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C-106 High-Level Waste Solids: Washing/Leaching and Solubility Versus Temperature Studies

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Table of Contents

1.0 Introduction

This report describes the results of a test conducted by Battelle to assess the effects of inhibited water washing and caustic leaching on the composition of the Hanford tank C-106 high-level waste (HLW) solids. The objective of this work was to determine the composition of the C-106 solids remaining after washing with 0.01 M NaOH or leaching with 3 M NaOH. Another objective of this test was to determine the solubility of various C-106 components as a function of temperature. The work was conducted according to test plan BNFL-TP-29953-8, Rev. 0, *Determination of the Solubility of HLW Sludge Solids*. The test went according to plan, with only minor deviations from the test plan. The deviations from the test plan are discussed in the experimental section.

2.0 Personnel

The Battelle personnel and their responsibilities in performing this test are given below.

Staff Member	Responsibilities
G.J. Lumetta	Cognizant scientist. Prepared test plan and designed experiment. Supervised performance of the test. Prepared analytical service request. Interpreted data and reported results.
F.V. Hoopes	Hot cell technician. Performed test.
R.C. Lettau	Hot cell technician. Performed test.
D.J. Bates	Statistical analysis of data.
G.F. Piepel	Statistical analysis of data.
M.W. Urie	Managed chemical and radiochemical analytical work.
B.M. Rapko	Technical reviewer.
K.P. Brooks	Task Leader.

3.0 Experimental

Sample Description. The sample used in this test was labeled as C-106-B. This material was a portion of the homogenized C-106 initial composite material prepared from twenty grab samples delivered to PNNL in June 1996. Upon storing in the High-Level Radiochemical Facility (HLRF) for ~3 years, the material had dried and consisted of dried chunks. A spatula was used to break up the larger chunks and mix the material. A 60-g sub-sample was placed into a jar labeled as C-106-B1, and this sub-sample was transferred to the Shielded Analytical Laboratory (SAL) for testing.

Apparatus. The apparatus used consisted of an aluminum heating block placed on a hot plate/stirrer. The hot plate/stirrer was modified so that separate power could be applied to the heating and stirring functions. This allowed for continuous stirring, while the hot plate was powered by a temperature controller. The temperature controller used was a J-KEM Model 270 (J-KEM Electronics, Inc., St. Louis, MO). This temperature controller consists of two separate circuits. One is the temperature control circuit, while the other serves as an over-temperature device, which shuts down the system if a preset temperature is exceeded. The set point for the over-temperature circuit was set at 100°C for this test. A dual K-type thermocouple (model number CASS-116G-12-DUAL, Omega Engineering, Stamford, CT) was used to provide inputs to the temperature controller and over-temperature circuits. Both the J-KEM Model 270 and the dual thermocouple were calibrated before use. The aluminum heating block contained two wells. A vial containing water was placed in one of the wells, with the thermocouple wedged between this vial and the aluminum block. The vessel containing the sample was placed in the other well.

Procedure.[a] In writing the test plan, it was assumed the HLW sludge material would exist as a wet solid. Thus, the test plan called for mixing the slurry to homogenize; 0.1 M NaOH was to be added to assist in homogenization if necessary. However, because the C-106 solids had dried, reasonable homogenization could be achieved by breaking up the chunky material and mixing with a spatula. Weighed aliquots of the homogenized dry solids were then taken for the various tests: 1) solubility versus temperature, 2) determination of aqueous-insoluble fraction, and 3) determination of caustic-insoluble fraction.

Solubility Versus Temperature. A 10.1157-g aliquot was transferred from C-106-B1 to a 60-mL high density polyethylene (HDPE) bottle (this bottle also contained a Teflon®-coated magnetic stir bar). Aqueous 0.1 M NaOH (~50 mL, 49.43 g) was added and the bottle was capped. The sample was then heated and stirred at 30 ± 2°C for 19 h. During this time, stirring was inconsistent and at one point had stopped. A larger stir bar was added which lead to more consistent stirring. The sample stirred at 30 ± 2°C for another 3 h. Two aliquots (4-mL each) were taken for analysis. Each aliquot was immediately filtered through a 0.45-μm nylon syringe filter that had been preheated by immersion in a boiling water bath. The filter was preheated to reduce the possibility of precipitation during the filtration step. The temperature was increased to 40 ± 2°C and the sample was stirred for 18.5 h. The mixture was sampled in the same manner as described above. The temperature was increased to 50 ± 2°C and the sample was stirred for 24 h. Again, the mixture was sampled in the same manner as described above. The filtered samples were subjected to the following analytical procedures: ion chromatography (IC) for anions, total organic carbon (TOC), total inorganic carbon (TIC), acid digestion, inductively-coupled plasma atomic emission spectroscopy (ICP/AES), inductively-coupled plasma mass spectrometry (ICP-MS) for ^{99}Tc, ^{90}Sr, total alpha, total uranium, and gamma energy analysis (GEA).

Determination of Aqueous-Insoluble Fraction. A 23.4777-g aliquot was transferred from C-106-B1 to a 125-mL high density polyethylene (HDPE) bottle (this bottle also contained a Teflon®-coated magnetic stir bar). The bottle was filled to capacity with aqueous 0.01 M NaOH (104.41 g added). The bottle was equipped with a condenser tube, which allowed the system to vent during heating, but minimized evaporation. The mixture was heated and stirred at 85 ± 2°C for 18 h. The test plan indicated that the washing slurry should be cooled prior to filtration, but per instructions from BNFL, the slurry was filtered while hot. The hot washing slurry was filtered through a pre-weighed 0.45-μm nylon filtration unit. The weight of the filtrate was 102.76 g.

Several ~10-mL aliquots of 0.01 M NaOH were used to transfer the filtered solids back into the HDPE bottle. The total slurry volume was made to ~100 mL with additional 0.01 M NaOH (total slurry weight = 108.83 g). The mixture was heated and stirred at 85 ± 2°C for 23 h. The washing slurry was again filtered while hot, yielding 94.00 g of washing solution. This process was repeated a third time. For the final washing step, the slurry was heated at 85 ± 2°C for 27 h, and 125.92 g of

[a] See Appendix A for a copy of the test plan and procedural notes.

washing liquid was collected. A composite sample of the three wash solutions was prepared for analysis.

After the final washing step, the filtered solids were transferred to a pre-weighed glass jar using demonized water. Excess water was evaporated at 80°C, then the solids were dried overnight at 105°C yielding 2.0286 g of dried washed solids. However, a significant fraction of the washed solids could not be recovered in this manner because they were stuck to the magnetic stir bar (Figure 1). These magnetic solids were treated differently. The stir bar with the solids was placed in a glass jar and the gross weight was determined (99.8543 g). Concentrated (12 M, 10 mL) HCl was added and the mixture was stirred. After ~3.5 h, there was still a small amount of solid remaining. Most of this solid dissolved upon gently heating for ~1 h. However, dilution of this solution led to precipitation of solids that would not re-dissolve, even in HCl/HNO_3. After transferring to a beaker, excess acid was boiled off and the resulting dried solids were submitted for analysis. The weight of the empty glass jar+stir bar was determined to be 99.7529 g, so the weight of the magnetic solids was deduced to be 0.1014 g.

