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Prepared for British Nuclear Fuels Ltd., Inc.
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Summary

Hanford low-activity waste solutions contain sulfate, which can cause accelerated corrosion of the vitrification melter and unacceptable operating conditions. A method is needed to selectively separate sulfate from the waste. An experiment was conducted to evaluate evaporation for removing sulfate ion from Tank AN-107 low-activity waste. Two evaporation steps were performed. In the first step, the volume was reduced by 55% while in the second step, the liquid volume was reduced another 22%. Analysis of the solids precipitated during these evaporations revealed that large amounts of sodium nitrate and nitrite co-precipitated with sodium sulfate. Many other waste components precipitated as well. It can be concluded that sulfate removal by precipitation is not selective, and thus, evaporation is not a viable option for removing sulfate from the AN-107 liquid.

Acronyms

BNFL	British Nuclear Fuels, Ltd.
GEA	gamma energy analysis
IC	ion chromatography
ICP	inductively coupled plasma
ICP/AES	inductively coupled plasma/atomic emission spectroscopy
LAW	low-activity waste
MRQ	Minimum reportable quantity
SRTC	Savannah River Technology Center
TRU	transuranic
VSL	Vitreous States Laboratory

Contents

1.0	Introduction	1.1
2.0	Experimental.....	2.1
3.0	Results and Discussion.....	3.1
4.0	Conclusion.....	4.1
5.0	References	5.1
	Appendix A: Test Instruction.....	A.1
	Appendix B: Raw Data	B.1
	Appendix C: Calculations	C.1

Figure

Figure 3.1.	Schematic Representation of the AN-107 Evaporation Test	3.2
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Tables

Table 3.1.	Analyte Concentrations in Starting Archive AN-107 Sample, Evaporation Concentrates, and Precipitate.....	3.3
Table 3.2.	Mass Balances for Detected Analytes	3.5

1.0 Introduction

The presence of sulfate ion in the Hanford low-activity waste (LAW) solutions at up to 0.07 mole per mole sodium (Contract number DE-AC27-96RL13308, Modification 14, Specification 7) generates several potential processing difficulties. Preliminary testing of the LAW vitrification system at the Vitreous States Laboratory (VSL) indicates that 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 SO₂ or SO₃. BNFL is evaluating four pretreatment technologies for separating sulfate from LAW solutions:

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

Savannah River Technology Center (SRTC) personnel have conducted tests with LAW Envelope B simulant to demonstrate sulfate separation using evaporation, precipitation, and low-temperature crystallization. IBC Technologies personnel have conducted tests with a range of simulants to demonstrate sulfate separation using SuperLig® 655 resin. Additional tests are required with radioactive and other simulated LAW solutions to evaluate these sulfate pretreatment technologies.

This report describes the results of an experiment using actual AN-107 waste to evaluate evaporation for removal of sulfate ion from the Tank AN-107 LAW (Envelope C) fraction. The test also provides information on the degree to which AN-107 can be concentrated without solids formation. Information on the evaporation of the pretreated AN-107 waste is valuable for understanding storage conditions for the pretreated waste.

2.0 Experimental

Sample Description. The sample used in this test was derived from an AN-107 archive sample. This material was collected and processed to remove cesium before its use for the BNFL project (Hendrickson 1997). It was collected as 45 grab samples in 125-mL bottles taken during January 1997. Approximately 5.4 L of in-tank material was transferred to the 222-S laboratory and 0.53 M sodium hydroxide was added to dilute the waste to 5 M sodium and adjust the free hydroxide concentration to 0.24 M. Solids were separated from the supernatant by settling. The supernatant was decanted and passed through small columns containing cesium-selective crystalline silicotitanate material. Analysis of the waste after cesium removal indicated the free hydroxide concentration to be 0.126 M. Following cesium removal, the sample was transferred to the Pacific Northwest National Laboratory in five 1-L poly bottles where it has been stored in the Shielded Analytical Laboratory hot cells in the Radiochemical Processing Laboratory.

As part of an effort to prepare feed for sulfate-removal tests, a subsample of the AN-107 archive sample was further treated to remove Sr/transuranic (TRU) components (Hallen et al. 2000) by precipitation. This activity involved adjusting the subsample to a hydroxide concentration of 0.8 M with the addition of 19 M NaOH. A solution of 1 M strontium nitrate was added to provide a Sr concentration of 0.05 M, and a solution of 1 M sodium permanganate was added to provide a permanganate concentration of 0.03 M. The precipitated solids were separated using a 0.45-micron dead-end filter. The clarified liquid was used for the tests described in this report.

Apparatus. The apparatus used consisted of a Dataplate® Digital Hot Plate/Stirrer Series 710 coupled to a Dataplate® Multi-Controller Model 740 (PMC Industries, San Diego, California).

Procedure.^(a) An aliquot (20 mL) of the pretreated AN-107 sample was set aside for analysis. The rest of the sample (101 mL, 122.2 g) was placed into a 500-mL beaker equipped with a stir bar and marked at the 45-mL level (this was the target volume for the first evaporation step). The beaker was placed on the hot plate/stirrer and stirring was begun. The hot-plate temperature was ramped up to 115°C over a period of ~1 h. (Note: The temperature of the solution in the beaker was not measured, but was likely ~20 to 30°C less than the plate temperature.) The solution was evaporated to a volume of 45 mL; this process took 2.5 to 3 h. The evaporation proceeded smoothly with no foaming. No solids were observed to form as the volume was reduced to about 50 mL. Precipitation of solids occurred as the volume was further reduced to 45 mL.

The concentrated slurry was stirred at ambient temperature (~23°C) for about 20 h, and the weight was then determined to be 59.6 g. The concentrated slurry was filtered through a 0.45-µm-nylon membrane. The filtration proceeded very slowly, with 45 to 60 min required to filter the entire slurry.

^(a) The test instruction and the associated procedural notes are included as Appendix A to this report. These tests were conducted per the instructions provided in Test Specification TSP-W375-99-00012 Rev 0, Test Specification for Evaluating Sulfate Separation from LAW Solutions, September 13, 1999.

Since a significant amount of the solids remained in the 500-mL beaker, the filtrate was used to rinse the beaker. The resulting rinse slurry was passed through the same filter membrane to collect additional solids and the clarified liquid. Because the concentrated sample was very viscous, weight losses occurred during the filtration process. The weight of slurry actually filtered was 57.6 g (compared to 59.6 g total), and the loss of material can be attributed to the residual remaining in the beaker. The weight of the clarified liquid was 32.3 g while that of the filtered solids was 25.2 g.

The solids were divided between two 20-mL glass vials (the volume of solids was too great to fit into one vial) and dried overnight at 105°C. The solids in one of the vials (AN107-Solid1) did not appear (visually) to be completely dry. This was supported by the fact that only a 24.6 wt% loss was measured for vial AN107-Solid1 compared to a 33.6 wt% loss for the other vial (AN107-Solid1B). The amount of material in AN107-Solid1B was less than in AN107-Solid1, and consequently, drying occurred more rapidly. Using the larger water loss value of 33.6 wt%, the total weight of dried solids was determined to be 16.7 g. Because it was more thoroughly dried, an aliquot (0.189 g) was taken from Vial AN107-Solid1B for analysis. The aliquot of solid was rapidly dissolved in 20 mL of deionized water at room temperature.

The clarified liquid concentrate was transferred to a graduated cylinder, and the volume was determined to be 22 mL. The liquid was then returned to the filter flask. A 10-mL aliquot of the liquid in the filter flask was set aside for analysis. The remaining 12 mL was transferred to a 100-mL beaker equipped with a stir bar and marked at the 9-mL level (this was the target volume for the second evaporation step). The sample was evaporated to 9-mL (12.5 g) and filtered in a manner similar to that described above. The clarified liquid weighed 5.0 g and had a volume of 3 mL. The wet solids were dried overnight at 105°C (4.2 g of dried solids recovered; 20.2 wt% loss on drying). A 0.242-g aliquot of the dried solids was dissolved for analysis in 20 mL of deionized water at room temperature.

The untreated sample, the first and second concentrated slurries, and the first and second precipitates were subjected to the following analytical procedures: ion chromatography (IC) (anions), acid digestion, inductively coupled plasma/atomic emission spectroscopy (ICP/AES), ⁹⁰Sr, total alpha, and gamma energy analysis (GEA).

3.0 Results and Discussion

The process steps used in the evaporation tests are summarized in Figure 3.1. The actual sample volumes and masses are provided along with values corrected for holdup losses and sampling. The corrected values are provided to more accurately calculate a mass balance and represent an actual process flowsheet. Details of these calculations and the assumptions may be found in Appendix C.

The concentrations of the analytes in the starting archive AN-107 sample, the liquid concentrates, and the solid precipitates are shown in Table 3.1 along with the BNFL-specified minimum reportable quantities (MRQs). The concentrations in the solids are given as $\mu\text{g/g}$ of solid precipitate on a dry basis (i.e., dried at 105°C). The boron and silicon found in the samples are believed to be due primarily to contamination from the borosilicate glass used for sample storage and handling. Some of the zinc and calcium found in the samples is believed to be due to laboratory contamination during sample preparation since both of these analytes were detected in the preparatory blank. The values reported for the metals determined by ICP appear low for the second filtered concentrate. A review of the sample preparation bench sheets indicates this is likely because of a mass or volume recording error. Fluoride reported in the samples is likely due to organic anion interference. A high probability exists that there is no fluoride in the samples. All specified MRQs, except those for chloride and phosphate, were readily met.

