Laboratory Control Standard (LCS):
(Aqueous and solid samples)

No LCS was prepared for PNL-ALO-128 acid digested samples.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

1/4/00

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/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	10.0	25.0	50.0	83.3	16.7
	ALO#=	00-461-PB	00-461 @5	00-462 @5	00-463 @25	00-464 @5
	Client ID=	Process Blank	TI-71-AN107	TI-71-AN107 DUP	TI-71-CaPS-filtrate	TI-71-CaPS-filtrate DUP
Det. Limit (ug/mL)	Run Date= (Analyte)	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	--	2,380	2,310	852	849
0.250	As	--	--	--	--	--
0.050	B	--	19.4	[18]	--	[1.8]
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	168	175	975	907
0.015	Cd	--	27.4	26.7	25.1	24.9
0.200	Ce	--	--	--	--	--
0.050	Co	--	[2.1]	--	--	[1.8]
0.020	Cr	--	43.1	42.8	36.9	35.7
0.025	Cu	--	13.5	[12]	[11]	12.3
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.44]	8.98	[8.6]	[3.1]	[3.4]
2.000	K	--	716	[690]	[630]	662
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	[1.4]	--	--	--
0.050	Mo	--	16.2	[16]	[15]	14.7
0.150	Na	--	110,000	110,000	103,000	104,000
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.79]	208	207	199	192
0.100	P	--	306	297	[50]	47.7
0.100	Pb	--	58.2	57.9	[25]	24.4
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	[32]	[31]	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	131	129	--	[0.78]
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	[76]	--	--	[65]
0.050	Y	--	--	--	--	--
0.050	Zn	[1.2]	[6.5]	[7.2]	[4.8]	[4.6]
0.050	Zr	--	[3.0]	[2.6]	--	--

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	25.0	50.0	16.7	10.0	2.0
	ALO#=	00-473 @5	00-473-REP @5	00-474 @5	00-476 @5	00-477
	Client ID=	Tl-71-BaSO4-filtrate	Tl-71-BaSO4-filtrate REP	Tl-71-BaSO4-filtrate DUP	Tl-71-BaSO4-NaOH wash 1	Tl-71-BaSO4-NaOH wash 2
Det. Limit (ug/mL)	Run Date= (Analyte)	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL	12/3/99 ug/mL
0.025	Ag	–	–	–	–	–
0.060	Al	793	788	746	213	55.1
0.250	As	–	–	–	–	–
0.050	B	[1.3]	–	[1.1]	[1.00]	–
0.010	Ba	112	113	101	42.1	16.2
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	922	919	851	308	103
0.015	Cd	22.9	22.8	21.5	5.84	1.43
0.200	Ce	–	–	–	–	–
0.050	Co	[1.7]	–	[1.6]	–	–
0.020	Cr	6.57	[6.7]	6.16	2.08	0.738
0.025	Cu	11.2	[10]	10.6	2.73	0.699
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[3.0]	[3.0]	[2.6]	[0.95]	[0.24]
2.000	K	773	[760]	736	212	50.3
0.050	La	–	–	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	–	–	–	–
0.050	Mo	13.6	[14]	12.7	[3.9]	1.11
0.150	Na	95,900	99,200	91,800	27,700	7,530
0.100	Nd	–	–	–	–	–
0.030	Ni	181	184	169	46.9	11.7
0.100	P	43.5	[44]	41.3	11.2	2.74
0.100	Pb	[23]	[23]	21.6	[6.0]	[1.4]
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	–	–	–	–
1.500	Sn	–	–	–	–	–
0.015	Sr	7.31	[7.3]	6.68	3.19	1.53
1.500	Te	–	–	–	–	–
1.000	Th	–	–	–	–	–
0.025	Ti	–	–	–	–	–
0.500	Tl	–	–	–	–	–
2.000	U	–	–	–	–	–
0.050	V	–	–	–	–	–
2.000	W	[60]	–	[56]	–	[4.8]
0.050	Y	–	–	–	–	–
0.050	Zn	[4.4]	[5.0]	[4.0]	[1.2]	[0.45]
0.050	Zr	–	–	–	–	–

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

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	50.0					
	ALO#=	00-480 @5					
		Tl-71-BaSO4-HNO3 wash					
	Client ID=	2					
Det. Limit	Run Date=	12/3/99					
(ug/mL)	(Analyte)	ug/mL					
0.025	Ag	–	–	–	–	–	
0.060	Al	[5.8]	–	–	–	–	
0.250	As	–	–	–	–	–	
0.050	B	–	–	–	–	–	
0.010	Ba	7,900	–	–	–	–	
0.010	Be	–	–	–	–	–	
0.100	Bi	–	–	–	–	–	
0.250	Ca	959	–	–	–	–	
0.015	Cd	[1.4]	–	–	–	–	
0.200	Ce	–	–	–	–	–	
0.050	Co	–	–	–	–	–	
0.020	Cr	55.7	–	–	–	–	
0.025	Cu	–	–	–	–	–	
0.050	Dy	–	–	–	–	–	
0.100	Eu	–	–	–	–	–	
0.025	Fe	[1.5]	–	–	–	–	
2.000	K	–	–	–	–	–	
0.050	La	–	–	–	–	–	
0.030	Li	–	–	–	–	–	
0.100	Mg	–	–	–	–	–	
0.050	Mn	–	–	–	–	–	
0.050	Mo	–	–	–	–	–	
0.150	Na	592	–	–	–	–	
0.100	Nd	–	–	–	–	–	
0.030	Ni	[4.8]	–	–	–	–	
0.100	P	[6.1]	–	–	–	–	
0.100	Pb	–	–	–	–	–	
0.750	Pd	–	–	–	–	–	
0.300	Rh	–	–	–	–	–	
1.100	Ru	–	–	–	–	–	
0.500	Sb	–	–	–	–	–	
0.250	Se	–	–	–	–	–	
0.500	Si	–	–	–	–	–	
1.500	Sn	–	–	–	–	–	
0.015	Sr	110	–	–	–	–	
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	–	–	–	–	–	
0.050	Zr	–	–	–	–	–	

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	78.3	528.8	16.7	2.5	294.6
	ALO#=	00-465-PB	00-465 @5	00-466 @5	00-467	00-468 @5
	Client ID=	Process Blank	IT-71-CaPS ppt prior to wash	IT-71-CaPS - NaOH wash 1	IT-71-CaPS - NaOH wash 2	IT-71-CaPS - H2O wash 1
Det. Limit (ug/mL)	Run Date= (Analyte)	12/7/99 ug/g	12/7/99 ug/g	12/7/99 ug/mL	12/7/99 ug/mL	12/7/99 ug/g
0.025	Ag	–	–	–	–	–
0.060	Al	–	12,100	[9.5]	3.14	21,700
0.250	As	–	–	–	–	–
0.050	B	–	392	[5.0]	4.10	275
0.010	Ba	[1.0]	[17]	–	[0.055]	[25]
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	–	236,000	331	132	360,000
0.015	Cd	–	[75]	6.60	1.07	[24]
0.200	Ce	–	–	–	–	–
0.050	Co	–	–	–	–	–
0.020	Cr	–	110	10.2	2.67	[32]
0.025	Cu	–	[31]	[3.0]	0.669	[14]
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	–	[51]	–	–	79.0
2.000	K	–	–	[140]	[36]	–
0.050	La	–	[79]	–	–	–
0.030	Li	–	–	–	–	–
0.100	Mg	–	652	–	–	1,050
0.050	Mn	–	–	–	–	[16]
0.050	Mo	–	[36]	[4.1]	[0.94]	–
0.150	Na	–	267,000	25,500	7,150	50,500
0.100	Nd	–	–	–	–	–
0.030	Ni	[5.7]	480	54.4	11.8	[82]
0.100	P	–	2,040	[11]	2.64	2,940
0.100	Pb	–	[260]	[2.3]	[0.37]	423
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	[1,200]	[37]	32.3	[360]
1.500	Sn	–	–	–	–	–
0.015	Sr	–	1,090	[0.54]	0.495	1,690
1.500	Te	–	–	–	–	–
1.000	Th	–	–	–	–	–
0.025	Ti	–	–	–	–	[7.5]
0.500	Tl	–	–	–	–	–
2.000	U	–	–	–	–	–
0.050	V	–	–	–	–	–
2.000	W	–	–	–	–	–
0.050	Y	–	–	–	–	–
0.050	Zn	[4.5]	[31]	[0.96]	[0.34]	[35]
0.050	Zr	–	–	–	–	[24]

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	12.5 / 00-469 @5 / IT-71-CaPS - HNO3 wash 1 / 12/7/99 / ug/mL	10.0 / 00-470 @5 / IT-71-CaPS - HNO3 wash 2 / 12/7/99 / ug/mL	10.0 / 00-471 @5 / IT-71-CaPS - H2O wash 1 / 12/7/99 / ug/mL	375.5 / 00-472 @5 / IT-71-CaCO3-2 / 12/7/99 / ug/g	439.8 / 00-472-DUP @5 / IT-71-CaCO3-2 / 12/7/99 / ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	19.8	9.70	[2.1]	51,500	54,700
0.250	As	--	--	--	--	--
0.050	B	[1.9]	20.2	8.64	[140]	[140]
0.010	Ba	[0.74]	[0.45]	[0.14]	[37]	[38]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	10,900	17,100	5,330	296,000	327,000
0.015	Cd	[0.36]	[0.95]	[0.34]	[29]	[31]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[0.82]	[0.36]	[0.42]	96.2	104
0.025	Cu	--	[0.40]	--	[12]	[14]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.69]	[1.7]	[0.97]	145	156
2.000	K	--	--	--	--	--
0.050	La	[3.9]	--	[1.8]	[120]	[140]
0.030	Li	--	--	--	--	--
0.100	Mg	[11]	80.7	29.0	1,080	1,150
0.050	Mn	--	--	--	[28]	[30]
0.050	Mo	--	--	--	--	--
0.150	Na	4,560	1,940	537	2,190	2,310
0.100	Nd	--	--	--	[82]	[93]
0.030	Ni	3.91	[3.0]	[1.2]	[70]	[74]
0.100	P	--	--	--	6,940	7,470
0.100	Pb	[6.1]	21.3	[8.0]	521	576
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	[220]	[230]
1.500	Sn	--	--	--	--	--
0.015	Sr	86.9	76.3	23.6	1,040	1,120
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	[0.34]	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	[24]	[26]
0.050	Zn	--	[1.8]	[0.85]	[44]	[46]
0.050	Zr	--	--	--	[59]	[65]