Determination of Caustic-Insoluble Fraction. A 24.7022-g aliquot was transferred from C-106-B1 to a 125-mL high density polyethylene (HDPE) bottle (this bottle also contained a Teflon®-coated magnetic stir bar). Aqueous NaOH (3 M, 108.08 g) was added. The bottle was equipped with a condenser tube, which allowed the system to vent during heating, but minimized evaporation. The mixture was heated and stirred at 85 ± 2°C for 20 h. As per instructions from BNFL, the leaching slurry was filtered while hot. The hot slurry was filtered through a pre-weighed 0.45-μm nylon filtration unit. The weight of the filtrate was 104.07 g. A sample of this leaching solution was taken for analysis.

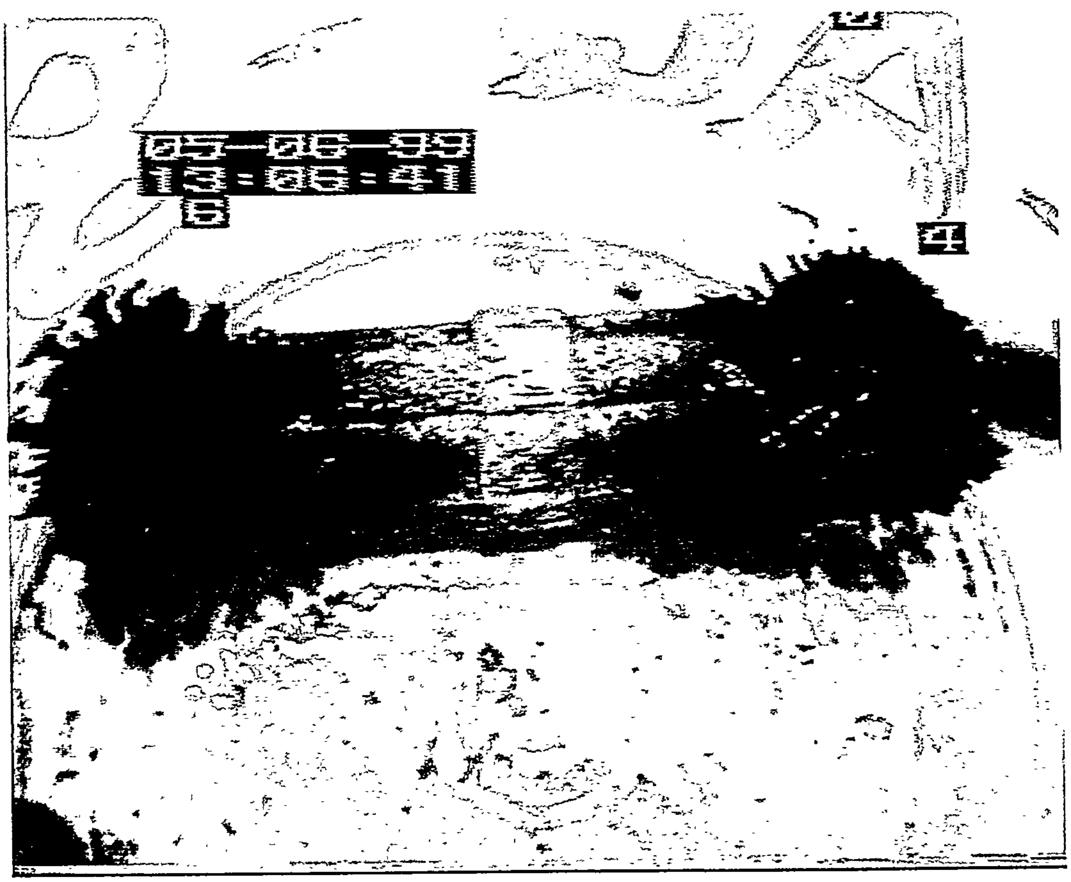

Figure 1. Magnetic Solids on Stir Bar from Dilute-Hydroxide Washing Test

Most of the filtered solids were transferred back into the HDPE bottle using a spatula. Several ~10-mL aliquots of 0.01 M NaOH were used to transfer the remaining filtered solids back into the HDPE bottle. The slurry volume was made to ~100 mL with additional 0.01 M NaOH (total slurry weight = 114.88 g). The mixture was heated and stirred at 85 ± 2°C for 23 h. The washing slurry was again filtered while hot yielding 91.73 g of washing solution. The washing process was repeated. For the final washing step, the slurry was heated at 85 ± 2°C for 28 h, and 123.95 g of washing liquid was collected. A composite sample of the two wash solutions was prepared for analysis.

After the final washing step, the filtered solids were transferred to a pre-weighed glass jar using deionized water. Excess water was evaporated at 80°C, then the solids were dried overnight at 105°C yielding 5.8149 g of dried leached solids. The appearance of these solids was unusual. A white solid had collected around the walls of the jar, while a brown solid remained in the bottom of the jar. Before analyzing, the white solid was scraped from the wall of the jar and mixed with the brown solid using a spatula.

As with the washing test, a significant fraction of the leached solids could not be recovered by filtration because they were stuck to the magnetic stir bar. These magnetic solids were dissolved in HCl/HNO_3, then excess acid was boiled off and the resulting dried solids were submitted for analysis. The weight of the magnetic solids was deduced to be 1.9464 g. Note that this latter value is much greater than what was obtained for the magnetic washed solids. As the amount of magnetic solids left after dilute hydroxide washing visually appeared to be similar to that left after leaching, the value for the washed solids is suspect.

4.0 Results

4.1 Solubility Versus Temperature

Tables 1, 2, and 3 present the concentrations of various waste components at 30, 40, and 50°C, respectively. Two sets of values are presented in each table. The first set of values is the analyte concentrations as determined directly on the aliquots analyzed. In the second set of values, the concentrations have been adjusted for loss in the sample weight that occurred between the time the aliquot was taken and the time the analyses were initiated. These adjustments were made assuming the weight losses were due to evaporation.[a]

Tables 4 and 5 show the changes in the concentrations at 40 and 50°C relative to those at 30°C. Because aliquot C106-SOL-50-1 appeared to have leaked, only the concentration values obtained for aliquot C106-SOL-50-2 were used to determine the adjusted concentration changes at 50°C relative to 30°C. Appendix D discusses a graphical analysis of the data, as well as linear regression results of fitting component concentrations versus temperature. Based on this data set, only limited conclusions can be drawn. The following discussion will be limited to those analytes for which meaningful conclusions can be drawn. The discussion is organized according to the following types of components: 1) radionuclides, 2) bulk metals and carbon, and 3) anions.

(a) In the case of aliquot C106-SOL-50-1, there was solid encrusted on the outside of the vial suggesting that material actually leaked from the sample vial, which probably contributed more to the weight loss than did evaporation.