A comparison of the solids compositions indicates that there is little, if any, selective precipitation of sulfate. As seen in Table 3.1, this is indicated by the similarity of the two solids compositions. Although the sulfate concentration does increase in the solids from the second evaporation, it is still a relatively small fraction of the total precipitated salts. An examination of the major components in the solids indicates that the precipitate consists largely of sodium nitrate, nitrite, and sulfate salts.

The mass balances for each analyte, corrected for holdup and subsampling, are summarized in Table 3.2. The sulfate mass balance for the first concentration step is very good and is reasonably good for the second concentration step. Approximately $\frac{2}{3}$ of the sulfate remained in the filtrate after the first concentration step. The analytical results only accounted for $\sim 91\%$ of the sulfate in the second concentration step; normalizing the results to 100% gives 15% in the remaining liquid and 85% in the solids for a total removal of 86% for the 93% volume reduction.^(b)

The nitrate mass balance for the first concentration step was excellent, and the mass recovery was somewhat high for the second concentration step. The first concentration produced a solid that contains approximately 60% of the nitrate of the pretreated AN-107 archive sample. The second concentration step produced a solid that contains approximately 80% of the remaining nitrate or approximately 32% of the nitrate originally present in the pretreated AN-107 archive sample. The two steps removed 92% of the nitrate for a 93% sample-volume reduction.^(b)

^(b) The removal values should be viewed as maximum values, since they do not take into account any material dissolved in the interstitial liquid of the wet solids.

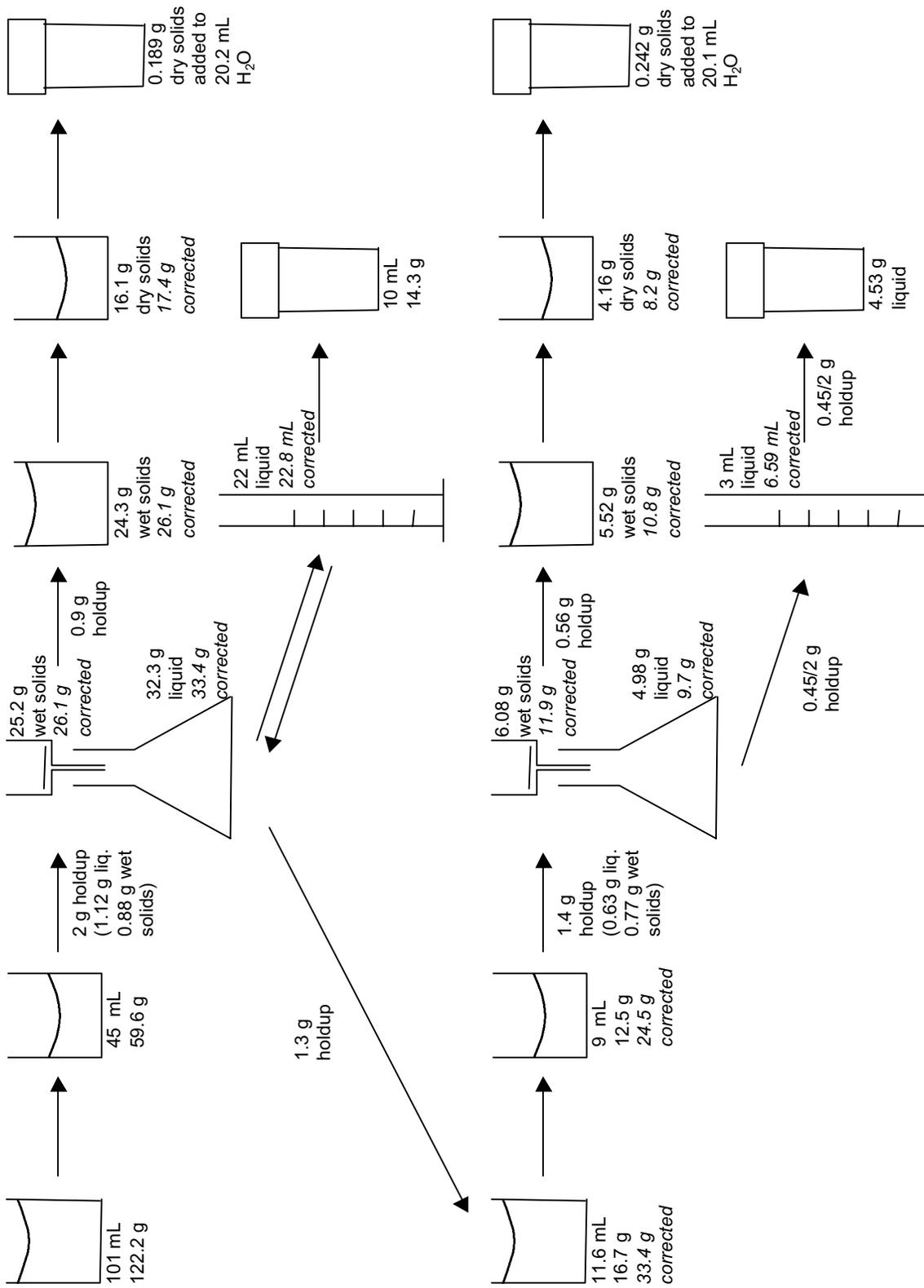


Figure 3.1. Schematic Representation of the ANI07 Evaporation Test

Table 3.1. Analyte Concentrations in Starting Archive AN-107 Sample, Evaporation Concentrates, and Precipitate

Analyte	Initial Sample	Filtered Concentrate, First Evaporation	Filtered Concentrate, Second Evaporation	Solids, First Evaporation	Solids, Second Evaporation	MRQs
Metals Determined by ICP						
	µg/mL	µg/mL	µg/mL	µg/g	µg/g	µg/mL
Al	132	412	346	236	223	75
Ba	<0.082	<0.36	<0.38	<2	2.8	78
Ca	161	507	372	460	503	150
Cd	26.3	81	76.2	48.6	49.6	7.5
Co	2.06	6.57	6.02	<10.6	<8.3	30
Cr	13.1	41.4	38.4	23.5	24.9	15
Cu	17.8	53.3	47.6	32.1	29.1	17
Fe	3.11	9.29	7.13	<5.3	4.73	150
K	718	2,230	2,140	1,180	1,250	75
La	<0.41	<1.8	<1.9	<10.6	<8.3	35
Mn	2.06	2.86	<1.9	<10.6	<8.3	150
Na	113,000	256,000	139,000	320,000	297,000	75
Ni	220	702	656	435	432	30
Pb	76.1	240	227	139	150	300
Si	100	340	190	1960	1550	170
Sn	<12	<55	<57	<320	<250	1,500
Ti	<0.21	<0.91	<0.95	<5.3	<4.1	17
U	41.1	123	<76	<420	<330	600
Zn	5.72	22.9	17.4	56.6	19.9	165
B	20.9	73.5	55.8	586	422	--
Mo	15	47.6	43.7	26.7	28.2	--
Nd	1.33	<3.6	<3.8	<21	<17	--
P	201	604	372	333	453	--
Sr	90.5	273	170	174	286	--
W	72.6	229	222	<420	<330	--
Zr	1.21	5.43	4.44	<10.6	<8.3	--
Anions						
Cl ⁻	930	2,100	5,600	<2,600	<2,100	3
F ⁻	3,600	9,500	19,000	8,600	7,500	150
NO ₂ ⁻	28,000	81,000	151,000	57,700	50,700	--
NO ₃ ⁻	111,000	208,000	170,000	385,000	482,000	3,000
SO ₄ ⁻	4,000	11,000	5,200	7,700	19,100	2,300
PO ₄ ⁻	1,400	2,100	3,300	<5,300	<4,100	2,500

Table 3.1. Continued

Radionuclides						
	μCi/mL	μCi/mL	μCi/mL	μCi/g	μCi/g	
⁹⁰ Sr	7.165E-01	1.88	2.16	1.27	2.23	1.5E-01
¹³⁷ Cs	2.67E-02	7.74E-02	1.49E-02	4.87E-02	5.04E-02	9E+00
²⁴¹ Am	4.52E-03	1.11E-02	1.92E-02	1.04E-02	9.8E-03	7.2E-04
total alpha	5.2E-03	8.7E-03	1.66E-02	1.19E-2	1.5E-02	2.3E-01
⁶⁰ Co	5.72E-02	1.63E-01	3.07E-01	1.1E-01	1.1E-01	--
¹²⁵ Sb	5.62E-04	9.54E-04	1.32E-03	<3.2E-03	<1.7E-03	--
¹²⁶ SnSb	2.87E-04	6.56E-04	1.51E-03	<1E-03	6E-04	--
¹⁵⁴ Eu	1.1E-02	2.42E-02	3.62E-02	2.89E-02	2.35E-02	--
¹⁵⁵ Eu	7.95E-03	1.75E-02	2.66E-02	2.24E-02	1.71E-02	--

The nitrite mass balance for the first concentration was excellent, and the mass recovery was somewhat low for the second concentration step. Approximately $\frac{2}{3}$ of the nitrite remained in the filtered liquid after the first concentration step. The analytical results only accounted for ~65% of the nitrite in the second concentration step; normalizing the results gives 15% in the remaining liquid and 85% in the solids for a total removal of 86% for the 93% volume reduction.

Chloride and phosphate were detected only in the liquid fractions of both concentrates, so no mass balance could be made.