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	20.0	12.5			
	ALO#=	00-479 @5 IT-71-BaSO4-HNO3 wash 1	00-481 @5 IT-71-BaSO4-H2O wash 1			
	Client ID=					
Det. Limit	Run Date=	12/7/99	12/7/99			
(ug/mL)	(Analyte)	ug/mL	ug/mL			
0.025	Ag	–	–	–	–	–
0.060	Al	21.3	[1.3]	–	–	–
0.250	As	–	–	–	–	–
0.050	B	–	–	–	–	–
0.010	Ba	22,100	4,720	–	–	–
0.010	Be	–	–	–	–	–
0.100	Bi	–	–	–	–	–
0.250	Ca	3,080	263	–	–	–
0.015	Cd	3.73	[0.49]	–	–	–
0.200	Ce	–	–	–	–	–
0.050	Co	–	–	–	–	–
0.020	Cr	55.2	26.2	–	–	–
0.025	Cu	[0.63]	–	–	–	–
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[1.0]	[0.57]	–	–	–
2.000	K	–	–	–	–	–
0.050	La	[2.2]	–	–	–	–
0.030	Li	[0.99]	–	–	–	–
0.100	Mg	–	–	–	–	–
0.050	Mn	–	–	–	–	–
0.050	Mo	[1.1]	–	–	–	–
0.150	Na	2,630	162	–	–	–
0.100	Nd	[2.4]	–	–	–	–
0.030	Ni	9.88	[2.1]	–	–	–
0.100	P	[9.3]	[2.1]	–	–	–
0.100	Pb	–	–	–	–	–
0.750	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
1.100	Ru	–	–	–	–	–
0.500	Sb	–	–	–	–	–
0.250	Se	–	–	–	–	–
0.500	Si	–	–	–	–	–
1.500	Sn	–	–	–	–	–
0.015	Sr	356	33.1	–	–	–
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	–	–	–	–	–
0.050	Zr	–	–	–	–	–

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%.
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Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: S. Fiskum

ACL Number(s): 00-0456

Client ID: "0.002 M Ba"

ASR Number: 5596

Total Samples: 1

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

Analyst: D.R. Sanders

Analysis Date (Filename): 11-17-99 (A0556)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry Wagner 1-14-00
Reviewed by

M W Thomas 1-17-00
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

One aqueous sample, **0.002 M Ba** (ACL# 00-0456) was analyzed by ICPAES in an "as received" condition and after diluting 5 fold and 25 fold using 0.32N nitric acid. Barium is the only analyte of interest requested. The original concentration is calculated as shown below and reported as molarity of barium (mols per liter). Concentration uncertainty is approximately ± 10% or better. Concentration of barium reported is based upon the 25-fold sample dilution. The 25 fold diluted sample measurement result for barium has nearly the same concentration as that used to calibrate the instrument (10 µg/ml). The 5-fold dilution measurement is also within about 3% of the 25-fold dilution measurement for barium after correcting for analytical dilution.

Barium molarity is calculated as follows:

[Ba] = $(\mu g/ml)_{ICP}$ * 1.0E-03 (conversion factor to convert µg/ml to g/liter) / gram formula wt of barium (137.34 g/L) = mols Ba

[Ba] = 251 µg/ml * 1.0E-03) / 137.34 g/L = 0.00183 M Ba

Measurement results reported have been corrected for preparation and analytical dilution. Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous sample) All analytes of interest results are within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):
(Aqueous sample) None prepared.

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

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

Blank Spike:
(Aqueous sample) None.

1/14/00

Matrix Spiked Sample:
(Aqueous sample) None.

Quality Control Check Standards (aqueous sample):

Concentration for all analytes of interest is within tolerance limit of ± 10% accuracy for check standard QC_MCVA. All calibration blanks using check standard ICP98.0 for barium was within two times IDL.

High Calibration Standard Check (aqueous sample):

Verification of the high-end calibration concentration for all analytes in QC_SST check standard is within tolerance of ± 5% accuracy except for potassium (17% too high). A single element standard of potassium at 100 µg/ml was measured twice during the ICP run. Both measurement results (103 and 97.0 µg/ml) were both within acceptable tolerance limits.

Process Blank:
(Aqueous sample) None.

Laboratory Control Standard (LCS):
(Aqueous sample) None.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

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

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

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v 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.

1/14/00

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	1.0 / 00-0456 / Barium nitrite / 11/17/99 / ug/mL	5.0 / 00-0456 @5 / Barium nitrite / 11/17/99 / ug/mL	25.0 / 00-0456 @25 / Barium nitrite / 11/17/99 / ug/mL	
0.025	Ag	--	--	--	--
0.060	Al	--	--	--	--
0.100	As	--	--	--	--
0.050	B	[0.06]	--	--	--
0.010	Ba	>129	259	251	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.250	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	--	--	--
0.025	Cu	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	--	--	--	--
2.000	K	[8]	--	--	--
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	--	--	--
0.150	Na	2.42	[2.3]	--	--
0.100	Nd	--	--	--	--
0.030	Ni	[0.05]	--	--	--
0.100	P	--	--	--	--
0.050	Pb	--	--	--	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.050	Se	--	--	--	--
0.500	Si	--	--	--	--
0.750	Sn	--	--	--	--
0.015	Sr	2.22	2.11	[2.1]	--
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	--	--	--	--
0.050	Zr	--	--	--	--

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).

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

Client: S. Fiskum
ACL Numbers: 00-0461 to 00-0482
Analyst: MJ Steele

Charge Code/Project: W53400 / 29953
ASR Number: 5600
Analysis Date: November 22-24, 1999

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.

Final Results:

Lab ID	Sample ID	(1) F µg/ml	Cl µg/ml	NO_2 µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
00-00461	TI-71-AN107	3,600	< 500	28,700	113,000	1,400	4,100	1,400
00-00462	TI-71-AN107 Dup	3,500	< 500	28,800	112,000	1,400	4,000	1,500
	RPD	3%	n/a	0%	1%	0%	2%	7%
00-00463	TI-71-Ca PS filtrate	3,000	< 500	25,800	197,000	< 1000	3,300	< 1000
00-00464	TI-71-Ca PS filtrate Dup	3,000	< 500	26,000	199,000	< 1000	3,300	< 1000
	RPD	0%	n/a	1%	1%	n/a	0%	n/a
00-00466	TI-71-Ca PS NaOH Wash 1	900	< 250	7,100	58,500	< 500	1,100	< 500
00-00467	TI-71-Ca PS NaOH Wash 2	230	< 50	1,700	12,300	< 100	240	< 100
00-00469	TI-71-Ca PS HNO3 Wash 1	< 250	< 250	< 500	50,700	< 500	< 500	< 500
00-00470	TI-71-Ca PS HNO3 Wash 2	< 250	< 250	< 500	62,300	< 500	760	< 500
00-00471	TI-71-CA PS H2O Wash 1	< 125	< 125	< 250	16,500	< 250	< 250	< 250
00-00473	TI-71-BaSO4 filtrate	3,000	< 500	29,600	174,000	< 1000	< 1000	< 1000
00-00474	TI-71-BaSO4 filtrate Dup	3,000	< 500	29,500	172,000	< 1000	< 1000	< 1000
	RPD	0%	n/a	0%	1%	n/a	n/a	n/a
00-00476	TI-71-BaSO4 NaOH Wash 1	960	< 500	8,100	54,500	< 1000	< 1000	< 1000
00-00477	TI-71-BaSO4 NaOH Wash 2	290	< 250	2,000	13,700	< 500	< 500	< 500
00-00479	TI-71-BaSO4 HNO3 Wash 1	< 250	< 250	< 500	44,600	< 500	< 500	< 500
00-00480	TI-71-BaSO4 HNO3 Wash 2	< 250	< 250	< 500	51,800	< 500	< 500	< 500
00-00481	TI-71-BaSO4 H2O Wash	< 250	< 250	< 500	21,100	< 500	< 500	< 500
00-00481 Rep	TI-71-BaSO4 H2O Wash Rep	< 125	< 125	< 250	20,700	< 250	< 250	< 250
	RPD	n/a	n/a	n/a	2%	n/a	n/a	n/a
00-00481 MS	TI-71-BaSO4 H2O Wash MS %Rec	99%	95%	101%	89%	100%	104%	103%
Blank Spike	Blank Spike % Rec	96%	94%	100%	103%	101%	103%	101%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS %Rec = Matrix Spike Standard % recovery
(1) Fluoride is most likely formate or acetate due to slight peak shift and broadening