Radionuclides. The data indicate that the ^{137}Cs concentration increased with temperature. Based on the adjusted concentration values, the ^{137}Cs concentration increased 16% when the temperature was increased from 30 to 40°C. A 30% increase in the ^{137}Cs concentration occurred in going from 30 to 50°C. Linear regressions of the adjusted ^{137}Cs concentrations versus temperature had a statistically significant positive slope (see Appendix D). However comparison of the percent change in the concentration to the standard deviation in the percent change suggests the increase is not statistically significant at the 90% confidence level (Table 5). Results for the other radionuclides were not statistically meaningful.

Bulk Metals and Carbon. Only linear regressions of the adjusted Cr, Cu, Ni, P, Si, and U concentrations versus temperature had statistically significant positive slopes (see Appendix D). The analysis presented in Table 5 indicates the increase in the U concentration is not statistically significant at the 90% confidence level. The considerable variability observed for many of the other components might have been due to precipitation of these components. Visual inspection of the analytical samples immediately prior to processing indicated the presence of precipitates. Chromium, Cu, and Ni all showed substantial increases, with concentrations more than doubling in going from 30 to 50°C. Phosphorus and Si displayed more modest concentration increases. The increases for Si should be viewed as qualitative because the analytical process blank contained a relatively high Si content.

Linear regressions of the total organic carbon (TOC) and total inorganic carbon (TIC) concentrations versus temperature showed no statistically significant trends (Appendix D). Thus, no conclusive trend was seen in these data.

Anions. In all cases, F^-, NO_3^-, SO_4^{2-}, and PO_4^{3-} were below the analytical detection limit. Linear regressions of the adjusted Cl^- and $C_2O_4^{2-}$ concentrations versus temperature had statistically significant positive slopes (see Appendix D). Based on the adjusted concentration values, the Cl^- concentration increased 14% when the temperature was increased from 30 to 40°C and a 20% increase in the Cl^- concentration occurred in going from 30 to 50°C. Similarly, the $C_2O_4^{2-}$ concentration increased 16% when the temperature was increased from 30 to 40°C, and 27% in going from 30 to 50°C.

4.2 Dilute Hydroxide Washing

Table 6 presents the concentration of the analyzed C-106 components in a composite of the three wash solutions. The composite wash sample was prepared by mixing measured quantities of each wash solution; the relative weight of each wash solution used corresponded to the fraction of the total wash solution represented by each. The composite wash solution was weighed immediately before analytical work was begun. The total weight of the sample had decreased 6.3% since the time the composite was first prepared. The concentrations determined were adjusted for this weight loss, assuming the weight loss was due to evaporation. The adjusted concentrations were then multiplied by the total combined weight of the three washing solutions (322.68 g) to yield the quantity of each component present in the wash solutions.

Table 7 presents the results of the analysis of the non-magnetic fraction of the dilute hydroxide-washed C-106 solids. The solids were solubilized for ICP/AES analysis by KOH and Na_2O_2 fusion methods. Duplicate fusions and ICP/AES analyses were done for each type of fusion. Mean values from these determinations are presented in the table along with the standard deviation from the mean and the relative error. The relative error was obtained by the following formula: %RSD = 100(Std.Dev./Mean). In most cases the relative error is less than 20% for the elements determined by ICP/AES, indicating good agreement between the duplicate measurements. For those ICP/AES analytes where the relative error was significantly greater than 20%, the concentrations of these analytes were typically low or near the detection limit. Closer inspection of the P data indicates differences between the KOH and Na_2O_2 fusion methods. Analysis of the solutions obtained by the KOH fusion indicated higher P concentrations than obtained by the Na_2O_2 fusion. This suggests the Na_2O_2 fusion failed to completely dissolve the P contained in the non-magnetic washed solids. Indeed, for most analytes examined, the Na_2O_2 fusion method resulted in concentration values less than those obtained by the KOH method. For this reason, only the values obtained from the KOH fusion were used to determine the quantity of each component in the washed solids. The exceptions to this were K and Ni, which were only available from the Na_2O_2 fusion.

The Hg concentration was determined on the non-magnetic washed solids by cold vapor atomic absorption spectrophotometry following an oxidative acidic leaching of the solids. The mean Hg concentration was 351 μg/g and good agreement was achieved between duplicates.

TIC/TOC determination on the non-magnetic washed solids was performed using the hot persulfate method. This analysis was performed directly on the washed solids (not on fused material). Very good reproducibility was achieved between duplicate TIC/TOC analyses. To date, no reliable method has been developed to quantify the anions present in Hanford tank solids. Anion (Cl^-, F^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, and $C_2O_4^{2-}$) analysis was done by IC on a solution obtained by leaching the washed solids with deionized water. This in essence yielded the water-soluble anions not completely removed by the washing test. The results indicate significant additional soluble Cl^-, NO_3^-, and $C_2O_4^{2-}$ remained in the washed solids (or was present in interstitial liquid prior to drying), although reproducibility between duplicate measurements was poor. The IC results for "C106-AQ-8DUP" were approximately a factor of two lower than those for "C106-AQ-8." Review of the sample preparation bench sheets revealed no obvious cause for this discrepancy. The $C_2O_4^{2-}$ concentration of 20,300 μg/g (the value obtained for C106-AQ-8) corresponds to 5,540 μg/g organic carbon. Thus, ~19% of the TOC in the non-magnetic fraction of the washed solids is attributable to oxalate. The low PO_4^{3-} concentration revealed by IC suggests that P found by ICP is indeed due to some water-insoluble P-containing phase(s).

Cyanide analysis on the non-magnetic fraction of the washed solids revealed very little CN^- to be present. Reproducibility between duplicate CN^- analyses was poor. This might have been due to sample inhomogeneity. Ammonia was determined by ion-selective electrode using water-slurries of the solids. Very little (~4 μg/g) NH_3 was indicated; however the value should be treated with caution since the solids were dried at 105°C prior to analysis, which would likely have volatilized any NH_3 present.

Radiochemical analyses were performed on the solutions prepared by KOH fusion. Cesium-137, ^{241}Am, ^{154}Eu, and ^{155}Eu were determined by gamma spectroscopy. Americium-241 was also determined by alpha spectroscopy following Pu separation , as were ^{238}Pu, $^{239+240}$Pu, ^{242}Cm, and $^{243+244}$Cm. The total alpha values reported were obtained by summing the alpha activity indicated by the activities of the various alpha-emitters indicated in the alpha spectrum. Direct-mount determination of the total alpha activity was not reliable due to self-attenuation. Technetium-99, ^{129}I, ^{235}U, ^{238}U, ^{237}Np, ^{239}Pu, and ^{240}Pu were determined by ICP-MS. Strontium-90 was determined by proportional beta-counting following separation of this isotope.

With the exception of ^{242}Cm, agreement between duplicate measurements was good. The values obtained for ^{241}Am by gamma and alpha spectroscopies agreed within 20%. On the other hand, there were inconsistencies between the ICP-MS results and the alpha spectroscopic results. The combined activities for ^{239}Pu and ^{240}Pu as determined by ICP-MS were 3.81 μCi/g, yet the $^{239+240}$Pu value obtained by alpha spectroscopy was 27% lower (2.78 μCi/g). To be conservative, the higher value should probably be used. There was also inconsistency regarding the U analysis. The ICP-MS analysis revealed ~275 $\mu g/g$ (^{235}U + ^{238}U), but only 176 μg total U was indicated by laser fluorimetry analysis.