The mass balance for both the metals and the radionuclides was excellent for the first concentration steps, but the mass recovery was low for the second concentration step. This is attributed to a mass or volume recording error during sample preparation resulting in apparently low concentrations for the filtered concentrate from the second evaporation.^(a)

Table 3.2. Mass Balances for Detected Analytes

	AN107-1 Filtered Liquid (%)	AN107-PPT1 Dry solids (%)	Total Recovery 1 st Volume Reduction (%)	AN107-2 Filtered Liquid (%)	AN107-PPT2 Dry Solids (%)	Total recovery 2 nd Volume Reduction (%)
Cl ⁻	50.9	(a)	(a)	77.2	(a)	(a)
SO ₄ ⁻²	61.9	33.1	95.0	13.7	77.4	91.1
NO ₃ ⁻	42.2	59.6	101.8	23.7	103.2	126.9
NO ₂ ⁻	65.2	35.4	100.6	54.0	27.9	81.9
PO ₄ ⁻³	47.8	(a)	(a)	21.8	(a)	(a)
⁶⁰ Co	64.2	32.8	97.0	54.6	29.7	84.3
¹²⁵ Sb	38.2	(a)	(a)	40.1	(a)	(a)
¹²⁶ Sn & ¹²⁶ Sb	51.5	(a)	(a)	66.7	41.3	108.0
¹³⁷ Cs	65.3	31.4	96.7	55.8	29.0	84.8
¹⁵⁴ Eu	49.6	45.1	94.7	43.3	43.3	86.6
¹⁵⁵ Eu	49.6	48.5	98.1	44.0	43.6	87.6
²⁴¹ Am	55.3	39.5	94.8	50.1	39.3	89.5
Total α	37.7	39.3	77.0	55.3	77.0	132.3
⁹⁰ Sr	59.1	30.5	89.6	33.3	52.8	86.0
Al	70.3	30.9	101.2	29.2	24.1	53.3
Ca	71.0	49.1	120.1 ^(b)	25.5	44.3	69.8
Cd	69.5	32.0	101.5	32.7	27.3	60.0
Cr	71.4	30.9	102.4	32.2	26.8	58.9
Cu	67.5	31.0	98.5	31.0	24.3	55.3
Fe ^(c)	67.3	<i>0–29.5</i>	<i>67.3–96.8</i>	26.7	22.7	49.4
K	70.0	28.2	98.2	33.3	24.9	58.2
Mn ^(c)	31.3	<i>0–89.3</i>	<i>31.3–120.6</i>	<i>0–30.4</i>	<i>0–129.5</i>	<i>0–159.9</i>
Mo	71.4	30.6	102.1	31.9	26.4	58.4
Na	50.9	48.5	99.4	18.9	51.8	70.7
Ni	71.8	34.1	105.9	32.5	27.5	60.0
P	67.8	28.7	96.5	21.4	33.5	54.9
Pb	71.1	33.8	104.9	32.8	27.7	60.5
Sr	67.9	33.3	101.2	21.6	46.8	68.4
U ^(c)	67.3	<i>0–178.7</i>	<i>67.3–246.0</i>	<i>0–28.3</i>	<i>0–120.5</i>	<i>0–148.7</i>
Zn	90.0	173.4	263.4 ^(b)	26.5	38.8	65.3

- (a) Analyte was not detected in one or more fractions; thus a mass balance could not be made.
- (b) High calcium and zinc mass balances are attributed to laboratory contamination, as these elements were found in the analytical blank.
- (c) Iron, manganese, and uranium results were below the analytical detection limit for the first precipitated solids, so a true mass balance could not be made. The values given in italics are a range of recoveries based on the detection limit and the factors given in Equations 1 through 4.

4.0 Conclusion

Although sulfate can be partially precipitated from the AN-107 liquid after concentration by evaporation, the separation is not selective. When the liquid is concentrated by more than 50%, a large amount of sodium nitrate and nitrite precipitated along with the sulfate. As seen in Table 3.2, many other species are also precipitated in approximately the same ratio along with sulfate. The first precipitated solids, which are 21.4% of the mass of the starting material, contain only 33.1% of the sulfate. Selective removal of sulfate by precipitation after liquid concentration by evaporation does not appear to be an attractive option.

5.0 References

Hallen, R. T., K. P. Brooks, and L. K. Jagoda. 2000. *Demonstration of Entrained Solids and Sr/TRU Removal Processes with Archived AN-107 Waste*, BNFL-RPT-026 (draft), Battelle Pacific Northwest Division, Richland, Washington.

Hendrickson, D. W. 1997. *Hanford Complexant Concentrate Cesium Removal Using Crystalline Silicotitanate*, SESC-EN-RPT-005, Rev. 0, SGN Eurisys Services Corporation, Richland, Washington.

Appendix A: Test Instruction

Appendix B: Raw Data

Discussion of the Analytical Results and Quality Assurance

Fluoride was reported in the anion analytical data; however, the report included the notation that the positive fluoride values were likely due to interference from organic anions; therefore, fluoride is not included in the mass-balance calculations. The sulfate measurements for both sets of precipitated solids were measured at the lower end of the calibration curve, near the method detection limit. The replicate for the first precipitated solid had poor agreement with the sample (>240% relative percent difference [RPD]). Since the sample and duplicate were done at different dilutions and injection sizes, the data used for the sulfate mass balance use the least diluted sample-injection result. Both nitrate and nitrite were measured in the filtrates at concentrations near or slightly above the calibration curve; however, the data were reported with the notation that good linearity had been demonstrated for sample concentrations at up to twice the concentration of the highest standard. All the reported data were less than this concentration.

The radionuclide data had relatively low uncertainties for ^{60}Co , ^{137}Cs , ^{154}Eu , ^{155}Eu , and ^{90}Sr for all samples. However, the ^{241}Am results for both precipitated solids had high uncertainties, 22% and 15%, respectively. Antimony-125 had high uncertainties for the original sample and each of the filtered concentrates, 19%, 30%, and 27%, respectively. Antimony-126 and ^{126}Sn total results had a high uncertainty for the second precipitated solid (17%). Total Alpha measurements had high uncertainties for both precipitated solids, 24% and 19%, respectively.

The metals data had results for some fractions that were below the detection limits for copper, iron, manganese, neodymium, rhodium, rhenium, uranium, and zirconium. Both boron and silicone had very high recoveries, which are attributed to the use of borosilicate glass for sample handling. The process blank for the metals analysis contained traces of calcium, nickel, and zinc. The levels of nickel detected in the samples were many orders of magnitude greater than the levels found in the blank; consequently, there was no impact to the data. The levels of both calcium and zinc detected in the samples were much lower relative to those detected in the blank.

A single acid-digestion preparation was performed for the ICP metals analysis. In the case of the second concentrate (AN-107-2), the weight and volume used for this preparation did not give a density that matched the density determined in the precipitation experiment. Additionally, upon investigation, it was determined that the volume entered on the acid digestion benchsheet did not include an aliquot removed for ^{90}Sr testing. A correction for this was made to the mass-balance data, but this did not entirely resolve the discrepancy.

The total carbon, total organic carbon, and ^{99}Tc tests were cancelled for logistical and financial reasons.

Appendix C: Calculations

The holdup quantities were calculated using the following corrections. The holdup upon transfer of the 45-mL concentrate to the filter apparatus was 2 g. Of this, 2 g held up in the beaker, 1.12 g was assigned to the liquid fraction, and 0.88 g was assigned to the wet solid fraction, based on the ratio of liquid and solids recovered. The density of the liquid fraction was determined to be 1.43 g/mL, based on the weight of the 10-mL aliquot of the filtered concentrate set aside for analytical work. This gives a liquid holdup volume of 0.78 mL for the transfer step to the filter apparatus, based on the weight measured for this step. The filtrate was then transferred to a graduated cylinder to measure the volume, which was 22 mL. The holdup on transfer to the graduated cylinder was estimated to be 0.75 mL, based on the density of the liquid (1.43 g/mL) and the sample weight before the transfer (32.3 g) to the graduate. The difference in the sample weight transferred from the graduated cylinder (16.7 g [further processing] + 14.3 g [analytical sample]) account for an additional holdup of 1.3 g (0.91 mL) on the graduated cylinder and filter flask.

Using the same approach, the volume of filtrate transferred to the 100-mL beaker for the second concentration was estimated to be 11.6 mL ($16.7 \text{ g} \div 1.43 \text{ g/mL}$). The second concentrate was transferred to a filtration apparatus. The holdup on this transfer was found to be 1.4 g, of which 0.63 g was assigned to the liquid fraction and 0.77 g was assigned to the wet solids, based on the ratio of the recovered amounts. The weight of the filtered liquid was found to be 4.98. The liquid was transferred to a graduated cylinder. The measured volume of the filtrate in the graduated cylinder was 3.0 mL. It was then transferred to a vial and weighed again. The weight after the transfer was found to be 4.53 g. The combined holdup for both these transfers was 0.46 g. Assuming that an equal holdup of filtrate occurred in the transfer from the filtrate receiving vessel to the graduated cylinder and the transfer from the graduated cylinder to the vial, the weight of the liquid in the graduate (3.0 mL) can be estimated to be 4.755, yielding an estimated density of 1.585 g/mL. Using this density, the total volume of the liquid fraction of the concentrate, corrected for holdup, is estimated to be 3.6 mL ($[4.98 \text{ g} \div 1.585 \text{ g/mL}] + [0.63 \text{ g} \div 1.585 \text{ g/mL}]$).

As discussed in the experimental section, the solids from the first concentration step were split among two vials, and the dry weight was normalized to the driest sample (AN107 Solids1B). The holdup amount assigned to the transfer of the wet solids to the filtration apparatus was 0.88 g. An additional 0.9-g holdup was assigned to the transfer of the wet solids from the filter to the two vials. The holdup corrections for the second precipitation include the holdup on transfer of the filtrate to the graduated cylinder (0.45 mL), the holdup for the transfer of the second concentrated slurry to the filter apparatus (0.77 g), and the transfer of the solids from the filter to the vial for drying (0.56 g).