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

Lab ID	(1) Sample ID	(2) Solids Dil Fctr	(3) F µg/g	Cl µg/g	NO_2 µg/g	NO_3 µg/g	PO_4 µg/g	SO_4 µg/g	C_2O_4 µg/g
Leach Blank	Leach Blank	262	< 70	< 70	< 140	< 140	< 140	< 140	< 140
00-00465	TI-71-Ca PS ppt prior to Wash	325	5,600	< 1700	40,100	334,000	< 3300	8,000	< 3300
00-00468	TI-71-CaCO3-1	270	1,100	< 680	4,200	29,100	< 1400	4,300	< 1400
00-00468 Rep	TI-71-CaCO3-1 Rep	270	1,100	< 680	3,900	27,400	< 1400	3,800	< 1400
	RPD		0%	n/a	7%	6%	n/a	12%	n/a
00-00472	TI-71-CaCO3-2	190	430	< 240	< 480	17,300	< 480	1,300	< 480
00-00472 Dup	TI-71-CaCO3-2 Dup	195	530	< 250	< 490	29,800	< 490	1,500	< 490
	RPD		21%	n/a	n/a	53%	n/a	14%	n/a
00-00472 MS	TI-71-CaCO3-2 MS %Rec		96%	94%	100%	104%	98%	104%	102%
Blank Spike	Blank Spike % Rec		96%	94%	100%	103%	101%	103%	101%
00-00475 (4)	TI-71-BaSO4 prior to Wash								
00-00478 (4)	TI-71-BaSO4-1								
00-00482 (4)	IT-71-BsSO4-2								

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS %Rec = Matrix Spike Standard % recovery
(1) Carbonate solids -- approximately 50% of each sample did not dissolve.
(2) Leached Solids Dil Factor is average Dil Factor for solids, provides equivalent MDL for solids at 1x IC dilution.
(3) Fluoride is most likely formate or acetate due to slight peak shift and broadening
(4) Barium sulfate precipitates not soluble by water leaching and therefore not analyzed.

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation up to from 200-fold to 10,000-fold to ensure that all anions were within the calibration range. The solids samples were diluted from 5-fold to 50-fold following leaching per water leach method ALO-103. Due to the high anion concentration and the necessity for large dilutions to obtain usable ion chromatograms, most of the required Minimum Reportable Quantities listed in Table 6.1 (ASR attachment) could not be met. Attempts to analyze samples at lower dilution resulted in significantly distorted peaks and significant shifts in retention time making quantitation of the anion peaks impossible. The reported results are the best available. It should be noted that the fluoride results are suspect due to peak distortions and slight shift in retention times from the fluoride calibration standard.

Q.C. Comments:

Duplicates: The relative percent differences (RPD) for liquid samples, as well as the replicate measurement of the leachate from sample TI-71-$CaCO_3$-1 (00-0468), were all within the acceptance criteria of 20%. Although no apparent solids (precipitates) duplicates were provided, sample TI-71-$CaCO_3$-2 was leached in duplicate. The RPDs for this sample are very poor for those anions above the estimated quantitation level, particularly nitrate (i.e., RPD=53%). Following leaching approximately 50% of the solids remained undissolved, probably adding to the difficulty in obtaining good reproducibility on the leachates.

Matrix Spike: Both the liquid and solids matrix spikes measured anion recoveries from 89% to 104%, well within the 75% to 125% acceptance criteria.

Blank Spike: The blank spike recovery was within the acceptance criteria of 80% to 120%.

System Blank/Processing Blanks: Nine system blanks were process during the analysis of the samples. Only a single nitrate blank was detected in the system blanks above the estimate quantitation level. Since the nitrate results for the samples is extremely high, this small blank has no affect on the reported results.

Quality Control Calibration Verification Check Standards: Seven mid-range verification standards were analyzed throughout the analysis runs. Except for only a single chloride value, the reported results for all anions of interest were recovered within the acceptance criteria of ±10% for the verification standard. The single chloride failure measured –11%, only 1% below the acceptance criteria, and is not considered to affect any reported results.

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.

Analyst: M J Steele **Date** 1/13/00

Approval: M W Chin **Date** 1/14/00

Archive Information:

Files: ASR 5600 Fiskum.doc	ASR 5600.xls

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

Client: S. Fiskum
ACL Numbers: 00-0461 to 00-0482
Analyst: MJ Steele
Charge Code/Project: W53400 / 29953
ASR Number: 5600
Analysis Date: March 16-20, 2000

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).

Final Results:

ALO	Sample ID	Wt (g)	TIC (µg C/g)	TIC RPD (%)	TOC (µg C/g)	TOC RPD (%)	TC (µg C/g)	TC RPD (%)
00-00465	TI-71-CaPS ppt prior to wash	0.0433	36,200		27,000		63,200	
00-00465 dup	TI-71-CaPS ppt prior to wash dup (analysis)	0.0071	50,100	32	32,000	17	82,100	26
00-00468	TI-71-CaCO3-1	0.0114	72,800		12,900		85,800	
00-00468 dup	TI-71-CaCO3-1Dup (analysis)	0.0101	74,500	2	13,100	1	87,600	2
00-00472	TI-71-CaCO3-2	0.0100	77,200		28,900		106,000	
00-00472 dup	TI-71-CaCO3-2 Dup (analysis)	0.0210	77,600	0	26,700	8	104,000	2
00-00475	TI-71-BaSO4 ppt prior to wash	0.0162	24,600		23,800		48,400	
00-00475 dup	TI-71-BaSO4 ppt prior to wash dup (analysis)	0.0266	24,400	1	24,100	1	48,500	0
00-00475 ms	TI-71-BaSO4 ppt prior to wash MS Recovery	0.0185	94%		103%		98%	
00-00478	TI-71-BaSO4 - 1*	0.0132	30,200		20,700		50,900	
00-00482	TI-71-BaSO4 - 2	0.0321	670		1,800		2,500	
00-00482 dup	TI-71-BaSO4 - 2 Dup (analysis)	0.0240	650	n/a	1,700	n/a	2,300	n/a
ALO	**Sample ID**	**Vol**	**TIC (µgC/ml)**	**TIC RPD (%)**	**TOC (µgC/ml)**	**TOC RPD (%)**	**TC (µgC/ml)**	**TC RPD (%)**
00-00461	TI-71-AN107	0.10	8300		16100		24,400	
00-00461 dup	TI-71-AN107 dup (analysis)	0.10	8200	1	16700	0	24,300	0
00-00461 MS	TI-71-AN107 MS Recovery	0.10	101%		100%		101%	
00-00463	TI-71-CaPS Filtrate	0.10	2300		13300		15,500	
00-00464	TI-71-CaPS Filtrate Dup	0.10	2300		13400		15,700	
00-00466	TI-71-CaPS NaOH wash -1	0.20	430		4100		4,500	
00-00467	TI-71-CaPS NaOH wash -2	0.40	150		980		1,100	
00-00469	TI-71-CaPS -HNO3 wash -1	1.00	26		390		410	
00-00470	TI-71-CaPS -HNO3 wash -2	1.00	37		260		290	
00-00471	TI-71-CaPS H2O wash - 1	2.00	13		91		100	
00-00473	TI-71-BaSO4 filtrate	0.20	2100		13400		15,500	
00-00474	TI-71-BaSO4 filtrate Dup	0.20	2000		12800		14,800	
00-00476	TI-71-BaSO4 NaOH wash -1	0.40	740		3600		4,400	
00-00477	TI-71-BaSO4 NaOH wash -2	1.00	350		1000		1,400	
00-00479	TI-71-BaSO4 HNO3 wash - 1	0.20	<33		920		920	
00-00480	TI-71-BaSO4 HNO3 wash - 2	0.20	<33		460		460	
00-00481	TI-71-BaSO4 H2O wash - 1	1.00	<7		160		160	

* Insufficient sample for duplicate analysis

Shaded samples pH <2; no TIC anticipated

RPD = Relative Percent Difference (between sample and duplicate/replicate)

The analyses of the samples submitted under ASR 5600 were performed by 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. Solid samples were analyzed as received (i.e., no weight correction for moisture content has been applied).

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC) in either solid or liquid form.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being about 99% for TIC and 97% for TOC. This is well within the acceptance criteria of 90% to 110%.

Calibration Blanks: The eight calibration blanks run at the beginning, middle, and end of the analysis run were acceptable, averaging about 14 μgC TIC and 40 μgC TOC. These calibration/system blanks are considered good and the standard deviations for the TIC and TOC blanks is within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: No actual sample duplicates were identified on the ASR. However, the relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all values measured above the EQL, except for sample 00-0465, which has an RPD of 32% for TIC. Due to total quantity of sample material available, the quantity used for the duplicate analysis was significantly less than that used for the sample analysis (i.e., 0.007 g versus 0.043 g), and sample heterogeneity is the most likely reason for the poor precision.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. Matrix spikes were prepared from samples 00-0461 (liquid) and 00-0475 (solid), and the matrix spike recovery for both TIC and TOC were well within the acceptance criteria of 75% to 125% recovery. No effort was made to spike the samples that were acidic.