Table 8 presents the results of the analysis of the magnetic fraction of the dilute hydroxide-washed C-106 solids. TIC/TOC, IC, CN^-, and NH_3 analyses were not performed on this material as the acid dissolution conducted upon the magnetic solids would have influenced the results of these analyses. As expected, the magnetic solids were very rich in Fe (~50 wt%). The values for Ag, Ca, P, and Si had relatively high relative errors (> 20%) between the KOH and Na_2O_2 fusions. The reason for the relatively high error for Ag is most probably the use of HCl in the solubilization of the fusion fluxes. The process blank had a high value for Ca, so the Ca value is suspect. Phosphorus was near the analytical detection limit, so high uncertainties are expected for this element. The error in Si is possibly due to incomplete dissolution of this element in the Na_2O_2 fusion preparation. Similar to the analysis of the nonmagnetic fraction, for most analytes examined, the Na_2O_2 fusion method resulted in concentration values less than those obtained by the KOH method. For this reason, only the values obtained from the KOH fusion were used to determine the quantity of each component in the washed solids.

Table 9 presents the composition of the dilute hydroxide-washed C-106 solids (magnetic plus non-magnetic fractions) and the percent of each component removed by dilute hydroxide washing. In addition, the composition of the "untreated" C-106 sample used in this test is presented. These values were obtained by summing the amount of the given component found in the wash solutions (Table 6), the non-magnetic washed solids (Table 7), and magnetic washed solids (Table 8), then dividing this total by the weight of the C-106 sample used. The washed solids were dominated by Fe (28.6 wt%), Si (10.5 wt%), Al (6.2 wt%), Na (5.4 wt%) and Ca (1.5 wt%). The concentrations of the major radionuclides contained in the washed solids were 5.7 μCi TRU/g (as indicated by the total alpha concentration), 2.6 μCi ^{241}Am/g, 3.0 μCi ^{239}Pu/g, 908 μCi ^{90}Sr/g, and 377 μCi ^{137}Cs/g, indicating the solids should be treated as HLW.

Upon standing for six days, a white precipitate formed in the first wash solution. This solid material was collected by filtration so that it could be analyzed. The mass of this solid was 0.765 g after air-drying at ambient temperature. The solid was readily soluble in water. ICP/AES analysis indicated this material to be 22.3 wt% Na. No other metals were detected above trace levels. IC analysis indicated the solid was 49.5 wt% $C_2O_4^{2-}$. Thus, it can be concluded that this material is predominantly $Na_2C_2O_4 \bullet 2H_2O$ (Theoretical: 27.0 wt% Na, 51.8 wt% C_2O_4).

4.3 Caustic Leaching

Table 10 presents the concentration of the analyzed C-106 components in the caustic leach solution and in a composite of the two wash solutions. The composite wash sample was prepared by mixing measured quantities of each wash solution; the relative weight of each wash solution used corresponded to the fraction of the total wash solution represented by each. The samples were weighed immediately before analytical work was begun. The weight of the leach solution sample had decreased 34.6% and that of the composite wash solution sample had decrease 6.3% since the time the samples were first prepared. The concentrations determined were adjusted for this weight loss, assuming the weight loss was due to evaporation. The adjusted concentrations were then multiplied by the weight of the leach solution (104.07 g) or the combined weight of the two wash solutions (215.68 g) to yield the quantity of each component present in the leach and the wash solutions, respectively.

Table 11 presents the results of the analysis of the non-magnetic fraction of the caustic leached C-106 solids. Analysis of these solids was conducted in the same way as for the dilute hydroxide-washed solids. In most cases the relative error is less than 20% for the elements determined by

ICP/AES, indicating good agreement between duplicate measurements. In some cases (Cu and P from the Na_2O_2 fusions) where the relative error was significantly greater than 20%, the concentrations were near the detection limit. However, this is not the case for some of the other analytes with high relative errors. For example, Ag had a 55% relative error (Na_2O_2 fusion). In this case, the value of 1,460 μg/g obtained for one of the Na_2O_2 fusion duplicates seems disproportionately high. (Again, this is likely due to use of HCl in the solubilization procedure.) There are also significant differences in the duplicate Zr analyses. The reasons for these discrepancies are not clear. The Si values are also inconsistent. In this case, the values obtained by the KOH fusion method appear to be ~2 times higher than those obtained by the Na_2O_2 fusion method. Perhaps not all the Si was solubilized by the Na_2O_2 fusion. As was done with the dilute hydroxide washed solids, only the values from the KOH fusion were used to determine the amount of each component in the leached solids.

The IC results indicate significant additional soluble $C_2O_4^{2-}$ remained in the washed solids (or interstitial liquid prior to drying). This reflects dependence of sodium oxalate solubility on the Na concentration. The $C_2O_4^{2-}$ concentration of 394,500 μg/g corresponds to 148,000 μg/g organic carbon. Thus, virtually all the TOC in the non-magnetic fraction of the leached solids is attributable to oxalate.

Cyanide analysis on the non-magnetic fraction of the leached solids revealed very little CN^- to be present. Ammonia was determined by ion-selective electrode using water-slurries of the solids. Very little (~10 μg/g) NH_3 was indicated; however the value should be treated with caution since the solids were dried at 105°C prior to analysis.

The relative errors for many of the radionuclides (^{155}Eu, ^{14}C, ^{129}I, Pu isotopes, and Cm isotopes) were greater than 20%, indicating poor reproducibility between duplicates. The values obtained for ^{241}Am by gamma and alpha spectroscopies agreed within 20%. Again, the combined activities for ^{239}Pu and ^{240}Pu as determined by ICP-MS were significantly higher than the $^{239+240}$Pu value obtained by alpha spectroscopy. To be conservative, the higher value should probably be used. Also like the washed solids, the U values obtained by ICP-MS [~285 μg/g (^{235}U + ^{238}U)], disagreed with the value of 176 μg total U indicated by laser fluorimetry analysis.

Table 12 presents the results of the analysis of the magnetic fraction of the caustic-leached C-106 solids. TIC/TOC, IC, CN^-, and NH_3 analyses were not performed on this material as the acid dissolution conducted upon the magnetic solids would have influenced the results of these analyses. As expected, the magnetic solids were very rich in Fe (~44 wt%). There was more residual Na present in these solids than in magnetic solids from the dilute hydroxide wash (compare Tables 8 and 12), which attributed to the slightly less Fe weight percent. However, the nature of the magnetic solid obtained from the caustic leaching test is undoubtedly the same as that obtained from the dilute hydroxide washing test (except that the Na salts were not washed out to the same extent).