The equations used to calculate the volumetric factors applied to the analytical results are given below:

$$22.0 \text{ mL} + \left(\frac{32.3 \text{ g}}{59.6 \text{ g}} \times \frac{10.0 \text{ mL}}{14.3 \text{ g}} \times 2 \text{ g} \right) = 22.8 \text{ mL} \quad (1: \text{AN107-1})$$

where:

22.0 mL is the isolated liquid volume,

32.3 g ÷ 59.6 g is the fraction of holdup mass as liquid,

10.0 mL ÷ 14.3 g is the liquid density,

2 g is the holdup mass,

22.8 is the total volume of the liquid.

$$\frac{26.1 \text{ g} \times \frac{16.2 \text{ g}}{24.3 \text{ g}}}{0.189 \text{ g}} \times 20.2 \text{ mL} = 1860 \text{ mL} \quad (2: \text{AN107-PPT1})$$

where:

26.1 g is the corrected solid mass from the first precipitation,

16.2 g ÷ 24.3 g is the weight fraction of dry solids,

0.189 g is the analysis sample mass,

20.2 mL is the analysis sample volume,

1860 mL is the total volume if all the solids were used to prepare the sample.

$$\frac{22.8 \text{ mL}}{11.6 \text{ mL}} \times \left[3 \text{ mL} + \left(\frac{4.98 \text{ g}}{12.5 \text{ g}} \times \frac{3.0 \text{ mL}}{4.775 \text{ g}} \times 1.4 \text{ g} \right) \right] = 6.59 \text{ mL} \quad (3: \text{AN107-2})$$

where:

22.8 mL ÷ 11.6 mL is the fraction of the first concentrated liquid used in the second precipitation,

3 mL is the isolated volume of the second evaporated liquid,

4.98 g ÷ 12.5 g is the liquid fraction from the second evaporation,

3.0 mL ÷ 4.775 g is the density of the second evaporated liquid,

1.4 g is the holdup mass,

6.59 mL is the total volume of the liquid.

$$\frac{22.8 \text{ mL}}{11.6 \text{ mL}} \times \frac{6.85 \text{ g} \times \frac{4.16 \text{ g}}{5.52 \text{ g}}}{0.242 \text{ g}} \times 20.1 \text{ mL} = 842 \text{ mL} \quad (4: \text{AN107-PPT2})$$

where:

22.8 mL ÷ 11.6 mL is the fraction of the first concentrated liquid used in the second precipitation,

6.85 g is the corrected solid mass from the second precipitation,

4.16 g ÷ 5.52 g is the weight fraction of dry solids,

*0.242 g is the analysis sample mass,
20.1 mL is the analysis sample volume,
842 mL is the total volume if all the solids were used to prepare the sample.*

The volumetric factor applied to the starting material sample (AN107-Start) results was 101 mL. The second-step evaporation results are corrected for the quantity of sample removed from the first filtrate.

The initial density of the AN-107 starting material was 1.210 g/mL. Evaporation of the sample to a volume of 45 mL resulted in a slurry with a density of 1.324 g/mL and a filtered liquid with a density of 1.429 g/mL. The slurry resulting from the second concentration step had a density of 1.393 g/mL. As described above, the estimated density of the second filtrate was 1.585 g/mL. Since the analytical results were provided in volumetric units, the mass balance was calculated using corrected volumes.

The following is a sample calculation for sulfate and shows how the values in Table 3.2 were obtained for each analyte. The mass balances for each analyte, corrected for holdup and subsampling, are summarized in Table 3.2.

AN107 Start: The SO_4^{-2} concentration reported for this sample was 4000 ppm. The total sulfate in the starting AN107 material was determined as follows:

$$4000 \text{ mg/L} \times 0.101 \text{ L} = 404 \text{ mg}$$

AN107-1 (filtered liquid): The SO_4^{-2} concentration reported for this sample was 11000 ppm. The quantity of sulfate in the filtered liquid was determined as follows:

$$11000 \text{ mg/L} \times 0.0228 \text{ L} = 250.3 \text{ mg}$$

The percentage left in solution was found to be

$$100 \times 250.3 \text{ mg} \div 404 \text{ mg} = 61.9\%$$

AN107-PPT1 (precipitated solids): The SO_4^{-2} concentration reported for this sample was 72 ppm. The quantity of sulfate in the precipitated solids were determined as follows:

$$72 \text{ mg/L} \times 1.860 \text{ L} = 133.6 \text{ mg}$$

The percentage precipitated from the solution was found to be

$$100 \times 133.6 \text{ mg} \div 404 \text{ mg} = 33.1\%$$

The quantities of sulfate in the concentrated liquid and the precipitate account for 95% of the original sulfate in the AN107-Start sample.

AN107-2 (filtered liquid): The SO_4^{-2} concentration reported for this sample was 5200 ppm.

The quantity of sulfate in the filtered liquid was determined as follows:

$$5200 \text{ mg/L} \times 0.00659 \text{ L} = 34.2 \text{ mg}$$

The percentage left in solution was found to be

$$100 \times 34.2 \text{ mg} \div 250.3 \text{ mg} = 13.7\%$$

AN107-PPT2 (precipitated solids): The SO_4^{-2} concentration reported for this sample was 230 ppm. The quantity of sulfate in the precipitated solids was determined as follows:

$$230 \text{ mg/L} \times 0.842 \text{ L} = 193.66 \text{ mg}$$

The percentage precipitated from the solution was found to be

$$100 \times 98.4 \text{ mg} \div 250.3 \text{ mg} = 77.4\%$$

The quantities of sulfate in the concentrated liquid and the precipitate accounts for 91% of the original sulfate in the AN107-1 sample.

The values given in Table 3.2 were calculated from the analytical data in the same way. Where only the method detection limit was provided, a calculated range of the lower boundary is given in the table.

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Appendix A: Test Instruction

Test Instruction

Unique Numerical Designation: 29953-055
 Revision number: 0
 Effective Date: September 10, 1999
 Controlling Procedure No: RPG-OP-511

Author Approval: *Suzanne J. Linneth 9/10/99*
 Technical Reviewer: *D E Linneth 9/10/99*

TITLE: Evaporation of Archive AN-107 to Separate Sulfate

Work Instructions

*9/13/99
 m.t.*

1. Prepare sample vials (plastic, 20-mL) according to the following table

Sample ID	Tare wt., g
AN107-Start	6.748
AN107-1	6.694
AN107-2	6.752
AN107-PPT1	6.767
AN107-PPT2	6.749

*Balance still recal 3
 Scintorius L420 5+
 384-06-01-005
 Cal. Date 8/18/99
 Exp 8/2000*

2. Obtain a 500 mL beaker, and insert a magnetic stir bar; determine tare weight

$$\text{Wt. beaker + stir bar} = \underline{251.666} \text{ g} \quad (2a)$$

3. Measure 45 mL of deionized water in a graduated cylinder

4. Place the 45 mL of water in the beaker

5. Using a Sharpie pen, mark the 45-mL level on the beaker (the solution will be evaporated to this point); discard the water

*6.150m
 5 mL pipet 175 4214
 weighed 5-mL portions
 of H₂O → 4.994
 5.006
 4.981
 4.969
 4.976*

6. Measure 100 mL of the AN-107 sample in a graduated cylinder

7. Place the 100 mL of sample into the 500-mL beaker; determine weight

$$\text{Wt. beaker + stir bar + sample} = \underline{373.854} \text{ g} \quad (7a)$$

$$\text{Wt. sample} = 7a - 2a = \underline{122.245} \text{ g} \quad (7b)$$

8. Transfer 20 mL of the AN-107 sample into sample vial AN107-Start; weigh sample vial

$$\text{Wt. AN107-Start} = \underline{31.196} \text{ g}$$

9. Set up video camera to record the progress of the evaporation; set the camera in the time delay mode with 0.5 min between frames

10. While stirring, heat the beaker at ~90°C. *Note: Temporarily remove from the hot plate if foaming becomes excessive.*

No foaming occurred.

*Set hot plate temp to 90°C @ ~9:30
 110°C @ 9:50
 115°C @ 10:20
 Solid formed sometime between 12:00 and 12:30.*

S. J. Linneth 9/13/99

9/14/99 M.A.L.

21. Transfer 15 mL of the filtered liquid to sample vial AN107-1 weigh sample vial

Wt. AN107-1 = 20.981 g

22. Label a glass vial as "AN107-Solid1" and record tare weight

Wt. AN107-Solid1 = 16.455 g

23. Transfer the wet solids from the filter to vial AN107-Solid1

24. Dry the solids at 105°C for a minimum of 1 h, then weigh

9/16/99 Wt. AN107-Solid1 = 29.850 g
wt. AN107-Solid1 B = 20.946

25. Measure out 0.1 to 0.2 g of the dried solids into sample vial AN107-PPT1

9/16/99 Wt. AN107-PPT1 = 6.956 g

Wt. solid in AN107-PPT1 = 0.189 (6.956 - 6.767)

26. Add 20 mL of deionized water to AN107-PPT1 to dissolve the solids for analysis; weigh

9/16/99 Wt. AN107-PPT1 = 27.151 g

$$\begin{array}{r} 27.151 \\ - 6.956 \\ \hline 20.195 \text{ g H}_2\text{O added} \end{array}$$

Note: Record any observations regarding kinetics of dissolution → Dissolved quickly.