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

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.
- 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: M W Urie

Review/Approval by: D E Baldwin **Date** 4-5-00

Archive Information:

Files: ASR 5600 Fiskum.doc	ASR 5600 Fiskum Solids&Liquids.xls

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

Client: Sandra Fiskum
Project :
Work Pkg: W53400 (CMC K88409)
Analyzed: March 23, 2000
ASR: 5600

Analyzer M&TE: WA92040 -- 701
Balance M&TE: 360-06-01-023
TOC STD: Glucose CSM-53219>>> 40.00% Carbon <<[G]
TIC STD: CaCO3 CMS-139285>>> 11.99% Carbon <<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch)	16.7	36.6
	Calibration blank (start of batch)	15.9	41.9
	Calibration blank	12.3	46.0
	Calibration blank	13.1	35.6
	Calibration blank	14.9	41.0
	Calibration blank	11.9	35.7
	Calibration blank	14.5	41.2
	Calibration blank (end of batch)	13.1	44.0

TIC	TOC	
14.1	40.3	<<< Average (ug C)
1.7	3.9	<<< Std Dev (ug C)
2.16	5.8	<<< Pooled Std Dev (ug C)
6.5	17.3	<<< Method Det. Limit (ug C)

		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) 3/16	1599	14	0.01330	99.4	1190	40	0.00300	95.8
	Calibration Standard (start of batch) 3/16	1730	14	0.01480	96.7	1358	40	0.00340	96.9
	Calibration Standard	820	14	0.00670	100.3	1133	40	0.00270	101.2
	Calibration Standard (end of batch) 3/16	1640	14	0.01370	99.0	970	40	0.00240	96.8
	Calibration Standard (start of batch) 3/22	1238	14	0.01020	100.1	1770	40	0.00450	96.1
	Calibration Standard (start of batch) 3/22	1037	14	0.00840	101.6	893	40	0.00220	96.9
	Calibration Standard (start of batch) 3/23	1081	14	0.00920	96.7	1550	40	0.00390	96.8
	Calibration Standard	1208	14	0.01000	99.6	1295	40	0.00320	98.0
	Calibration Standard (end of batch) 3/23	1149	14	0.00950	99.6	1177	40	0.00290	98.0
		[L] Average TIC % Rec >>>>			99.2 <<[L]	[P] Average TOC % Rec >>>>			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) = (I-J)/(K*L/100)
Sample TOC (ug C/ml) = (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.

Sample Results

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam Vol (ml)	TIC (ug C/ml)	TIC RPD (%)	[M] Raw OC (ug C	[N] Blk (ug C)	[O] Sam Vol (ml)	TOC (ug C/ml)	TOC RPD (%)	TC (ug C/ml)	TC RPD (%)
00-00461	AN107 Starting material	840	14	0.10	8,324		1608	40	0.10	16,097		24,421	
00-00461DUP	AN107 Starting material	828	14	0.10	8,203	1	1608	40	0.10	16,097	0	24,301	0
00-00461 MS	AN107 Starting material	2625	14	0.10	see below		2540	40	0.10	see below		see below	
00-00463	TI-71-CaPS Filtrate	239	14	0.10	2,267		1332	40	0.10	13,263		15,530	
00-00464	TI-71-CaPS Filtrate Dup	244	14	0.10	2,317		1346	40	0.10	13,407		15,724	
00-00466	TI-71-CaPS NaOH wash -1	99	14	0.20	428		830	40	0.20	4,054		4,482	
00-00467	TI-71-CaPS NaOH wash -2	72	14	0.40	146		423	40	0.40	982		1,128	
00-00469	TI-71-CaPS -HNO3 wash -1	40	14	1.00	26		418	40	1.00	388		414	
00-00470	TI-71-CaPS -HNO3 wash -2	51	14	1.00	37		290	40	1.00	256		294	
00-00471	TI-71-CaPS H2O wash - 1	39	14	2.00	13		217	40	2.00	91		103	
00-00473	TI-71-BaSO4 filtrate	435	14	0.20	2,121		2650	40	0.20	13,398		15,519	
00-00474	TI-71-BaSO4 filtrate Dup	416	14	0.20	2,025		2537	40	0.20	12,818		14,843	
00-00476	TI-71-BaSO4 NaOH wash -1	309	14	0.40	743		1457	40	0.40	3,637		4,380	
00-00477	TI-71-BaSO4 NaOH wash -2	360	14	1.00	349		1025	40	1.00	1,011		1,360	
00-00479	TI-71-BaSO4 HNO3 wash - 1	15	14	0.20	5 (<mdl)		220	40	0.20	923		923	
00-00480	TI-71-BaSO4 HNO3 wash - 2	14	14	0.20	0 (<mdl)		130	40	0.20	461		461	
00-00481	TI-71-BaSO4 H2O wash - 1	13	14	1.00	-1 (<mdl)		192	40	1.00	156		156	

Matrix Spike Results

ACL Number	Client Sample ID	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/ml)	[T] MS Sam Vol (ml)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS Recovery	
00-00461 MS	TIC Recovery	2625	14	8324	0.1000	832	0.0148	1775	**101.4**	TIC
	TOC Recovery	2540	40	16097	0.1000	1610	0.0024	960	**99.7**	TOC
	Total Carbon Recovery (TIC + TOC)							2735	**100.8**	TC

Preparer/date: Marilyn Sheets 4/3/00

Reviewer/date: D L Blackburn 4/5/00

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

Client: Sandra Fiskum
Project :
Work Pkg: W48486 (CMC K88409)
Analyzed: March 23, 2000
ASR: 5600

Analyzer M&TE: WA92040 -- 701
Balance M&TE: 360-06-01-023
TOC STD: Glucose CSM-53219>>> 40.00% Carbon <<[G]
TIC STD: CaCO3 CMS-139285>>> 11.99% Carbon <<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch)3/23	13.1	35.6
	Calibration blank (start of batch)	14.9	41.0
	Calibration blank (end of batch)3/23	11.9	35.7
	Calibration blank (end of batch)3/23	14.5	41.2
	Calibration blank (end of batch)3/23	13.1	44.0

TIC	TOC	
13.5	39.5	<<< Average (ug C)
1.2	3.7	<<< Std Dev (ug C)
2.16	5.8	<<< Pooled Std Dev (ug C)
6.5	17.3	<<< Method Det. Limit (ug C)

		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	1238	14	0.01020	100.1	1770	40	0.00450	96.1
	Calibration Standard	1037	14	0.00840	101.6	893	40	0.00220	97.0
	Calibration Standard	1081	14	0.00920	96.8	1550	40	0.00390	96.8
	Calibration Standard	1208	14	0.01000	99.6	1295	40	0.00320	98.1
	Calibration Standard	1149	14	0.00950	99.7	1177	40	0.00290	98.1
		[L] Average TIC % Rec >>>>			99.6	[P] Average TOC % Rec >>>>			97.2

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/g) = (I-J)/(K*L/100)
Sample TOC (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: Sandra Fiskum
Project :
Work Pkg: W48486 (CMC K88409)
Analyzed: March 23, 2000
ASR: 5600

Analyzer M&TE: WA92040 -- 701
Balance M&TE: 360-06-01-023
TOC STD: Glucose CSM-53219>>> 40.00% Carbon <<[G]
TIC STD: CaCO3 CMS-139285>>> 11.99% Carbon <<[C]

Sample Results

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 OC (ug C	[N] Blk (ug C)	[O] Sam wt (g)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
00-00475	TI-71-BaSO4 ppt prior to wash	410	14	0.0162	24,581		415	40	0.0162	23,842		48,423	
00-00475 dup	TI-71-BaSO4 ppt prior to wash	660	14	0.0266	24,410	1	662	40	0.0266	24,071	1	48,481	0
00-00475 ms	TI-71-BaSO4 ppt prior to wash	1698	14	0.0185	see below		1509	40	0.0185	see below		see below	
00-00465	TI-71-CaPS ppt prior to wash	1575	14	0.0433	36,219		1175	40	0.0433	26,974		63,193	
00-00465 dup	TI-71-CaPS ppt prior to wash	368	14	0.0071	50,145	32	260	40	0.0071	31,944	17	82,089	26
00-00468	TI-71-CaCO3-1	840	14	0.0114	72,815		183	40	0.0114	12,947		85,762	
00-00468 dup	TI-71-CaCO3-1	763	14	0.0101	74,530	2	168	40	0.0101	13,086	1	87,616	2
00-00472	TI-71-CaCO3-2	782	14	0.0100	77,184		320	40	0.0100	28,852		106,036	
00-00472 dup	TI-71-CaCO3-2	1635	14	0.0210	77,551	0	585	40	0.0210	26,719	8	104,269	2
00-00478	TI-71-BaSO4 - 1	410	14	0.0132	30,168		305	40	0.0132	20,689		50,856	
00-00482	TI-71-BaSO4 - 2	35	14	0.0321	672		95	40	0.0321	1,778		2,450	
00-00482 dup	TI-71-BaSO4 - 2	29	14	0.0240	648	n/a	78	40	0.0240	1,650	n/a	2,298	n/a

Matrix Spike Results

ACL Number		[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
00-00475 ms	TIC Recovery	1698	13.5	24581	0.0185	455	0.0110	1319	93.8 TIC
	TOC Recovery	1509	39.5	23842	0.0185	441	0.0026	1040	102.9 TOC
	Total Carbon Recovery (TIC + TOC)							2359	97.8 TC