Table 13 presents the composition of the caustic-leached C-106 solids (magnetic plus non-magnetic fractions) and the percent of each component removed by caustic leaching. In addition, the composition of the "untreated" C-106 sample used in this test is presented. These values were obtained by summing the amount of the given component found in the wash solutions (Table 10), the non-magnetic leached solids (Table 11), and magnetic leached solids (Table 12), then dividing this total by the weight of the C-106 sample used. The leached solids were dominated by Fe (17.2 wt%), Si (7.8 wt%), Na (13.6 wt%), Al (2.6 wt%) and Ca (1.0 wt%). The higher relative Na content in the leached solids (compared to the dilute hydroxide-washed solids) is largely attributable to the sodium oxalate in the leached solids. The concentrations of the major radionuclides contained in the washed solids were 1.7 μCi TRU/g (as indicated by the total alpha concentration), 0.8 μCi ^{241}Am/g, 1.0 μCi ^{239}Pu/g, 260 μCi ^{90}Sr/g, and 90 μCi ^{137}Cs/g, indicating the solids should be treated as HLW.

It should be noted that the composition for the original C-106 solid listed in Table 9 should agree with that listed in Table 13. However, the compositions vary widely. This suggests sample inhomogeneity. That is, the aliquot taken for dilute hydroxide washing was different from that taken for caustic leaching.

4.4 Comparison to Previous Work

Table 14 presents a comparison of the current work to previous studies of C-106 sludge. To facilitate comparisons between the various data sets, the concentrations have been normalized to the Fe concentration. This treatment of the data assumes that no Fe is removed by washing or leaching; this assumption is validated by the low Fe concentrations in the washing and leaching solutions. Clearly, there are differences in the composition of the dilute hydroxide-washed solid determined in 1996 compared to the current work. This could be due to differences in the sample aliquots used or the different conditions used in the earlier test. Alternatively, the difference might be due to some of the Fe being "missed" in the earlier test. In the test conducted in 1996, a magnetic stir bar was used to agitate the washing slurries. Presumably, that sample aliquot contained magnetic Fe like the sample used in the current work. As no attempt was made to recover the magnetic Fe in the previous work, that fraction of the Fe would have been missed in the analysis of the solids. This would lead to the concentrations relative to Fe that are higher than those found in the current work. Indeed, the relative concentrations reported in 1996 are consistently higher than those found in the current work. Furthermore, they are generally high by a factor of ~2. For example, the relative Al concentration from 1996 (491,747 μg/g Fe) is about 2.3 times that found in this work (214,892 μg/g Fe). Similarly, the Cr concentration (7,399 μg/g Fe) from 1996 is 1.9 times that found in this work (3,894 μg/g). However, some of the other analytes (e.g., Ca and Si) differ by factors other than 2.

Similar trends are seen for the caustic-leached solids. That is, the concentrations relative to Fe tend to be less in the current work compared to the work conducted by Lumetta et al. in 1996. However, the concentrations relative to Fe in the current work also tend to be less than those found in the bench-scale caustic leaching test conducted by Brooks et al. (1997). In the latter test, no magnetic stir bars were used. So, at least in this case, the differences must be due to differences in the composition of the starting sample or to differences in the specific leaching conditions used in the two tests.

5.0 Conclusions and Recommendations

The solubility versus temperature test indicated that the concentrations of ^{137}Cs, Cr, Cu, Ni, P, Si, Cl^- and $C_2O_4^{2-}$ increased with temperature. Data for many of the other analytes were scattered to the point that statistically meaningful conclusions could not be drawn. The considerable variability observed for many of the components might have been due to precipitation of these components. Also, the apparent evaporation of the samples during storage might have also contributed to the experimental uncertainty. It is recommended that the solubility versus temperature test plan be revised for future tests. The revised test should allow for larger sample sizes, immediate acidification of analytical samples (where appropriate), and should describe actions to be taken to minimize sample evaporation during interim storage.

Dilute hydroxide washing largely removed the Na salts from the C-106 sludge. Only 14% of the Al, 8% of the Cr, and 36% of the P were removed by dilute hydroxide washing. Surprisingly, 86% of the U was removed in the dilute hydroxide washing process. Cesium-137 (56%) and ^{99}Tc (76%) were appreciably removed by dilute hydroxide washing, whereas the transuranic elements (as represented by the total alpha data) showed little solubility in the washing solutions.

Caustic leaching resulted in only modest increases in Al (22%), Cr (27%), P (41%), and ^{137}Cs (68%) removal. The amount of ^{99}Tc removed appeared to drop slightly, but the changes observed are within the experimental uncertainty. The caustic-leached solids contained considerable sodium oxalate. More extensive washing of the caustic-leached solids would be required to remove this. For future tests, it is recommended that the caustic leaching test plan be revised to include more extensive washing of the leached solids.

Considerable uncertainty was introduced in the washing and leaching tests by using a magnetic stir bar. A large fraction of the solids was magnetic in nature and adhered to the stir bar. It is recommended that future tests with C-106 sludge avoid the use of magnetic stir bars. In addition, future washing and leaching tests should allow for acidification of analytical samples (where appropriate) to prevent precipitation, and should describe actions to be taken to minimize sample evaporation during interim storage. Sampling of dry solids should not be done in the future because this apparently can result in significant sample inhomogeneity. Rather, dry sludge samples should be slurried in dilute hydroxide or water prior to pulling samples for testing.

6.0 References

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Lumetta, G.J., M.J. Wagner, F.V. Hoopes, and R.T. Steele. 1996. *Washing and Caustic Leaching of Hanford Tank C-106 Sludge*, PNNL-11381, Pacific Northwest National Laboratory, Richland, Washington.

Table 1. C-106 Component Concentrations in Solution at 30°C.[a]