27. Obtain a 100 mL beaker, and insert a magnetic stir bar; determine tare weight

Wt. beaker + stir bar = 55.367 g (27a)

28. Pour the remaining liquid in AN107 First Concentrate into a graduated cylinder; record the volume *Initial determined from vol in step 19 and sample taken in 21.*

Vol. of solution = 12 mL
$$\begin{array}{r} 22 \\ - 10 \\ \hline 12 \text{ mL} \end{array}$$
 (28a)

28A. Determine the target volume

Target volume = 0.75 * 28a = 9.0 mL (28b)

29. Measure the target volume (28b) of deionized water in a graduated cylinder

30. Place the water in the 100-mL beaker

31. Using a Sharpie pen, mark the target volume level on the beaker (the solution will be evaporated to this point); discard the water

32. Pour the sample into the 100-mL beaker; determine weight

Wt. beaker + stir bar + sample = 72.065 g (32a)

Wt. sample = 32a - 27a = 16.698 g (32b)

(b) AN107-Solid1 → 29.850 - 16.455 = 13.395 g dry solids
AN107-Solid1 B → 20.946 - 16.579 = 4.367 g dry solids
So total mass dry solid → (24.337 g wet) * (1 - 0.336) = 16.16 g

$$\frac{17.760 - 12.385}{17.760} \cdot 100 = 24.6\% \text{ wt loss on drying}$$

$$\frac{6.577 - 4.367}{6.577} \cdot 100 = 33.6\% \text{ wt. loss}$$

All solids weighed wt. in AN107-Solid1. Put remainder in AN107-Solid1 B.
AN107-Solid1 B 16.579
wet wts. AN107-Solid1 34.215
AN107-Solid1 B 23.156
wt. wet solids = 34.215 - 16.455 = 17.760
23.156 - 16.579 = 6.577
24.337
AN107-Solid1 did not look completely dry. (b)

M.A. Smith
9/14/99

9/15/99 M.A.L.

43. Pour the filtered liquid into a graduated cylinder to measure the volume

Vol. Liquid = 3.0 mL

44. Pour the liquid back into AN107 Second Concentrate → did not do.

45. Transfer ^{all} ~~15~~ mL of the filtered liquid to sample vial AN107-2 weigh sample vial

Wt. AN107-2 = 11.278 g

46. Label a glass vial as "AN107-Solid2" and record tare weight

Wt. AN107-Solid2 = 16.553 g

wt w/ wet solids = 21.767
wt wet solids = 21.767
- 16.553

5.214

47. Transfer the wet solids from the filter to vial AN107-Solid2

48. Dry the solids at 105°C for a minimum of 1 h, then weigh

Dried ~~sample~~

9/16/99 Wt. AN107-Solid2 = 20.712 g

20.712
- 16.553

4.159 g dry solids

5.214 - 4.159

1.055

49. Measure out 0.1 to 0.2 g of the dried solids into sample vial AN107-PPT2

9/16/99

Wt. AN107-PPT2 = 20.58 g

reweighed M.A.L. 9/16/99
6.991

= 20.2 wt%
loss

Wt. solid in AN107-PPT2 = ~~20.58~~ - 6.749 = 0.309 = 0.242
6.991

50. Add 20 mL of deionized water to AN107-PPT2 to dissolve the solids for analysis; weigh

9/16/99

Wt. AN107-PPT1 = 27.079 g

27.079 - 6.991 = 20.088 g H₂O added.

Note: Record any observations regarding kinetics of dissolution → dissolved rapidly.

51. Submit the samples for analysis—acid digestion, GEA, Sr-90, ICP-MS(⁹⁹Tc), total alpha, ICP, TIC/TOC, and IC

The End

Mary A. Hamilton
9/16/99

Appendix B: Raw Data

Discussion of the Analytical Results and Quality Assurance

Fluoride was reported in the anion analytical data; however, the report included the notation that the positive fluoride values were likely due to interference from organic anions; therefore, fluoride is not included in the mass-balance calculations. The sulfate measurements for both sets of precipitated solids were measured at the lower end of the calibration curve, near the method detection limit. The replicate for the first precipitated solid had poor agreement with the sample (>240% relative percent difference [RPD]). Since the sample and duplicate were done at different dilutions and injection sizes, the data used for the sulfate mass balance use the least diluted sample-injection result. Both nitrate and nitrite were measured in the filtrates at concentrations near or slightly above the calibration curve; however, the data were reported with the notation that good linearity had been demonstrated for sample concentrations at up to twice the concentration of the highest standard. All the reported data were less than this concentration.

The radionuclide data had relatively low uncertainties for ^{60}Co , ^{137}Cs , ^{154}Eu , ^{155}Eu , and ^{90}Sr for all samples. However, the ^{241}Am results for both precipitated solids had high uncertainties, 22% and 15%, respectively. Antimony-125 had high uncertainties for the original sample and each of the filtered concentrates, 19%, 30%, and 27%, respectively. Antimony-126 and ^{126}Sn total results had a high uncertainty for the second precipitated solid (17%). Total Alpha measurements had high uncertainties for both precipitated solids, 24% and 19%, respectively.

The metals data had results for some fractions that were below the detection limits for copper, iron, manganese, neodymium, rhodium, rhenium, uranium, and zirconium. Both boron and silicone had very high recoveries, which are attributed to the use of borosilicate glass for sample handling. The process blank for the metals analysis contained traces of calcium, nickel, and zinc. The levels of nickel detected in the samples were many orders of magnitude greater than the levels found in the blank; consequently, there was no impact to the data. The levels of both calcium and zinc detected in the samples were much lower relative to those detected in the blank.

A single acid-digestion preparation was performed for the ICP metals analysis. In the case of the second concentrate (AN-107-2), the weight and volume used for this preparation did not give a density that matched the density determined in the precipitation experiment. Additionally, upon investigation, it was determined that the volume entered on the acid digestion benchsheet did not include an aliquot removed for ^{90}Sr testing. A correction for this was made to the mass-balance data, but this did not entirely resolve the discrepancy.

The total carbon, total organic carbon, and ^{99}Tc tests were cancelled for logistical and financial reasons.

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

WO/Project: W51310/29953
Client: G. Lumetta

ACL Numbers: 99-02650 through 99-02654
ASR Number 5520

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Analyst: MJ Steele Analysis Date: September 21-22, 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: Michael W. Thur Date 10 25 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 $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Final Results:

The five 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 4,000-fold to ensure that all anions were within the calibration range. The anion results are presented in the table below.

Client : Kurath/Lumetta

Cognizant Scientist: B.K. Fiskum

Date : 10/12/99

Concur : T. Trang-le

Date : 10/12/99

Procedure: PNL-ALO-450

Measured Activities ($\mu\text{Ci/ml}$) and 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-2650 AN107 Start	5.72E-2 1%	5.62E-4 19%	2.87E-4 7%	<2.E-4	2.67E-2 2%	1.10E-2 2%	7.95E-3 3%	4.52E-3 5%
99-2651 AN107-1	1.63E-1 1%	9.54E-4 30%	6.56E-4 9%	<4.E-4	7.74E-2 2%	2.42E-2 2%	1.75E-2 3%	1.11E-2 6%
99-2652 AN107-2	3.07E-1 1%	1.32E-3 27%	1.51E-3 6%	<6.E-4	1.49E-1 2%	3.62E-2 2%	2.66E-2 3%	1.92E-2 5%
99-2653 AN107-PPT1	1.02E-3 2%	<3.E-5	<9.E-6	<2.E-5	4.56E-4 3%	2.70E-4 3%	2.10E-4 7%	9.71E-5 22%
99-2654 AN107-PPT2	1.31E-3 2%	<2.E-5	7.32E-6 17%	<8.E-6	6.07E-4 2%	2.83E-4 2%	2.06E-4 5%	1.18E-4 15%
99-2685 AN-SO-L22A	2.90E-2 2%	<3.E-4	1.35E-4 16%	9.69E-5 49%	1.64E-2 2%	4.50E-3 3%	3.19E-3 5%	1.87E-3 15%
99-2686 SO4-Effluent Composite	5.53E-2 1%	3.65E-4 22%	2.41E-4 7%	<2.E-4	2.39E-2 2%	1.06E-2 2%	7.58E-3 3%	4.45E-3 5%
99-2687 AN-SO-FD1	2.64E-3 2%	<5.E-5	<2.E-5	<2.E-5	5.93E-3 2%	2.61E-4 4%	2.03E-4 8%	1.28E-4 22%
99-2688 AN-SO-FD2	2.24E-4 2%	<3.E-5	<9.E-6	<4.E-6	4.71E-3 2%	1.07E-5 18%	<2.E-5	<3.E-5
99-2689 AN-SO-CW1	1.04E-4 2%	<3.E-5	<8.E-6	<3.E-6	3.42E-3 2%	8.50E-6 21%	<2.E-5	<2.E-5
99-2690 AN-SO-CW2	6.73E-5 2%	<2.E-5	<3.E-6	<2.E-6	2.30E-3 2%	1.38E-5 11%	<1.E-5	1.10E-5 42%
99-2692 AN-SO-E1-A	6.48E-3 2%	3.82E-5 36%	2.49E-5 12%	<2.E-5	1.38E-3 2%	5.91E-4 3%	4.14E-4 5%	2.27E-4 16%
99-2693 AN-SO-E2-A	2.02E-3 2%	<5.E-5	<2.E-5	<2.E-5	1.67E-3 2%	4.29E-3 1%	3.09E-3 3%	1.48E-3 6%
99-2694 AN-SO-E3-A	1.36E-3 2%	<3.E-5	8.81E-6 27%	<2.E-5	1.30E-3 2%	1.08E-3 2%	8.06E-4 3%	4.05E-4 7%
99-2695 AN-SO-E4-A	3.25E-4 2%	<3.E-5	<6.E-6	<7.E-6	1.04E-3 2%	9.65E-5 5%	6.81E-5 13%	6.20E-5 26%
99-2696 AN-SO-E5-A	9.28E-5 3%	<2.E-5	<7.E-6	<6.E-6	7.94E-4 2%	1.51E-5 17%	1.21E-5 33%	1.68E-5 35%
99-2697 AN-SO-E6-A	5.21E-5 5%	<2.E-5	<5.E-6	<6.E-6	6.04E-4 2%	1.28E-5 27%	<2.E-5	2.06E-5 36%