Preparer/date: M J Steele 3/24/00

Reviewer/date: D R McCalvin 4/5/00

HOT PERSULFATE WORKSHEET

Client Fiskum ASR 5600 Analyst MJ Steele Date 3/16/00 3/22/00

Procedure: PNL-ALO-381 Analyzer M&TE: WA92040--701 Balance M&TE: 360-06-01-023

TIC STD: Calcium Carbonate CMS-~~132985~~ 134285 mgk 3/23/00 TOC STD: Glucose CSM-53219

injection #	Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)		comments
1	3/16/00	Blank	—	—	16.7	36.6		T=20.0°C
2		↓	TIC	~~TOC~~	15.9	41.9		lg = 0.9998
3		CSM Std	.0133g	.0030	1599	1190		E109351
4		↓	.0148g	.0034	1730	1358		0.9937
5		00-461	.1ml	0.1245	840	1608	all mgk 4/13/00	0.9858
6		CSM Std	.0067g	.0027	820	~~332~~	1133	0.9944
7		00-461 Dup	0.1	0.1230	828	~~1346~~	1608	0.9928
8		00-463	0.1	0.1191	239	~~830~~	1332	111842
9		00-464	0.1	0.1217	244	~~423~~	1346	# ~~111842~~ mgk 4/13/00
10		00-466	0.2	0.2139	99	~~418~~	830	0.0998
11		00-467	0.4	0.4060	72	~~290~~	423	0.1000
12		00-469	1.0	1.0477	40	~~46~~	418	0.1001
13		00-470	1.0	1.0582	51		290	0.0999
14		Blank	—	—	123		46	
15	22 mgk 3/30	CSM Std	0.0137	0.0024	1640		970	
16	9/22/00	Blank		—	13.1	35.6		
17		CSM Std	0.0102	0.0045	1238	1770		T = ~~20.0~~ 21.6 °C mgk 4/13/00
18		CSM Std	0.0084	0.0022	1037	893	#E109351	lg = 0.9999
19		Blank	—	—	14.9	41.0	0.9939	#111842
20	00-471		2ml	2.0377	39.1	217	0.9928	0.1000
21	00-473		0.2	0.2442	435	2650	0.9946	0.0999
22		Std	.0093	0.0039	1081	1556	0.9899	0.0999
23		Blank			11.94	35.71		0.1001

5/23

2

HOT PERSULFATE WORKSHEET

3/23/00 mjs Page 2 of 2

injection #	Lab ID	Client ID	volume (ml)	sample wt (g)	TIC (ug)	TOC (ug)	comments
24	00-474		0.2	0.2428	416	2537	
25	00-476		0.4	0.4267	309	1457	
26	00-477		1.0	1.0216	360	1025	
27	00-479		0.2	0.2091	14.72	220	pH < 2
28	00-480		0.2	0.2057	13.72	130	
29	00-481		1.0	1.0200	12.96	192	↓ emf 4/3/00
30			SOLIDS				
31	00-465			0.0433	1575	1175	
32	Dup			0.0071	368	260	
33	00-468			0.0114	840	183	
34	Dup			0.0101	763	168	
35	Standard	0.0100 / 0.0032		—	1208	1295	
36	Blank	—		—	14.5	412	
37	00-461MS	Liquid (0.1ml) 0.0148 / 0.0024		0.1254	2625	2540	
38	00-475			0.0162	410	415	
39	Dup			0.0266	660	662	
40	00-472			0.0100	782	320	
41	Dup			0.0210	1635	585	
42	00-478			0.0132	410	305	insufficient Sample for Dup
43	00-482			0.0321	35	95	
44	Dup			0.0240	29	78	
45	00-475 MS	0.0110 / 0.0026		0.0185	1698	1509	
46	Blank				13.12	44.01	
47	Standard	0.0095 / 0.0029			1149	1177	
48							
49							
50							

PRELIMINARY RESULTS --- MW URIE 3/24/2000

Preliminary Mwlue 3/29/00

ALO Number	Sample ID	Wt (g)	TIC (ug C/g)	TIC RPD (%)	TOC (ug C/g)	TOC RPD (%)	TC (ug C/g)	TC RPD (%)
00-00465	TI-71-CaPS ppt prior to wash	0.0433	36,219		26,974		63,193	
00-00465 dup	TI-71-CaPS ppt prior to wash Dup (analysis) *	0.0071	50,145	32	31,944	17	82,089	26
00-00468	TI-71-CaCO3-1	0.0114	72,815		12,947		85,762	
00-00468 dup	TI-71-CaCO3-1Dup (analysis)	0.0101	74,530	2	13,086	1	87,616	2
00-00472	TI-71-CaCO3-2	0.0100	77,184		28,852		106,036	
00-00472 dup	TI-71-CaCO3-2 Dup (analysis)	0.0210	77,551	0	26,719	8	104,269	2
00-00475	TI-71-BaSO4 ppt prior to wash	0.0162	24,581		23,842		48,423	
00-00475 dup	TI-71-BaSO4 ppt prior to wash Dup (analysis)	0.0266	24,410	1	24,071	1	48,481	0
00-00475 ms	TI-71-BaSO4 ppt prior to wash MS Recovery	0.0185	94%		103%		98%	
00-00478	TI-71-BaSO4 - 1	0.0132	30,168		20,689		50,856	
00-00482	TI-71-BaSO4 - 2 **	0.0321	672		1,778		2,450	
00-00482 dup	TI-71-BaSO4 - 2 Dup (analysis)	0.0240	648	n/a	1,650	n/a	2,298	n/a

* Very small sample size for duplicate; results should be used with caution

** Insufficient sample for duplicate analysis

ALO Number	Sample ID	Vol	TIC (ugC/ml)	TIC RPD (%)	TOC (ugC/ml)	TOC RPD (%)	TC (ugC/ml)	TC RPD (%)
00-00461	TI-71-AN107	0.10	8324		16097		24,421	
00-00461 Dup	TI-71-AN107 Dup (analysis)	0.10	8203	1	16097	0	24,301	0
00-00461 MS	TI-71-AN107 Matrix Spike Recovery	0.10	101%		100%		101%	
00-00463	TI-71-CaPS Filtrate	0.10	2267		13263		15,530	
00-00464	TI-71-CaPS Filtrate Dup	0.10	2317		13407		15,724	
00-00466	TI-71-CaPS NaOH wash -1	0.20	428		4054		4,482	
00-00467	TI-71-CaPS NaOH wash -2	0.40	146		982		1,128	
00-00469	TI-71-CaPS -HNO3 wash -1	1.00	26		388		414	
00-00470	TI-71-CaPS -HNO3 wash -2	1.00	37		256		294	
00-00471	TI-71-CaPS H2O wash - 1	2.00	13		91		103	
00-00473	TI-71-BaSO4 filtrate	0.20	2121		13398		15,519	
00-00474	TI-71-BaSO4 filtrate Dup	0.20	2025		12818		14,843	
00-00476	TI-71-BaSO4 NaOH wash -1	0.40	743		3637		4,380	
00-00477	TI-71-BaSO4 NaOH wash -2	1.00	349		1011		1,360	
00-00479	TI-71-BaSO4 HNO3 wash - 1	0.20	<33		923		923	
00-00480	TI-71-BaSO4 HNO3 wash - 2	0.20	<33		461		461	
00-00481	TI-71-BaSO4 H2O wash - 1	1.00	<7		156		156	

Shaded samples pH <2; no TIC anticipated

MALLINCKRODT

CERTIFICATE OF ANALYSIS

A Division of Mallinckrodt Baker, Inc.
222 Red School Lane • Phillipsburg, NJ 08865
Telephone: (908) 859-2151 • Fax: (908) 859-9318

ITEM: CALCIUM CARBONATE (POWDER) CHELOMETRIC STANDARD ARR (ACS)
CODE: 4071
LOT : KTRT

TESTS	LIMITS	RESULTS
APPEARANCE	Fine, white powder	Fine, white powder
HEAVY METALS	0.001% Max.	0.0002%
IRON	0.002% Max.	0.002%
ALKALINITY	To Pass Test	Passes Test
CHLORIDE	0.001% Max.	0.0001%
INSOLUBLE IN HCl AND NH4OH PRECIPITATE	0.005% Max.	0.002%
SULFATE	0.005% Max.	0.004%
SILICA	0.001% Max.	0.0005%
OXIDIZING SUBSTANCES	0.005% Max.	0.002%
ASSAY	99.95 - 100.05%	100.05%
FLUORIDE	0.0015% Max.	0.0005%
BARIUM	0.005% Max.	0.002%
TRACE METALS		
MAGNESIUM (Mg)	0.01% Max.	0.002%
POTASSIUM (K)	0.01% Max.	0.001%
SODIUM (Na)	0.01% Max.	0.005%
SILICON (Si)	Report	0.0002%
STRONTIUM (Sr)	0.1% Max.	0.002%

It is hereby certified that the above is a true copy of the actual analysis of the lot indicated.

Mark Featherston

Mark Featherston
Manager, QA/QC
Mallinckrodt Baker, Inc
10/09/96 wwr

P.O. # 708706
4 X 125g containers
Accepted: _____
Date: 3/25/97

CMS # 139285

PRODUCT INFORMATION

CONTINUED FROM PREVIOUS PAGE

STARCH	PASS TEST
TITRATABLE ACID	<0.002 MEQ/G

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Aldrich Chemical Company
DAVID SWESSEL
NOVEMBER 7, 1991

aldrich chemical co.
chemists helping chemists in research & industry

PO# 170699
11-14-91 DB

CMS# 53219

PRODUCT INFORMATION

PRODUCT NUMBER: 25307-3 LOT NUMBER: 12603EY

PRODUCT NAME: ALPHA-D-GLUCOSE, A.C.S. REAGENT

FORMULA: C6H1206 FORMULA WEIGHT: 180.16

RECORD COPY

APPEARANCE	WHITE POWDER
INFRARED SPECTRUM	CONFORMS TO STRUCTURE AND STANDARD AS ILLUSTRATED ON PAGE 1908 OF EDITION I, VOLUME 1 OF "THE ALDRICH LIBRARY OF FT-IR SPECTRA".
OPTICAL ROTATION	+52.7 DEG (C=10% H2O + NH4OH)
LOSS ON DRYING	0.04%
RESIDUE ON IGNITION	<0.02%
INSOLUBLE MATTER	0.002 % (C=13.3%, H2O)
ARSENIC	<0.4PPM
CHLORIDE	<0.01%
IRON	<5 PPM
HEAVY METALS	<5PPM AS PB
SULFATE AND SULFITE	<0.005%

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accept
VN 11/18/91

CONTINUED ON NEXT PAGE

ALDRICH warrants that its products conform to the information contained in this and other Aldrich publications. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

D. Swessel
Aldrich Chemical Company
DAVID SWESSEL
NOVEMBER 7, 1991

aldrich chemical co.
P.O. Box 355, Milwaukee, Wisconsin 53201 USA • (414) 273-3850

Carbon -- Method Detection Limit Determination

The Method Detection Limit (MDL) for carbon by hot persulfate or furnace oxidation is estimated by taking the average of the standard deviations (SD) for blank measurements taken over a period from February 12, 1998 to June 3, 1998, and multiplying this average SD by 3. The estimated MDL is in µgC.