	Concentration at 30°C, Unadjusted					Concentration at 30°C, Adjusted[b]				
Analyte	C106-SOL-30-1	C106-SOL-30-2	Mean	Std. Dev.	% RSD	C106-SOL-30-1	C106-SOL-30-2	Mean	Std. Dev.	% RSD
Cesium-137	4.57	4.62	4.60	0.04	0.8	4.16	3.59	3.87	0.40	10.4
Strontium-90	0.0944	0.0639	0.0792	0.0216	27.2	0.086	0.050	0.068	0.026	37.8
Technetium-99	1.96E-03	1.40E-03	1.68E-03	3.92E-04	23.4	1.78E-03	1.09E-03	1.43E-03	4.88E-04	34.1
Americium-241	< 6E-03	< 6E-03	< 6E-03	--	--	< 5E-03	< 5E-03	< 5E-03	--	--
Europium-154	< 4E-04	< 6E-04	< 6E-04	--	--	< 4E-04	< 5E-04	< 5E-04	--	--
Europium-155	< 6E-03	< 6E-03	< 6E-03	--	--	< 5E-03	< 5E-03	< 5E-03	--	--
Total Alpha	2.15E-03	1.43E-03	1.79E-03	5.09E-04	28.4	1.95E-03	1.11E-03	1.53E-03	5.97E-04	39.0
Ag	1.81	(0.97)	(1.39)	0.59	42.7	1.65	(0.75)	(1.20)	0.63	52.6
Al	286	147	217	98	45.4	260	114	187	103	55.2
Ba	(0.27)	(0.27)	(0.27)	0.00	0.0	(0.25)	(0.21)	(0.23)	0.03	11.2
Ca	(3.2)	< 4.8	< 5.0	--	--	(2.9)	< 3.7	(3.3)	0.6	--
Cd	< 0.15	< 0.3	< 0.3	--	--	< 0.14	< 0.2	< 0.2	--	--
Co	< 0.3	< 0.5	< 0.5	--	--	< 0.3	< 0.4	< 0.4	--	--
Cr	(1.7)	(1.7)	(1.7)	0.0	0.0	(1.5)	(1.3)	(1.4)	0.2	11.2
Cu	(0.92)	(0.75)	(0.84)	0.12	14.4	(0.84)	(0.58)	(0.71)	0.18	25.4
Fe	(0.8)	< 0.5	< 0.9	--	--	(0.7)	< 0.4	< 0.8	--	--
K	< 20	< 39	< 39	--	--	< 18	< 30	< 30	--	--
La	< 0.3	< 0.5	< 0.5	--	--	< 0.2	< 0.4	< 0.4	--	--
Mg	< 1	< 2	< 2	--	--	< 1	< 2	< 2	--	--
Mn	< 0.1	< 0.1	< 0.1	--	--	< 0.1	< 0.1	< 0.1	--	--
Mo	(0.82)	(0.73)	(0.78)	0.06	8.2	(0.75)	(0.57)	(0.66)	0.13	19.3
Na	17800	16700	17250	778	4.5	16186	12964	14575	2278	15.6
Ni	(1.3)	(1.2)	(1.3)	0.1	5.7	(1.2)	(0.9)	(1.1)	0.2	16.8
P	43.9	42.2	43.05	1	2.8	39.9	32.8	36.3	5.1	13.9
Pb	< 0.6	< 1.2	< 1.2	--	--	< 0.5	< 0.9	< 0.9	--	--
Si[c]	29.1	29.2	29.2	0	0.2	26.5	22.7	24.6	2.7	10.9
Ti	(0.15)	(0.15)	(0.2)	(0.0)	0.0	(0.14)	(0.12)	(0.1)	(0.0)	11.2
U	23.4	27.0	25.2	2.5	10.1	21.3	21.0	21.1	0.2	1.1
Zn	< 0.2	< 0.4	< 0.4	--	--	< 0.2	< 0.3	< 0.3	--	--
Zr	< 0.3	< 0.5	< 0.5	--	--	< 0.3	< 0.4	< 0.4	--	--
TOC	6628	7041	6835	292	4.3	6027	5466	5746	397	6.9
TIC	1795	1841	1818	33	1.8	1632	1429	1530	143	9.4
Cl^-	410	440	425	21	5.0	373	342	357	22	6.2
F^-	< 250	< 250	< 250	--	--	< 227	< 194	< 227	--	--
NO_3^-	< 500	< 500	< 500	--	--	< 455	< 388	< 455	--	--
SO_4^{2-}	< 500	< 500	< 500	--	--	< 455	< 388	< 455	--	--
PO_4^{3-}	< 500	< 500	< 500	--	--	< 455	< 388	< 455	--	--
$C_2O_4^{2-}$	21400	22700	22050	919	4.2	19459	17622	18540	1299	7.0

(a) Concentrations for radionuclides are in units of µCi/g; all other components are in units of µg/g. Values in parentheses are within 10 times the analytical detection limit and have relative uncertainties > 15%.
(b) Value adjusted for loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(c) The process blank had a relatively high Si content.

Table 2. C-106 Component Concentrations in Solution at 40°C.[a]

Analyte	Concentration at 40°C, Unadjusted					Concentration at 40°C, Adjusted[b]				
	C106-SOL-40-1	C106-SOL-40-2	Mean	Std. Dev.	% RSD	C106-SOL-40-1	C106-SOL-40-2	Mean	Std. Dev.	% RSD
Cesium-137	5.31	4.86	5.09	0.32	6.3	4.77	4.24	4.50	0.37	8.3
Strontium-90	0.1	0.143	0.122	0.030	25.0	0.090	0.125	0.107	0.025	23.1
Technetium-99	1.65E-03	1.56E-03	1.61E-03	6.37E-05	4.0	1.48E-03	1.36E-03	1.42E-03	8.52E-05	6.0
Americium-241	< 7E-03	< 6E-03	< 7E-03	--	--	< 6E-03	< 5E-03	< 6E-03	--	--
Europium-154	< 6E-04	< 4E-04	< 6E-04	--	--	< 5E-04	< 3E-04	< 5E-04	--	--
Europium-155	< 7E-03	< 6E-03	< 7E-03	--	--	< 6E-03	< 5E-03	< 6E-03	--	--
Total Alpha	1.61E-03	2.93E-03	2.27E-03	9.33E-04	41.1	1.45E-03	2.56E-03	2.00E-03	7.85E-04	39.3
Ag	(1.6)	(2.6)	(2.1)	(0.7)	33.7	(1.4)	(2.3)	(1.9)	(0.6)	31.8
Al	67.9	333	200	187	93.5	61	291	176	162	92.4
Ba	(0.27)	(0.26)	(0.27)	0.01	2.7	(0.24)	(0.23)	(0.23)	0.01	4.7
Ca	< 5	(5.2)	< 6	--	--	< 4	(4.5)	< 5	--	--
Cd	< 0.3	< 0.3	< 0.3	--	--	< 0.3	< 0.3	< 0.3	--	--
Co	< 0.5	< 0.5	< 0.5	--	--	< 0.4	< 0.4	< 0.4	--	--
Cr	(2.8)	(3.4)	(3.1)	0.4	13.7	(2.5)	(3.0)	(2.7)	0.3	11.7
Cu	(1.0)	(1.2)	(1.1)	(0.1)	12.9	(0.9)	(1.0)	(1.0)	(0.1)	10.9
Fe	(1.0)	(3.6)	(2.3)	(1.8)	79.9	(0.9)	(3.1)	(2.0)	(1.6)	78.5
K	< 38	< 38	< 38	--	--	< 34	< 33	< 34	--	--
La	< 0.5	< 0.5	< 0.5	--	--	< 0.4	< 0.4	< 0.4	--	--
Mg	< 2	< 2	< 2	--	--	< 2	< 2	< 2	--	--
Mn	< 0.1	< 0.1	< 0.1	--	--	< 0.1	< 0.1	< 0.1	--	--
Mo	(0.76)	(0.81)	(0.79)	0.04	4.5	(0.68)	(0.71)	(0.69)	0.02	2.5
Na	18200	17100	17650	778	4.4	16338	14918	15628	1004	6.4
Ni	(1.6)	(2.0)	(1.8)	0.3	15.7	(1.4)	(1.7)	(1.6)	0.2	13.7
P	44.7	45.7	45.2	1	1.6	40.1	39.9	40.0	0	0.5
Pb	< 1.2	< 1.2	< 1.2	--	--	< 1.1	< 1.0	< 1.1	--	--
Si[c]	32.0	29.3	30.7	2	6.2	28.7	25.6	27.1	2	8.2
Ti	(0.16)	(0.13)	(0.1)	(0.0)	14.6	(0.14)	(0.11)	(0.1)	(0.0)	16.6
U	29.3	25.8	27.6	2.5	9.0	26.3	22.5	24.4	2.7	11.0
Zn	< 0.4	< 0.4	< 0.4	--	--	< 0.4	< 0.3	< 0.4	--	--
Zr	< 0.5	< 0.5	< 0.5	--	--	< 0.4	< 0.4	< 0.4	--	--
TOC	7172	6808	6990	258	3.7	6438	5939	6188	353	5.7
TIC	1912	1682	1797	163	9.1	1716	1467	1592	176	11.1
Cl^-	470	450	460	14	3.1	422	393	407	21	5.1
F^-	< 250	< 250	< 250	--	--	< 224	< 218	< 224	--	--
NO_3^-	< 500	< 500	< 500	--	--	< 449	< 436	< 449	--	--
SO_4^{2-}	< 500	< 500	< 500	--	--	< 449	< 436	< 449	--	--
PO_4^{3-}	< 500	< 500	< 500	--	--	< 449	< 436	< 449	--	--
$C_2O_4^{2-}$	24300	24300	24300	0	0.0	21814	21199	21506	435	2.0