Client : Kurath/Lumetta

Cognizant Scientist: J R Greenwood

Date : 10/22/99

Concur : T Trang-le

Date : 10/22/99

Procedure: PNL-ALO-420, 421

Measured Activities ($\mu\text{Ci/ml}$) and 1-sigma error

<u>ALO ID</u> <u>Client ID</u>	<u>Alpha</u> <u>Error %</u>	<u>Sr-90</u> <u>Error %</u>
99-2650 AN107 Start	5.12E-3 3%	6.87E-1 3%
99-2650 REP AN107 Start	5.27E-3 3%	7.46E-1 3%
RPD	3%	8%
99-2651 AN107-1	8.70E-3 3%	1.88E+0 4%
99-2652 AN107-2	1.66E-2 2%	2.16E+0 3%
99-2653 AN107-PPT1	1.11E-4 24%	1.19E-2 3%
99-2654 AN107-PPT2	1.81E-4 19%	2.68E-2 3%
99-2685 AN-SO-L22A		3.49E-1 3%
99-2686 SO4-Effluent Composite		6.02E-1 3%
99-2687 AN-SO-FD1		4.85E-2 3%
99-2688 AN-SO-FD2		7.38E-3 3%
99-2689 AN-SO-CW1		5.60E-3 3%
99-2690 AN-SO-CW2		6.51E-3 3%
99-2692 AN-SO-E1-A		5.04E-2 3%
99-2693 AN-SO-E2-A		3.92E-1 3%
99-2694 AN-SO-E3-A		1.29E-1 3%

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

Project: 29953
Client: G. Lumetta, and D. Kurath

ACL Number(s): 99-2650 through 99-2705

Client ID: "AAN107-Start" through "AN-SO-REGN"

ASR Number: 5520

Total Samples: 25

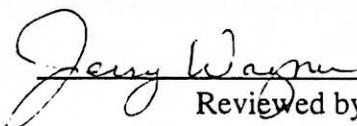
Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 11-05-99 (A0551) & 11-24-99 (A0559)

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

 01-10-00
Reviewed by

 01-10-00
Concur

1/10/00

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

Post-Spiked Samples (Group A):

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

Post-Spiked Samples (Group B):

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

Blank Spike:

None.

Matrix Spiked Sample:

None.

Quality Control Check Standards:

Concentration of all analytes of interest was within tolerance limit of $\pm 10\%$ accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMVCV except as follows.

Potassium was slightly high (<14%) in two of seven QC_MCVA check standard measurements. Single element reagent standards of potassium at 100 ppm measured at the beginning and end of the run were well within the tolerance limit.

One of seven sodium measurements of QC_MCVA was somewhat high (+17%) and also one of two measurements in QC_SSTMVCV (+13%). Sodium measurement of SRM-1643d, a NIST reference solution, was within tolerance limits at a concentration similar to that in QC_MCVA.

One of three tin measurements of QC_MCVB was low by about 14%. Measurement of a single element reagent standard of tin at 2 ppm was well within tolerance limit.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes of interest was within tolerance of $\pm 5\%$ accuracy except sodium in one of two measurements (+7%).

A single element sodium standard of 1000 ppm measured at the beginning and at the very end of the run was within tolerance limits.

1/10/00

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

	Multiplier=	1.0	8.2	36.4	37.8	2.0
	ALO#=	99-2650-PB	99-2650 ⑤5	99-2651 ⑩10	99-2652 ⑩10	99-2653
	Client ID=	Process Blank	AN107 START	AN107-1	AN107-2	AN107-PPT1
Det. Limit	Run Date=	11/5/99	11/5/99	11/5/99	11/5/99	11/5/99
(ug/mL)	(Analyte)	ug/mL	ug/g	ug/g	ug/g	ug/g
0.025	Ag	-	-	-	-	-
0.060	Al	-	109	288	218	2.22
0.250	As	-	-	-	-	-
0.050	B	-	17.3	51.4	35.2	5.51
0.010	Ba	-	-	-	-	-
0.010	Be	-	-	-	-	-
0.100	Bi	-	-	-	-	-
0.250	Ca	[0.27]	133	355	235	[4.3]
0.015	Cd	-	21.7	56.7	48.1	0.457
0.200	Ce	-	-	-	-	-
0.050	Co	-	[1.7]	[4.6]	[3.8]	-
0.020	Cr	-	10.8	29.0	24.2	[0.22]
0.025	Cu	-	14.7	37.3	30.0	[0.30]
0.050	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	-	2.57	[6.5]	[4.5]	-
2.000	K	-	593	1,560	1,350	[11]
0.050	La	-	-	-	-	-
0.030	Li	-	-	-	-	-
0.100	Mg	-	-	-	-	-
0.050	Mn	-	[1.7]	[2.0]	-	-
0.050	Mo	-	12.4	33.3	27.6	[0.25]
0.150	Na	-	93,600	179,000	87,800	2,990
0.100	Nd	-	[1.1]	-	-	-
0.030	Ni	[0.044]	182	491	414	4.09
0.100	P	-	166	423	235	3.14
0.100	Pb	-	62.9	168	143	[1.4]
0.750	Pd	-	-	-	-	-
0.300	Rh	-	[3.9]	-	-	-
1.100	Ru	-	[12]	-	-	-
0.500	Sb	-	-	-	-	-
0.250	Se	-	-	-	-	-
0.500	Si	-	82.9	238	[120]	18.4
1.500	Sn	-	-	-	-	-
0.015	Sr	-	74.8	191	107	1.64
1.500	Te	-	-	-	-	-
1.000	Th	-	-	-	-	-
0.025	Ti	-	-	-	-	-
0.500	Tl	-	-	-	-	-
2.000	U	-	[34]	[86]	-	-
0.050	V	-	-	-	-	-
2.000	W	-	[60]	[160]	[140]	-
0.050	Y	-	-	-	-	-
0.050	Zn	[0.068]	4.73	[16]	[11]	[0.54]
0.050	Zr	-	[1.0]	[3.8]	[2.8]	-

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)	2.0 99-2689 AN-SO-CW1 11/5/99 ug/g	2.0 99-2690 AN-SO-CW2 11/5/99 ug/g	2.0 99-2690-DUP AN-SO-CW2 11/5/99 ug/g	2.0 99-2703 AN-SO-ER1 11/5/99 ug/g	2.0 99-2704 AN-SO-ER2 11/5/99 ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	1.80	--	--	2.09	[0.48]
0.250	As	--	--	--	--	--
0.050	B	9.37	4.84	1.80	6.57	6.30
0.010	Ba	8.37	8.89	8.90	152	196
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[1.1]	[1.9]	[1.4]	9.62	[3.6]
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	[0.041]	--
0.025	Cu	[0.22]	0.582	0.578	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	0.829	[0.46]
2.000	K	107	78.3	76.7	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	[0.43]	--
0.050	Mn	--	[0.53]	--	[0.11]	--
0.050	Mo	--	--	--	--	--
0.150	Na	6,200	5,220	5,500	4,070	5,340
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.26]	[0.20]	[0.20]	[0.26]	[0.23]
0.100	P	[1.1]	[0.90]	[0.93]	[0.38]	[0.27]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	64.1	15.6	10.3	[3.0]	[1.5]
1.500	Sn	--	--	--	--	--
0.015	Sr	0.726	0.844	0.840	[0.053]	--
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	[0.12]	[0.37]	[0.23]	[0.26]	[0.16]
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=	24.2	24.7	24.7	24.8	24.7
	ALO#=	99-2692 @5	99-2693 @5	99-2694 @5	99-2695 @5	99-2696 @5
	Client ID=	AN-SO-E1-A	AN-SO-E2-A	AN-SO-E3-A	AN-SO-E4-A	AN-SO-E5-A
Det. Limit	Run Date=	11/24/99	11/24/99	11/24/99	11/24/99	11/24/99
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	-	-	-	-	-
0.060	Al	83.7	32.8	29.2	[8.6]	[7.7]
0.250	As	[11]	-	-	-	-
0.050	B	108	84.2	89.4	82.1	82.7
0.010	Ba	478	1,280	523	459	435
0.010	Be	-	-	-	-	-
0.100	Bi	-	-	-	-	-
0.250	Ca	110	144	85.1	98.3	[51]
0.015	Cd	14.2	5.07	[3.2]	-	-
0.200	Ce	-	-	-	-	-
0.050	Co	-	-	-	-	-
0.020	Cr	7.21	[3.3]	[2.1]	-	-
0.025	Cu	9.76	7.34	[2.1]	-	-
0.050	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	[2.2]	8.77	8.62	[2.0]	[1.1]
2.000	K	[77]	[260]	[190]	[150]	[110]
0.050	La	-	-	-	-	-
0.030	Li	-	-	-	-	-
0.100	Mg	-	-	-	-	-
0.050	Mn	[3.5]	54.4	41.1	[4.1]	-
0.050	Mo	[8.1]	[1.6]	-	-	-
0.150	Na	77,900	32,600	14,600	2,310	245
0.100	Nd	-	-	-	-	-
0.030	Ni	124	42.2	27.4	[2.3]	-
0.100	P	99.6	109	36.9	[4.6]	-
0.100	Pb	38.3	[7.0]	-	-	-
0.750	Pd	-	-	-	-	-
0.300	Rh	-	-	-	-	-
1.100	Ru	-	-	-	-	-
0.500	Sb	-	-	-	-	-
0.250	Se	-	-	-	-	-
0.500	Si	563	[62]	[96]	[80]	[66]
1.500	Sn	-	-	-	-	-
0.015	Sr	35.4	280	93.6	24.4	4.60
1.500	Te	-	-	-	-	-
1.000	Th	-	-	-	-	-
0.025	Tl	-	-	-	-	-
0.500	Tl	-	-	-	-	-
2.000	U	-	-	-	-	-
0.050	V	-	-	-	-	-
2.000	W	-	-	-	-	-
0.050	Y	-	-	-	-	-
0.050	Zn	[3.5]	[2.7]	[1.9]	16.8	[4.7]
0.050	Zr	-	[1.7]	-	-	-

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).