TOC = Total Organic Carbon; TIC = Total Inorganic Carbon; TC = Total Carbon

Hot Persulfate	File Seq #	Date	Project	ASR Number	Laboratory ALO Numbers	TIC SD	TOC SD
	95	02/12/98	17654	4593	98-02061/02063	0.7	13.1
	96	02/19/98	17654	4598	98-02099/02108	2.5	7.7
	98	03/04/98	17654	4625	98-02206/02207	1.5	1.4
	99	03/05/98	17654	4635, 4646	98-02305/02307	2.1	7.8
	103	03/27/98	17654	4692, 4693	98-02997/03005	0.8	3.7
	106	03/20/98	17654	4733, 4734	98-03287/03294	1.3	2.7
	108	04/22/98	27297	4701	98-03043	1.0	3.3
	109	04/24/98	25396	4588	98-02031	6.6	3.5
	111	05/18/98	17654	4799, 4804	98-03698/03699, 03713	1.7	3.3
	113	06/03/98	17654	4751, 4831	98-03413/03416, 03916/03917	3.4	11.5
					Average	**2.16**	**5.80**
					3xAverage = MDL	**6.5**	**17.4**

Furnace Oxidation	File Seq #	Date	Project	ASR Number	Laboratory ALO Numbers	TC SD
	97	03/03/98	17654	4637	98-02312/02313	2.6
	101	03/11/98	17654	4659	98-02535/02538	1.8
	102	03/25/98	17654	4689	98-02974/02975	1.8
	105	04/13/98	17654	4735	98-03295	2.7
	105	04/14/98	17654	4735, 4736,4738	98-03296, 03299, 03303, 03317	0.1
	105	04/15/98	17654	4739, 4741	98-03321/03323, 03327/03328	0.9
	105	04/16/98	17654	4742	98-03329/03330	3.0
	107	04/22/98	17654	4756	98-04355/04357	2.2
	110	05/06/98	17654	4786	98-03619	2.5
	112	05/21/98	26019	4794	98-03665	2.8
					Average	**2.04**
					3xAverage = MDL	**6.1**

Prepared By: Michael W. Thie

Battelle : acific Northwest Laboratory
Radiochemical Processing Group-325 Building

prop 29953
Task 12
T 2.12.5.2

00-0461

p 1/1

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 12/21/99

Concur : T Trang-Le Date : 12/21/99

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Procedure: PNL-ALO-450 *Gamma Energy Analysis*

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

ALO ID Client ID	Be-7 Error %	Co-60 Error %	Nb-95** Error %	Tc-95m** Error %	Y-88 Error %	Sn-113 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0461 TI-71-AN107	<1.E-3	4.04E-2 2%	1.12E-3 8%	2.45E-2 2%	6.73E-4 3%	4.11E-4 14%	4.90E-4 25%	3.10E-4 7%	6.71E-2 2%	2.66E-2 2%	1.89E-2 3%	9.68E-3 3%
00-0463 TI-71-CaPS-Filtrate	<1.E-3	3.41E-2 2%	1.04E-3 6%	2.18E-2 2%	1.67E-4 7%		<3.E-4	<2.E-4	6.09E-2 2%	4.97E-3 2%	3.65E-3 4%	1.72E-3 8%
00-0466 TI-71-CaPS-NaOH Wash-1	<3.E-4	1.12E-2 2%	3.20E-4 5%	7.66E-3 2%			<8.E-5	<4.E-5	2.12E-2 2%	6.48E-4 3%	4.49E-4 5%	2.41E-4 10%
00-0467 TI-71-CaPS-NaOH Wash-2	<2.E-4	2.54E-3 2%	7.46E-5 10%	1.83E-3 2%			<4.E-5	<2.E-5	5.40E-3 2%	<3.E-5	3.86E-5 19%	3.05E-5 32%
00-0469 TI-71-CaPS-HNO3 Wash-1	<1.E-4	6.08E-4 2%	3.29E-5 16%	5.85E-4 2%			<4.E-5	<2.E-5	2.53E-3 2%	1.21E-4 5%	<7.E-5	6.13E-5 47%
00-0470 TI-71-CaPS-HNO3 Wash-2	<9.E-5	8.02E-4 2%	<1.E-5	2.44E-4 7%	1.74E-4 3%	4.72E-5 12%	2.25E-4 6%	2.10E-5 11%	7.78E-4 2%	1.75E-3 2%	1.28E-3 3%	5.65E-4 7%
00-0471 TI-71-CaPS-H2O Wash-1	<3.E-5	2.78E-4 2%	<3.E-6	5.32E-5 5%	6.15E-5 2%	1.18E-5 16%	8.88E-5 5%	9.43E-6 8%	2.19E-4 2%	4.49E-4 2%	3.08E-4 4%	1.37E-4 7%
00-0473 * TI-71-BaSO4- Filtrate	<2.E-4	3.28E-2 2%	9.06E-4 2%	2.20E-2 2%	<2.E-4	4.63E-5 22%	<7.E-5	<3.E-5	6.08E-2 2%	2.57E-3 2%	1.80E-3 3%	1.12E-3 4%
00-0476 TI-71-BaSO4-NaOH Wash-1	<2.E-4	9.32E-3 2%	2.83E-4 5%	7.08E-3 2%			<6.E-5	<2.E-5	1.97E-2 2%	4.19E-4 3%	2.80E-4 6%	1.90E-4 15%
00-0477 TI-71-BaSO4-NaOH Wash-2	<6.E-5	2.38E-3 2%	8.29E-5 6%	1.84E-3 2%			<2.E-5	<8.E-6	5.17E-3 2%	5.96E-5 5%	3.60E-5 10%	3.96E-5 15%
00-0479 TI-71-BaSO4-HNO3 Wash-1	3.42E-4 23%	4.64E-3 2%	<6.E-5	4.41E-4 4%	4.61E-4 2%	6.00E-5 18%	<7.E-5	<3.E-5	1.31E-3 2%	1.73E-2 2%	1.19E-2 3%	3.99E-3 3%
00-0480 TI-71-BaSO4-HNO3 Wash-2	3.12E-4 24%	4.85E-3 2%	<5.E-5	1.10E-4 10%	2.49E-4 3%	2.34E-4 7%	<6.E-5	<3.E-5	3.44E-4 4%	1.10E-2 2%	7.55E-3 3%	3.27E-3 3%
00-0481 TI-71-BaSO4-H2O Wash-1	9.90E-5 18%	1.90E-3 2%	<2.E-5	3.42E-5 8%	8.77E-5 3%	7.69E-5 6%	<2.E-5	<6.E-6	1.89E-4 3%	3.82E-3 2%	2.69E-3 3%	1.29E-3 3%

* Sample 00-0473 also contains Se-75 at 6.73E-5 uCi/g +/-8%.
** as of November 1, 1999 12:00 PDT

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

00-0475

2/15/2000

Client : S. Fiskum

Cognizant Scientist: LR Greenwood Date : 2-15-00

Concur : T Trang-Le Date : 2/15/00

Reference Date: November 1, 1999, 13:00 pm

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

ALO ID Client ID	Be-7 Error %	Tc-95M Error %	Se-75 Error %	Co-60 Error %	Eu-152 Error %	Y-88 Error %	Cs-137 Error %	Sb-125 Error %	SnSb-126 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0475 T1-71-BaSO4 Pr	<2.E-2	3.85E-2 4%		1.03E-1 2%	<2.E-3	5.47E-3 7%	1.03E-1 2%	<3.E-3	<7.E-4	1.42E-1 2%	9.80E-2 3%	3.94E-2 8%
00-0478 T1-71-BaSO4-1	<3.E-2	3.65E-3 24%		1.02E-1 2%	<3.E-3	8.88E-3 5%	1.06E-2 6%	<3.E-3	<8.E-4	2.45E-1 2%	1.63E-1 3%	5.37E-2 8%
00-0482 T1-71-BaSO4-2	<7.E-3	<7.E-4	4.60E-4 24%	5.24E-2 2%	<7.E-4	1.46E-3 8%	1.64E-3 10%	<7.E-4	<3.E-4	1.93E-2 2%	1.09E-2 3%	4.38E-3 9%
00-0465 TI-71 CaPS pp1	<4.E-2	6.93E-2 4%		1.39E-1 2%	<3.E-3	7.99E-3 6%	1.96E-1 2%	3.77E-3 30%	2.65E-3 8%	2.32E-1 2%	1.70E-1 3%	8.85E-2 8%
00-0468 T1-71-CaCO3-1	<2.E-2	6.10E-3 16%		6.63E-2 2%	5.54E-3 9%	1.16E-2 3%	2.60E-2 2%	5.58E-3 12%	4.12E-3 4%	3.42E-1 2%	2.51E-1 3%	1.28E-1 7%
00-0472 T1-71-CaCO3-2	5.70E-2 19%	<4.E-3		1.25E-1 2%	1.24E-2 6%	2.27E-2 3%	8.14E-4 46%	9.36E-3 11%	1.00E-2 3%	7.58E-1 2%	5.38E-1 3%	2.75E-1 3%
00-0472DUP T1-71-CaCO3-2	7.32E-2 18%	<4.E-3		1.36E-1 2%	1.35E-2 6%	2.28E-2 3%	1.14E-3 40%	1.08E-2 10%	1.01E-2 3%	8.03E-1 2%	5.69E-1 3%	3.05E-1 3%
RPD	25%			8%	8%	0%	33%	14%	1%	6%	6%	10%