(a) Concentrations for radionuclides are in units of µCi/g; all other components are in units of µg/g. Values in parentheses are within 10 times the analytical detection limit and have relative uncertainties > 15%.
(b) Value adjusted for loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(c) The process blank had a relatively high Si content.

Table 3. C-106 Component Concentrations in Solution at 50°C.[a]

Analyte	Concentration at 50°C, Unadjusted					Concentration at 50°C, Adjusted[b]				
	C106-SOL-50-1	C106-SOL-50-2	Mean	Std. Dev.	% RSD	C106-SOL-50-1	C106-SOL-50-2	Mean	Std. Dev.	% RSD
Cesium-137	5.85	5.96	5.91	0.08	1.3	3.09	5.02	4.06	1.36	33.6
Strontium-90	0.211	0.109	0.160	0.072	45.1	0.111	0.092	0.102	0.014	13.7
Technetium-99	1.99E-03	1.66E-03	1.82E-03	2.36E-04	12.9	1.05E-03	1.39E-03	1.22E-03	2.43E-04	19.9
Americium-241	<9E-03	<7E-03	<9E-03	--	--	<5E-03	<6E-03	<5E-03	--	--
Europium-154	<9E-04	<7E-04	<9E-03	--	--	<5E-04	<6E-04	<5E-04	--	--
Europium-155	<9E-03	<7E-03	<9E-03	--	--	<5E-03	<6E-03	<5E-03	--	--
Total Alpha	4.55E-03	3.71E-03	4.13E-03	5.94E-04	14.4	2.40E-03	3.12E-03	2.76E-03	5.09E-04	18.4
Ag	(4.1)	(2.7)	(3.4)	(1.0)	29.1	(2.2)	(2.3)	(2.2)	(0.1)	3.4
Al	442	66.2	254	266	104.6	234	55.8	145	126	86.9
Ba	(0.68)	(0.35)	(0.52)	0.23	45.3	(0.36)	(0.29)	(0.33)	0.05	14.0
Ca	<10	(4.9)	<10	--	--	<5	(4.1)	<5	--	--
Cd	<0.6	<0.3	<0.6	--	--	<0.3	<0.3	<0.3	--	--
Co	<1.0	<0.5	<1.0	--	--	<0.5	<0.4	<0.5	--	--
Cr	(4.8)	(5.0)	(4.9)	0.2	3.2	(2.5)	(4.2)	(3.4)	1.2	35.4
Cu	(2.1)	(1.9)	(2.0)	(0.1)	7.1	(1.1)	(1.6)	(1.4)	(0.3)	25.6
Fe	(1.2)	<0.5	<2.0	--	--	(0.6)	<0.4	<0.5	--	--
K	<75	<38	<75	--	--	<40	<32	<40	--	--
La	<1.0	<0.5	<1.0	--	--	<0.5	<0.4	<0.5	--	--
Mg	<4	<2	<4	--	--	<2	<2	<2	--	--
Mn	<0.2	<0.1	<0.2	--	--	<0.1	<0.1	<0.1	--	--
Mo	<1.2	(1.1)	<1.2	--	--	<0.6	(0.9)	<1	--	--
Na	25500	23400	24450	1485	6.1	13475	19709	16592	4408	26.6
Ni	(2.6)	(3.3)	(3.0)	0.5	16.8	(1.4)	(2.8)	(2.1)	1.0	47.9
P	73.8	63.7	68.8	7	10.4	39.0	53.7	46.3	10	22.4
Pb	<2.3	<1.2	<2.3	--	--	<1.2	<1.0	<1.2	--	--
Si[c]	47.6	46.1	46.9	1	2.3	25.2	38.8	32.0	10	30.2
Ti	(0.39)	(0.21)	(0.3)	(0.1)	42.4	(0.21)	(0.18)	(0.2)	(0.0)	10.8
U	32.7	30.8	31.8	1.3	4.2	17.3	25.9	21.6	6.1	28.3
Zn	<0.8	(0.42)	<0.8	--	--	<0.4	(0.35)	<0.4	--	--
Zr	<1.0	<0.5	<1.0	--	--	<0.5	<0.4	<0.5	--	--
TOC	8231	7376	7803	605	7.7	4349	6212	5281	1317	24.9
TIC	2174	2058	2116	82	3.9	1149	1733	1441	414	28.7
Cl^-	470	510	490	28	5.8	248	430	339	128	37.8
F^-	<250	<250	<250	--	--	<132	<211	<211	--	--
NO_3^-	<500	<500	<500	--	--	<264	<421	<421	--	--
SO_4^{2-}	<500	<500	<500	--	--	<264	<421	<421	--	--
PO_4^{3-}	<500.	<500	<500	--	--	<264	<421	<421	--	--
$C_2O_4^{2-}$	25900	28000	26950	1485	5.5	13686	23583	18634	6998	37.6

(a) Concentrations for radionuclides are in units of µCi/g; all other components are in units of µg/g. Values in parentheses are within 10 times the analytical detection limit and have relative uncertainties > 15%.
(b) Value adjusted for loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(c) The process blank had a relatively high Si content.