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	24.7 99-2702 @5 SO4-ELUANT- COMP-A 11/24/99 ug/g						
0.025	Ag	--	--	--	--	--	--	--
0.060	Al	[7.1]	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--
0.050	B	31.0	--	--	--	--	--	--
0.010	Ba	464	--	--	--	--	--	--
0.010	Be	--	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--	--
0.250	Ca	[59]	--	--	--	--	--	--
0.015	Cd	[0.54]	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--	--
0.020	Cr	--	--	--	--	--	--	--
0.025	Cu	--	--	--	--	--	--	--
0.050	Dy	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--
0.025	Fe	[2.3]	--	--	--	--	--	--
2.000	K	[73]	--	--	--	--	--	--
0.050	La	--	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--	--
0.050	Mn	[5.7]	--	--	--	--	--	--
0.050	Mo	--	--	--	--	--	--	--
0.150	Na	2,540	--	--	--	--	--	--
0.100	Nd	--	--	--	--	--	--	--
0.030	Ni	[5.1]	--	--	--	--	--	--
0.100	P	[6.5]	--	--	--	--	--	--
0.100	Pb	--	--	--	--	--	--	--
0.750	Pd	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--
0.500	Si	[33]	--	--	--	--	--	--
1.500	Sn	--	--	--	--	--	--	--
0.015	Sr	15.3	--	--	--	--	--	--
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	[3.5]	--	--	--	--	--	--
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).

ACL number	client ID	description	vial	aliquot (ml)	sample + vial	sample wt. (g)	mL of each acid	final volume
99-2650	AN107 START	AN107 SUPERNATE -20ML	8.1198	10	20.2734	12.1536	1.1	20
99-2651	AN107-1	AN107 SUPERNATE - 10ML	8.1071	2	10.8547	2.7476	0.5	10
99-2652	AN107-2	AN107 SUPERNATE - 2ML	8.0633	2	10.7068	2.6435	0.6	10
99-2653	AN107-PPT1	Precipitate - redissolved - 20 ml	8.0800	10	18.0253	9.9453	1.1	20
99-2654	AN107 -PPT2	Precipitate - redissolved 0- 20 ml	8.1355	10	18.1172	9.9817	1.0	20
99-2685	AN-SO-L22A	AN-107 supernate	8.1188	2	10.2693	2.1505	0.5	10
99-2686	SO4-Effluent-	AN-107 supernate	8.0159	10	20.0696	12.0537	1.1	20
99-2687	AN-SO-FDI	0.1 M NaOH/0.25 M NaNO3	8.0734	10	18.2572	10.1838	1.0	20
99-2688	AN-SO-FD2	0.1 M NaOH/0.25 M NaNO3	8.1228	10	18.0604	9.9376	1.0	20
99-2689	AN-SO-CW1	Water/dilute caustic/NaNO3	8.0897	10	18.1402	10.0505	1.0	20
99-2690	AN-SO-CW2	Water/dilute caustic/NaNO3	8.0697	10	17.9753	9.9056	1.0	20
99-2690 DUP	AN-SO-CW2	Water/dilute caustic/NaNO3	8.3154	10	18.4109	10.0955	1.0	20
99-2703	AN-SO-ER1	0.25 M NaNO3	8.0862	10	17.9977	9.9115	1.0	20
99-2704	AN-SO-ER2	0.25 M NaNO3	8.0567	10	18.0884	10.0317	1.0	20
99-2705	AN-SO-REGN	0.1 M NaOH/0.25 M NaNO3	8.0815	10	18.1536	10.0721	1.0	20

A0551 11-05-99

Completed

COMMENTS

ASR#	5520
CLIENT	Lumetta

Corrections

1. 99-2652 Acid Digest used GFA fraction
 2ml - 0.5 ml used for Sr90 Dilution
 wt is correct but vol = ~ 1.5 ml

2. ICP Dilutions were the remainder of the
 Sr90 Dilutions. The 1ml aliquots were
 not used for any determinations

analyst MJ Steele date 12/29/99

reviewer _____ date _____

5520
-2995

Analytical Chemistry Laboratory (ACL) Analytical Services Request (ASR)
(Cover Page ... information applicable to all samples in series)

RECORD COPY
376-6792

For AN107-Start
 AN107-1, AN107-PPT1
 AN107-2, AN107-PPT2
 RFS, CZS, LPD, RTR, JSW, JPB, TKL,
 WJS,

Requested By: Gregg J. Lumetta / D & Kurath D & Kurath 376-6911 P7-25
 Print Name Signature/Date Phone MSIN

Requester - Please Complete All Fields In This Section, Unless Specified "Optional" or ASR is a Revision

Request ID (optional): _____

PNL Project Number (if known): 29953

Work Order/Pkg.: W51310

Cost Estimate (\$): _____

Protocol Requirement: None RCRA CERCLA, or
 Other (specify): _____

Hold Time Requirement: None RCRA CERCLA, or
 Other (specify): _____

TPA Support: No, or
 Milestone No.: _____

QA Plan: MCS-033, or
 Other ACL QA Plan (specify): _____

Additional QA Requirements: No, or
 Reference Doc.: _____

ACL COC Req'd (PNL-ALO-010): No Yes

Sample Storage Requirements: No Refrigerate, or
 Other (specify): _____

Date Sampled (optional): _____

Time Sampled (optional): _____

Matrix: Samples vary (specify on Request Page), or
 Liquid: Aqueous Organic Multi-phasic
 Solid: Soil Sludge Sediment Glass
 Filter Smear Metal Organic Other Solids
 Solid/Liquid Mixture: Gas:

Biological: Tissue Urine Feces

Process Knowledge: Sample Information Check List, or
 Reference Doc.: _____

PCBs Present: No Yes

Sample Disposition ...
 Untreated Sample(s): Return Dispose Store, or
 Reference Doc.: _____
 Prep'd Sample(s): Dispose Return Store, or
 Reference Doc.: _____

Additional Instructions: No, or
 Reference Doc.: _____

Date Report Req'd: 9/23/99

Send Report to: G.J. Lumetta

MSIN: P7-25 Phone: 376-6911
 Fax (optional): _____

For ACL Use Only ... Do Not Complete This Section

Date Delivered: 9/19/99

Time Delivered (optional): _____

Deliv. By (if known): _____

Received By: MW Urie

Resp. ACL Mgr.: MW Urie

Signature/Date: _____

Job Group (optional): _____

Sample Group (optional): _____

PNL Impact Level: 1 2 3

DQ Review Req'd: No Yes ACL Waste: No Yes

ASR Number: 5520 Revision: Yes

ACL Numbers (99-2250)-(99-2108)

Acid Digest procedure #128

analyst

My Steeko

date

12/29/99

ACL number	client ID	description	vial	aliquot (ml)	sample + vial	sample wt. (g)	mL of each acid	final volume
99-2650	AN107 START	AN107 SUPERNATE -20ML	8.1198	10	20.2734	12.1536	1.1	20
99-2651	AN107-1	AN107 SUPERNATE - 10ML	8.1071	2	10.8547	2.7476	0.5	10
99-2652	AN107-2	AN107 SUPERNATE - 2ML	8.0633	2	1.5	2.6435	0.6	10
99-2653	AN107-PPT1	Precipitate - redissolved - 20 ml	8.0800	10	18.0253	9.9453	1.1	20
99-2654	AN107 -PPT2	Precipitate - redissolved 0- 20 ml	8.1355	10	18.1172	9.9817	1.0	20
99-2685	AN-SO-L22A	AN-107 supernate	8.1188	2	10.2693	2.1505	0.5	10
99-2686	SO4-Effluent-	AN-107 supernate	8.0159	10	20.0696	12.0537	1.1	20
99-2687	AN-SO-FD1	0.1 M NaOH/0.25 M NaNO3	8.0734	10	18.2572	10.1838	1.0	20
99-2688	AN-SO-FD2	0.1 M NaOH/0.25 M NaNO3	8.1228	10	18.0604	9.9376	1.0	20
99-2689	AN-SO-CW1	Water/dilute caustic/NaNO3	8.0897	10	18.1402	10.0505	1.0	20
99-2690	AN-SO-CW2	Water/dilute caustic/NaNO3	8.0697	10	17.9753	9.9056	1.0	20
99-2690 DUP	AN-SO-CW2	Water/dilute caustic/NaNO3	8.3154	10	18.4109	10.0955	1.0	20
99-2703	AN-SO-ER1	0.25 M NaNO3	8.0862	10	17.9977	9.9115	1.0	20
99-2704	AN-SO-ER2	0.25 M NaNO3	8.0567	10	18.0884	10.0317	1.0	20
99-2705	AN-SO-REGN	0.1 M NaOH/0.25 M NaNO3	8.0815	10	18.1536	10.0721	1.0	20