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

1/25/00

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1/25/00

Concur : T Trang-Le Date : 1/25/00

Procedure: PNL-ALO-4014

ALO ID Client ID	Uranium Measured Concentration (ug/ml) with 1-σ error Error %
00-0461PB Process Blank	4.27E-4 4%
00-0461 TI-71-AN107	8.17E+0 4%
00-0462 TI-71-AN107 Dup	8.33E+0 4%
RPD	2%
00-0463 TI-71-CaPS-Filtrate	6.11E+0 4%
00-0464 TI-71-CaPS-Filtrate Dup	6.06E+0 4%
RPD	1%
00-0465 PB Process Blank	4.44E-4 4%
00-0465 TI-71-CaPS-ppt prior to wash	3.16E+1 4%
00-0466 TI-71-CaPS-NaOH Wash-1	8.74E-1 4%
00-0467 TI-71-CaPS-NaOH Wash-2	1.56E-1 4%
00-0468 TI-71-CaCO3-1	3.85E+1 4%
00-0469 TI-71-CaPS-HNO3 Wash-1	6.95E-2 4%
00-0470 TI-71-CaPS-HNO3 Wash-2	6.65E-1 4%
00-0471 TI-71-CaPS-H2O Wash-1	1.66E-1 4%

Measured Concentration (ug/ml) with 1-σ error

ALO ID Client ID	Uranium Error %
00-0472 TI-71-BaSO4 Suspension	7.66E+1 4%
00-0472 -Dup TI-71-BaSO4 Suspension	8.91E+1 4%
RPD	15%
00-0473 TI-71-BaSO4- Filtrate	2.89E+0 4%
00-0474 TI-71-BaSO4 Filtrate Dup	2.87E+0 4%
RPD	1%
00-0476 TI-71-BaSO4-NaOH Wash-1	5.16E-1 4%
00-0477 TI-71-BaSO4-NaOH Wash-2	8.50E-2 4%
00-0479 TI-71-BaSO4-HNO3 Wash-1	2.08E+1 4%
00-0480 TI-71-BaSO4-HNO3 Wash-2	1.76E+1 4%
00-0481 TI-71-BaSO4-H2O Wash-1	6.17E+0 4%

	Standard	Observed	Expected	Yield
Before Run	Rec-319-4	1.02E-3	1.00E-3	**1.020**
	Rec-319-2-r	1.06E-1	1.00E-1	**1.060**
	Rec-319-2-r	1.04E-1	1.00E-1	**1.040**
	Blank	<2.E-5		
After Run	Rec-319-3-c	1.01E-2	1.00E-2	**1.010**
	Rec-319-3-c	9.97E-3	1.00E-2	**0.997**
	Rec-319-2-r	1.05E-1	1.00E-1	**1.050**
	Blank	<2.E-5		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1-14-00

Concur : T Trang-Le Date : 1/14/2000

Procedure: PNL-ALO-420/421

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

ALO ID Client ID	Alpha Error +/-	ALO ID Client ID	Alpha Error +/-
00-0461 TI-71-AN107	<3E-2	Blank Spike	114%
00-0463 TI-71-CaPS-Filtrate	<2.E-3	Blank	<2.E-6
00-0466 TI-71-CaPS-NaOH Wash-1	<7E-4		
00-0467 TI-71-CaPS-NaOH Wash-2	<3E-4		
00-0469 TI-71-CaPS-HNO3 Wash-1	<2E-2		
00-0469DUP TI-71-CaPS-HNO3 Wash-1	<2.E-2		
00-0470 TI-71-CaPS-HNO3 Wash-2	<2E-2		
00-0471 TI-71-CaPS-H2O Wash-1	<4E-3		
00-0473 TI-71-BaSO4- Filtrate	<2E-3		
00-0476 TI-71-BaSO4-NaOH Wash-1	<5.E-4		
00-0477 TI-71-BaSO4-NaOH Wash-2	<2E-4		
00-0479 TI-71-BaSO4-HNO3 Wash-1	3.13E-3 21%		
00-0480 TI-71-BaSO4-HNO3 Wash-2	3.84E-3 10%		
00-0481 TI-71-BaSO4-H2O Wash-1	1.45E-3 9%		

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1/14/00

Concur : T Traina-le Date : 1/14/2000

Procedure: PNL-ALO-476 Sr90

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

ALO ID Client ID	Sr-90 Error +/-
00-0461 TI-71-AN107	2.01E-1 3%
00-0463 TI-71-CaPS-Filtrate	6.49E-3 3%
00-0466 TI-71-CaPS-NaOH Wash-1	3.51E-3 3%
00-0467 TI-71-CaPS-NaOH Wash-2	2.77E-3 3%
00-0469 TI-71-CaPS-HNO3 Wash-1	6.53E-1 3%
00-0470 TI-71-CaPS-HNO3 Wash-2	3.18E-1 3%
00-0471 TI-71-CaPS-H2O Wash-1	3.36E-2 3%
00-0473 TI-71-BaSO4- Filtrate	2.01E-3 3%
00-0476 TI-71-BaSO4-NaOH Wash-1	8.06E-4 3%
00-0477 TI-71-BaSO4-NaOH Wash-2	4.77E-4 3%
00-0479 TI-71-BaSO4-HNO3 Wash-1	2.59E-2 3%
00-0479 DUP TI-71-BaSO4-HNO3 Wash-1	2.58E-2 3%
RPD	1%
00-0480 TI-71-BaSO4-HNO3 Wash-2	2.64E-2 3%

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

ALO ID Client ID	Sr-90 Error +/-
00-0481 TI-71-BaSO4-H2O Wash-1	8.60E-3 3%
00-0481DUP TI-71-BaSO4-H2O Wash-1	9.13E-3 3%
RPD	6%
Blank Spike*	105% 109%
Matrix Spike*	103% 126%
Blank *	3.34E-5 31%
Blank*	1.35E-4 14%

*Samples were analyzed in two batches.

File: 00-0461.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1/14/00

Concur : T Trang-Le Date : 1/17/00

Procedure: PNL-ALO-420/421

Measured Activities (uCi/~~ml~~ g) with 1-σ error

LRG 2-2-00

ALO ID Client ID	Alpha Error +/-
00-0465 TI-71-CaPS ppt prior to Wash	5.15E-2 17%
00-0468 TI-71-CaCO3-1	<4E-1
00-0472 TI-71-CaCO3-2	3.50E-1 27%
00-0475 BF+SF TI-71 BaSO4 prior to wash	6.36E-2 16%
00-0478 BF+SF TI-71-BaSO4-1	1.12E-1 12%
00-0482 BF+SF TI-71-BaSO4-2	1.17E-2 8%
Blank Spike	114%
Blank	<2.E-6

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1/11/00

Concur : T Trang-Le Date : 1/11/2000

Procedure: PNL-ALO-476 Sr90

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

ALO ID Client ID	Sr-90 Error +/-	
00-0461 TI-71-AN107	2.01E-1 3%	
00-0463 TI-71-CaPS-Filtrate	6.49E-3 3%	
00-0466 TI-71-CaPS-NaOH Wash-1	3.51E-3 3%	
00-0467 TI-71-CaPS-NaOH Wash-2	2.77E-3 3%	
00-0469 TI-71-CaPS-HNO3 Wash-1	6.53E-1 3%	
00-0470 TI-71-CaPS-HNO3 Wash-2	3.18E-1 3%	
00-0471 TI-71-CaPS-H2O Wash-1	3.36E-2 3%	
00-0473 * TI-71-BaSO4- Filtrate	2.43E-3 3%	Note: * sample reported in uCi/ml.
00-0476 TI-71-BaSO4-NaOH Wash-1	8.06E-4 3%	
00-0477 TI-71-BaSO4-NaOH Wash-2	4.77E-4 3%	
00-0479 TI-71-BaSO4-HNO3 Wash-1	2.59E-2 3%	
00-0479 DUP TI-71-BaSO4-HNO3 Wash-1	2.58E-2 3%	
RPD	1%	
00-0480 TI-71-BaSO4-HNO3 Wash-2	2.64E-2 3%	

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

ALO ID Client ID	Sr-90 Error +/-
00-0481 TI-71-BaSO4-H2O Wash-1	8.60E-3 3%
00-0481DUP TI-71-BaSO4-H2O Wash-1	9.13E-3 3%
RPD	6%
Blank Spike**	105% 109%
Matrix Spike**	103% 126%
Blank **	3.34E-5 31%
Blank**	1.35E-4 14%

*Samples were analyzed in two batches.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: L R Greenwood Date : 1/19/00

Concur : T Trang-Le Date : 1/19/2000

Procedure: PNL-ALO-476 Sr-90

Measured Activities (uCi/ml) with 1-σ error

ALO ID Client ID	Sr-90 Error +/-
00-0465 TI-71-CaPS ppt prior to Wash	8.93E+0 3%
00-0468 TI-71-CaCO3-1	1.42E+1 3%
00-0472 TI-71-CaCO3-2	8.94E+0 3%
00-0475 BF+SF TI-71 BaSO4 prior to wash	6.69E-1 4%
00-0478 BF+SF TI-71-BaSO4-1	1.03E+0 4%
00-0482 BF+SF TI-71-BaSO4-2	<6.E-3
Matrix Spike*	126% 99%
Reagent Spike*	109% 106%
Blank for CaCO3 batch	1.02E-2 14%
Blank for sulfate batch	2.27E-1 3%

*Note: Samples were analyzed in two batches.