Acid Digest procedure #128

analyst MG Steele date 10/28/99

G. Lumetta
ASR 5520
w.p.#

ACL number	client ID	description	vial	aliquot (ml)	sample + vial	sample wt. (g)	mL of each acid	final volume
99-2650	AN107 START	AN107 SUPERNATE -20ML	8.1198	10	20.2734	12.1536	1.1	20
99-2651	AN107-1	AN107 SUPERNATE - 10ML	8.1071	2	10.8547	2.7476	0.5	10
99-2652	AN107-2	AN107 SUPERNATE - 2ML	8.0633	2	10.7068	2.6435	0.6	10
99-2653	AN107-PPT1	Precipitate - redissolved - 20 ml	8.0800	10	18.0253	9.9453	1.1	20
99-2654	AN107 -PPT2	Precipitate - redissolved 0- 20 ml	8.1355	10	18.1172	9.9817	1.0	20
99-2685	AN-SO-L22A	AN-107 supernate	8.1188	2	10.2693	2.1505	0.5	10
99-2686	SO4-Effluent-	AN-107 supernate	8.0159	10	20.0696	12.0537	1.1	20
99-2687	AN-SO-FD1	0.1 M NaOH/0.25 M NaNO3	8.0734	10	18.2572	10.1838	1.0	20
99-2688	AN-SO-FD2	0.1 M NaOH/0.25 M NaNO3	8.1228	10	18.0604	9.9376	1.0	20
99-2689	AN-SO-CW1	Water/dilute caustic/NaNO3	8.0897	10	18.1402	10.0505	1.0	20
99-2690	AN-SO-CW2	Water/dilute caustic/NaNO3	8.0697	10	17.9753	9.9056	1.0	20
99-2690 DUP	AN-SO-CW2	Water/dilute caustic/NaNO3	8.3154	10	18.4109	10.0955	1.0	20
99-2703	AN-SO-ER1	0.25 M NaNO3	8.0862	10	17.9977	9.9115	1.0	20
99-2704	AN-SO-ER2	0.25 M NaNO3	8.0567	10	18.0884	10.0317	1.0	20
99-2705	AN-SO-REGN	0.1 M NaOH/0.25 M NaNO3	8.0815	10	18.1536	10.0721	1.0	20

2650-BLK
+ X-5
1-5-99

Acid Digest procedure #128

analyst Inf. Steele date 12/15/99

ACL number	client ID	description	vial	aliquot (ml)	sample + vial	sample wt. (g)	mL of each acid	final volume
99-2650	ANI07 START	ANI07 SUPERNATE -20ML	8.1198	10	20.2734	12.1536	1.1	20
99-2651	ANI07-1	ANI07 SUPERNATE - 10ML	8.1071	2	10.8547	2.7476	0.5	10
99-2652	ANI07-2	ANI07 SUPERNATE - 2ML	8.0633	2	10.7068	2.6435	0.6	10
99-2653	ANI07-PPT1	Precipitate - redissolved - 20 ml	8.0800	10	18.0253	9.9453	1.1	20
99-2654	ANI07 -PPT2	Precipitate - redissolved 0- 20 ml	8.1355	10	18.1172	9.9817	1.0	20
99-2685	AN-SO-L22A	AN-107 supernate	8.1188	2	10.2693	2.1505	0.5	10
99-2686	SO4-Effluent-	AN-107 supernate	8.0159	10	20.0696	12.0537	1.1	20
99-2687	AN-SO-FD1	0.1 M NaOH/0.25 M NaNO3	8.0734	10	18.2572	10.1838	1.0	20
99-2688	AN-SO-FD2	0.1 M NaOH/0.25 M NaNO3	8.1228	10	18.0604	9.9376	1.0	20
99-2689	AN-SO-CW1	Water/dilute caustic/NaNO3	8.0897	10	18.1402	10.0505	1.0	20
99-2690	AN-SO-CW2	Water/dilute caustic/NaNO3	8.0697	10	17.9753	9.9056	1.0	20
99-2690 DUP	AN-SO-CW2	Water/dilute caustic/NaNO3	8.3154	10	18.4109	10.0955	1.0	20
99-2703	AN-SO-ER1	0.25 M NaNO3	8.0862	10	17.9977	9.9115	1.0	20
99-2704	AN-SO-ER2	0.25 M NaNO3	8.0567	10	18.0884	10.0317	1.0	20
99-2705	AN-SO-REGN	0.1 M NaOH/0.25 M NaNO3	8.0815	10	18.1536	10.0721	1.0	20

Appendix C: Calculations

The holdup quantities were calculated using the following corrections. The holdup upon transfer of the 45-mL concentrate to the filter apparatus was 2 g. Of this, 2 g held up in the beaker, 1.12 g was assigned to the liquid fraction, and 0.88 g was assigned to the wet solid fraction, based on the ratio of liquid and solids recovered. The density of the liquid fraction was determined to be 1.43 g/mL, based on the weight of the 10-mL aliquot of the filtered concentrate set aside for analytical work. This gives a liquid holdup volume of 0.78 mL for the transfer step to the filter apparatus, based on the weight measured for this step. The filtrate was then transferred to a graduated cylinder to measure the volume, which was 22 mL. The holdup on transfer to the graduated cylinder was estimated to be 0.75 mL, based on the density of the liquid (1.43 g/mL) and the sample weight before the transfer (32.3 g) to the graduate. The difference in the sample weight transferred from the graduated cylinder (16.7 g [further processing] + 14.3 g [analytical sample]) account for an additional holdup of 1.3 g (0.91 mL) on the graduated cylinder and filter flask.

Using the same approach, the volume of filtrate transferred to the 100-mL beaker for the second concentration was estimated to be 11.6 mL (16.7 g ÷ 1.43 g/mL). The second concentrate was transferred to a filtration apparatus. The holdup on this transfer was found to be 1.4 g, of which 0.63 g was assigned to the liquid fraction and 0.77 g was assigned to the wet solids, based on the ratio of the recovered amounts. The weight of the filtered liquid was found to be 4.98. The liquid was transferred to a graduated cylinder. The measured volume of the filtrate in the graduated cylinder was 3.0 mL. It was then transferred to a vial and weighed again. The weight after the transfer was found to be 4.53 g. The combined holdup for both these transfers was 0.46 g. Assuming that an equal holdup of filtrate occurred in the transfer from the filtrate receiving vessel to the graduated cylinder and the transfer from the graduated cylinder to the vial, the weight of the liquid in the graduate (3.0 mL) can be estimated to be 4.755, yielding an estimated density of 1.585 g/mL. Using this density, the total volume of the liquid fraction of the concentrate, corrected for holdup, is estimated to be 3.6 mL ((4.98 g ÷ 1.585 g/mL) + [0.63 g ÷ 1.585 g/mL]).

As discussed in the experimental section, the solids from the first concentration step were split among two vials, and the dry weight was normalized to the driest sample (AN107 Solids1B). The holdup amount assigned to the transfer of the wet solids to the filtration apparatus was 0.88 g. An additional 0.9-g holdup was assigned to the transfer of the wet solids from the filter to the two vials. The holdup corrections for the second precipitation include the holdup on transfer of the filtrate to the graduated cylinder (0.45 mL), the holdup for the transfer of the second concentrated slurry to the filter apparatus (0.77 g), and the transfer of the solids from the filter to the vial for drying (0.56 g).

The equations used to calculate the volumetric factors applied to the analytical results are given below:

$$22.0 \text{ mL} + \left(\frac{32.3 \text{ g}}{59.6 \text{ g}} \times \frac{10.0 \text{ mL}}{14.3 \text{ g}} \times 2 \text{ g} \right) = 22.8 \text{ mL} \quad (1: \text{AN107-1})$$

*0.242 g is the analysis sample mass,
20.1 mL is the analysis sample volume,
842 mL is the total volume if all the solids were used to prepare the sample.*

The volumetric factor applied to the starting material sample (AN107-Start) results was 101 mL. The second-step evaporation results are corrected for the quantity of sample removed from the first filtrate.

The initial density of the AN-107 starting material was 1.210 g/mL. Evaporation of the sample to a volume of 45 mL resulted in a slurry with a density of 1.324 g/mL and a filtered liquid with a density of 1.429 g/mL. The slurry resulting from the second concentration step had a density of 1.393 g/mL. As described above, the estimated density of the second filtrate was 1.585 g/mL. Since the analytical results were provided in volumetric units, the mass balance was calculated using corrected volumes.

The following is a sample calculation for sulfate and shows how the values in Table 3.2 were obtained for each analyte. The mass balances for each analyte, corrected for holdup and subsampling, are summarized in Table 3.2.

AN107 Start: The SO_4^{-2} concentration reported for this sample was 4000 ppm. The total sulfate in the starting AN107 material was determined as follows:

$$4000 \text{ mg/L} \times 0.101 \text{ L} = 404 \text{ mg}$$

AN107-1 (filtered liquid): The SO_4^{-2} concentration reported for this sample was 11000 ppm. The quantity of sulfate in the filtered liquid was determined as follows:

$$11000 \text{ mg/L} \times 0.0228 \text{ L} = 250.3 \text{ mg}$$

The percentage left in solution was found to be

$$100 \times 250.3 \text{ mg} \div 404 \text{ mg} = 61.9\%$$

AN107-PPT1 (precipitated solids): The SO_4^{-2} concentration reported for this sample was 72 ppm. The quantity of sulfate in the precipitated solids were determined as follows:

$$72 \text{ mg/L} \times 1.860 \text{ L} = 133.6 \text{ mg}$$

The percentage precipitated from the solution was found to be

$$100 \times 133.6 \text{ mg} \div 404 \text{ mg} = 33.1\%$$

The quantities of sulfate in the concentrated liquid and the precipitate account for 95% of the original sulfate in the AN107-Start sample.

AN107-2 (filtered liquid): The SO_4^{-2} concentration reported for this sample was 5200 ppm.

The quantity of sulfate in the filtered liquid was determined as follows:

$$5200 \text{ mg/L} \times 0.00659 \text{ L} = 34.2 \text{ mg}$$

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