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329 File
Mike Urie

Date December 22, 1999

To Sandra Fiskum

From Tom Farmer

Subject ICPMS Analysis BNFL samples
(ALO# 00-00461 throught 00-00481)

Pursuant to your request, the 24 samples that you submitted for analysis were analyzed on our radioactively-contained ICPMS for the selected analytes. The concentration results for the isotopes of interest are displayed on the attached spreadsheet.

Dilutions of Isotope Products standards for ^{237}Np and ^{239}Pu, an Amersham ^{99}Tc standard were used to generate the calibration curves. Independent standards, from the same vendors, of each analyte were used as the continuing calibration verification (CCV) standards. Duplicates and a spiked samples were also analyzed. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. From the appearance of the Ru isotopic abundance, this appears to be a reasonable assumption; the fingerprint exhibited is obviously not natural.

A uranium hydride interference correction was performed for ^{239}Pu.

The results include your dilutions and are reported in ng analyte/ ml (ppb) of original sample ± one standard deviation. Results for samples 00-00465, 00-00468, 00-00472 and 00-00472DUP are reported in ng analyte/g (ppb) of submitted sample ± one standard deviation.

If you have any questions regarding this analysis, please give me a call at 372-0700 or James Bramson at 376-0624.

J P Brown 1/10/00

Fiskum Analysis

December 22, 1999

Results are reported in ng analyte/ ml of original solution.

Sample ID	Client ID	ICP/MS Number	*^{99}Tc ng/ml ± 1SD	^{237}Np ng/ml ± 1SD	^{239}Pu ng/ml ± 1SD
1%HNO3		9c20a1	0.035±0.005 0.005	<0.03	<0.01
1%HNO3		9c20a10	<0.024	<0.03	<0.01
1%HNO3		9c20a45	<0.024	<0.02	<0.008
00-461PB	Process Blank	9c20a11	2.0 ± 0.6	<0.32	<0.13
00-465PB	Process Blank	9c20a12	0.30 ± 0.21	<0.32	<0.13
00-461	TI-71-AN107	9c20a14	3450 ± 160	60.1 ± 1.8	24 ± 4
00-462	TI-71-AN107 Dup	9c20a15	3490 ± 110	58.1 ± 2.4	24 ± 10
00-463	TI-71-CaPs-filtrate	9c20a16	3080 ± 80	31.5 ± 0.7	11 ± 1.0
00-464	TI-71-CaPs-filtrate Dup	9c20a17	3400 ± 220	34.1 ± 2.2	11 ± 2
†00-465	TI-71-CaPS-ppt-prior to wash	9c20a38	†8410 ng/g ± 290	†233 ng/g ± 11	†137 ng/g ± 4
00-466	TI-71-CaPS-NaOH wash-1	9c20a39	850 ± 30	5.17 ± 0.33	1.8 ± 0.4
00-467	TI-71-CaPS-NaOH wash-2	9c20a40	184 ± 10	<0.42	0.33 ± 0.20
†00-468	TI-71-CACO3-1	9c20a21	†1720 ng/g ± 250	†280 ng/g ± 20	†154 ng/g ± 9
00-469	TI-71-CaPS-HNO3 wash-1	9c20a41	49.4 ± 3.2	<0.44	<0.18
00-470	TI-71-CaPS-HNO3 wash-2	9c20a42	41.5 ± 4.0	0.62 ± 0.21	0.67 ± 0.03
00-471	TI-71-CaPS-H2O wash-1	9c20a36	13.5 ± 1.0	<0.33	0.32 ± 0.56
†00-472	TI-71-CaCO3-2	9c20a25	†2810 ng/g ± 99	†588 ng/g ± 27	†379 ng/g ± 11
†00-472DUP	TI-71-CaCO3-2 Dup.	9c20a26	†2900 ng/g ± 30	†657 ng/g ± 30	†334 ng/g ± 12
00-473	TI-71-BaSO4-filtrate	9c20a27	2860 ± 50	17 ± 2	4.4 ± 0.8
00-473REP	TI-71-BaSO4-filtrate Rep.	9c20a28	2770 ± 140	18 ± 5	6.6 ± 1.0
00-474	TI-71-BaSO4-filtrate Dup.	9c20a29	2950 ± 50	17 ± 3.0	7.0 ± 1.6
00-476	TI-71-BaSO4-NaOH wash-1	9c20a37	769 ± 12	3.2 ± 0.4	1.2 ± 1.0
00-477	TI-71-BaSO4-NaOH wash-2	9c20a44	190 ± 20	0.50 ± 0.11	0.18 ± 0.08
00-479	TI-71-BaSO4-HNO3 wash-1	9c20a32	141 ± 7	99.7 ± 5.7	27.7 ± 1.2
00-480	TI-71-BaSO4-HNO3 wash-2	9c20a33	105 ± 0.2	38 ± 4	21 ± 4
00-481	TI-71-BaSO4-H2O wash-1	9c20a34	35.6 ± 2.7	13.4 ± 0.4	6.4 ± 1.1
00-481 + spike	TI-71-BaSO4-H2O wash-1	9c20a35	162 ± 0.6	62.9 ± 2.0	63.3 ± 3
Spike Recovery			**101%**	**99%**	**114%**
5ppb Tc-99		9c17a4	4.72 ± 0.15		
5ppb Tc-99		9c17a19	5.13 ± 0.03		
5ppb Tc-99		9c17a52	4.79 ± 0.39		
100ppb Co		9c17a53	<0.024		
1ppb Np CCV		9c20a6		0.996 ± 0.049	
1ppb Np CCV		9c20a47		0.968 ± 0.080	
1ppb Pu CCV		9c20a7			1.02 ± 0.09
1ppb Pu CCV		9c20a48			0.979 ± 0.013

*Results are from procedure 9c17a.

†Results are reported in ng analyte/g sample.

DATA REVIEW

Reviewed by: DJ. James III

Date: 10Jan00 Pages. 1 of 1

File: 00-0461.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: J R Greenwood Date : 3/23/00

Concur : T Trang-Le Date : 3/23/00

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

ALO ID Client ID	Pu-239 + Pu-240 Error +/-	Pu-238 Error +/-	Pu-236 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	Cm-242 Error +/-
00-0461 TI-71-AN107	1.19E-3 5%	3.21E-4 8%	<3.E-6	9.91E-3 5%	4.06E-4 8%	4.26E-5 20%
00-0472 TI-71-CaCO3-2	2.32E-2 5%	7.21E-3 8%	<2.E-4	2.71E-1 5%	1.05E-2 7%	1.17E-3 17%
00-0482 BF+SF TI-71-BaSO4-2	5.29E-3 4%	1.35E-3 5%	<3.E-6	4.38E-3 5%	1.71E-4 11%	1.66E-5 17%
Matrix Spike	104%			90%		
Reagent Spike	107%			97%		
Blank	<6.E-6	<2.E-5	<2.E-5	<8.E-6	<4.E-6	<4.E-6

File: 00-0461.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

1/13/2000

Client : Fiskum

Cognizant Scientist: _____ Date : 1-13-00

Concur : _____ Date : 1-13-00

Procedure: PNL-ALO-440

ALO ID Client ID	Measured Concentration (uCi/g) with 1-σ error Se-79 Error %
00-0461PB Process Blank	<2.E-6
00-0461* TI-71-AN107	1.23E-5 24%
00-0462* TI-71-AN107 Dup	1.36E-5 30%
00-0463 TI-71-CaPS-Filtrate	1.51E-5 3%
00-0464 TI-71-CaPS-Filtrate Dup	1.18E-5 5%
00-0468 TI-71-CaCO3-1	<9.E-5
00-0472 TI-71-CaCO3-2	<1.E-4
00-0473 TI-71-BaSO4- Filtrate	<1.E-6
00-0474 TI-71-BaSO4 Filtrate Dup	<2.E-6

*Note: The beta energy spectra for samples 00-0461 and 00-0462 do not show a peak for Se-79. Hence, although counts were observed above background, Se-79 is most likely not present in these samples. Samples 00-0463 and 00-0464 clearly show peaks in the beta energy spectra indicative of Se-79.

File: 00-0461.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

Client : Fiskum

Cognizant Scientist: C Soderquist Date : 2-18-00

Concur : R Greenwood Date : 2-18-00

Measured Concentrations (ug/g) with 1-σ error

ALO ID Client ID	Ammonia Error +/-
00-0465 TI-71-CaPS ppt prior to Wash	3.75 11%
00-0468 TI-71-CaCO3-1	< 5.0
00-0472 TI-71-CaCO3-2	< 4.
00-0472 Dup TI-71-CaCO3-2	< 4.
00-0475 BF + SF TI-71 BaSO4 prior to wash	< 6.
00-0478 BF + SF TI-71-BaSO4-1	< 18.
00-0480 TI-71-BaSO4-HNO3 Wash-2	0.24 18%
00-0482 BF + SF TI-71-BaSO4-2	< 4.

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