Table 4. Concentration Changes Relative to 30°C Using the Unadjusted Values[a]

Analyte	Change, %[b]		Pooled	SD of % Change[c]		%Change / SD of % Change[c]	
	40°C	50°C	%RSD[f]	40°C	50°C	40°C	50°C
Cesium-137	**11**	**29**	3.7	5.8	6.8	**1.83**	**4.22**
Strontium-90	54	102	33.7	73.1	96.3	0.73	1.06
Technetium-99	-4.1	8.6	15.6	21.1	23.9	-0.19	0.36
Americium-241	(d)	(d)	--	--	--	--	--
Europium-154	(d)	(d)	--	--	--	--	--
Europium-155	(d)	(d)	--	--	--	--	--
Total Alpha	27	131	30.0	53.9	98.0	0.50	1.33
Ag	(51)	(145)	35.6	76.1	123.2	0.67	1.17
Al	-7.4	17	85.1	111.5	141.3	-0.07	0.12
Ba	-(2)	(91)	26.2	36.4	70.7	-0.05	1.28
Ca	(d)	(d)	--	--	--	--	--
Cd	(d)	(d)	--	--	--	--	--
Co	(d)	(d)	--	--	--	--	--
Cr	**(82)**	**(189)**	8.1	20.9	33.1	**3.94**	**5.7**
Cu	(32)	**(140)**	11.9	22.1	40.2	1.44	**3.47**
Fe	(d)	(d)	--	--	--	--	--
K	(d)	(d)	--	--	--	--	--
La	(d)	(d)	--	--	--	--	--
Mg	(d)	(d)	--	--	--	--	--
Mn	(d)	(d)	--	--	--	--	--
Mo	(d)	(d)	--	--	--	--	--
Na	2.3	**42**	5.1	7.3	10.1	0.32	**4.12**
Ni	(44)	**(136)**	13.7	27.8	45.6	1.58	**2.98**
P	5.0	**60**	6.3	9.3	14.2	0.54	**4.21**
Pb	(d)	(d)	--	--	--	--	--
Si[e]	5.1	**61**	3.8	5.7	8.7	0.90	**7.0**
Ti	-(3)	(100)	25.9	35.4	73.3	-0.09	1.36
U	9.3	**26**	8.2	12.6	14.6	0.74	**1.78**
Zn	(d)	(d)	--	--	--	--	--
Zr	(d)	(d)	--	--	--	--	--
TOC	2.3	14	5.5	8.0	8.9	0.28	1.59
TIC	-1.1	**16**	5.8	8.1	9.5	-0.14	**1.72**
Cl^-	8.2	**15**	4.7	7.3	7.7	1.13	**1.97**
F^-	(d)	(d)	--	--	--	--	--
NO_3^-	(d)	(d)	--	--	--	--	--
SO_4^{2-}	(d)	(d)	--	--	--	--	--
PO_4^{3-}	(d)	(d)	--	--	--	--	--
$C_2O_4^{2-}$	**10**	**22**	4.0	6.2	6.9	**1.64**	**3.22**

(a) Values in parentheses are for analytes that were within 10 times the analytical detection limit.
(b) The percent change is given by: %Change = $100*(C_T - C_{30})/C_{30}$, where C_T is the average concentration at temperature T (40 or 50°C) and C_{30} is the average concentration at 30°C.
(c) Pooled %RSD is the pooled percent relative standard deviation, obtained as the root mean square of the %RSD values at 30°C, 40°C, and 50°C. SD of % Change is the standard deviation of the % Change values at 40°C and 50°C, both relative to 30°C. Using propagation of error techniques it is computed as C_T/C_{30}*Sqrt(2)*Pooled %RSD. % Change/SD of % Change is the number of standard deviations the % Change value is from zero. Assuming a statistical t-distribution with 3 degrees of freedom, % Change/SD of % Change values must be larger than 1.64 to be significant at the 90% (one-sided) confidence level. Such values, and their corresponding % Change values, are shown in boldface.
(d) Analyte not detected.
(e) The values for Si should be veiwed with caution because of the high process blank.

Table 5. Concentration Changes Relative to 30°C Using the Adjusted Values[a]

Analyte	Change, %[b] 40°C	Change, %[b] 50°C	Pooled %RSD[f]	SD of % Change[c] 40°C	SD of % Change[c] 50°C	%Change / SD of % Change[c] 40°C	%Change / SD of % Change[c] 50°C
Cesium-137	16	30	9.4	15.5	17.2	1.06	1.72
Strontium-90	58	36	31.3	70.2	60.1	0.83	0.59
Technetium-99	-0.6	-2.6	24.5	34.4	33.7	-0.02	-0.08
Americium-241	(d)	(d)	--	--	--	--	--
Europium-154	(d)	(d)	--	--	--	--	--
Europium-155	(d)	(d)	--	--	--	--	--
Total Alpha	31	104	39.1	72.2	112.8	0.42	0.92
Ag	(54)	(90)	43.5	94.9	116.6	0.57	0.77
Al	-6.1	-70	76.1	101.1	32.1	-0.06	-2.19
Ba	(3)	(30)	8.6	12.5	15.7	0.25	1.88
Ca	(d)	(d)	--	--	--	--	--
Cd	(d)	(d)	--	--	--	--	--
Co	(d)	(d)	--	--	--	--	--
Cr	(91)	(195)	11.4	30.9	47.7	2.95	4.09
Cu	(37)	(126)	19.5	37.8	62.2	0.98	2.02
Fe	(d)	(d)	--	--	--	--	--
K	(d)	(d)	--	--	--	--	--
La	(d)	(d)	--	--	--	--	--
Mg	(d)	(d)	--	--	--	--	--
Mn	(d)	(d)	--	--	--	--	--
Mo	(d)	(d)	--	--	--	--	--
Na	7.2	35	11.9	18.1	22.9	0.40	1.54
Ni	(51)	(163)	15.3	32.6	57.0	1.55	2.86
P	10.1	48	9.9	15.3	20.6	0.66	2.32
Pb	(d)	(d)	--	--	--	--	--
Si[e]	10.5	58	9.7	15.1	21.6	0.69	2.68
Ti	(2)	(40)	14.2	20.4	28.0	0.08	1.42
U	15.6	23	7.8	12.8	13.6	1.22	1.68
Zn	(d)	(d)	--	--	--	--	--
Zr	(d)	(d)	--	--	--	--	--
TOC	7.7	8	6.3	9.6	9.7	0.80	0.84
TIC	4.0	13	10.2	15.1	16.4	0.27	0.81
Cl^-	14	20	5.7	9.1	9.6	1.53	2.10
F^-	(d)	(d)	--	--	--	--	--
NO_3^-	(d)	(d)	--	--	--	--	--
SO_4^{2-}	(d)	(d)	--	--	--	--	--
PO_4^{3-}	(d)	(d)	--	--	--	--	--
$C_2O_4^{2-}$	16	27	5.2	8.5	9.3	1.89	2.93

(a) Values in parentheses are for analytes that were within 10 times the analytical detection limit.

(b) The percent change is given by: %Change = $100*(C_T - C_{30})/C_{30}$, where C_T is the average concentration at 40°C or the single measure at 50°C) and C_{30} is the average concentration at 30°C.

(c) Pooled %RSD is the pooled percent relative standard deviation, obtained as the root mean square of the %RSD values at 30°C and 40°C. SD of % Change is the standard deviation of the % Change values at 40°C and 50°C, both relative to 30°C. Using propagation of error techniques it is computed as C_T/C_{30}*Sqrt(2)*Pooled %RSD. % Change/SD of % Change is the number of standard deviations the % Change value is from zero. Assuming a statistical t-distribution with 2 degrees of freedom, % Change/SD of % Change values must be larger than 1.89 to be significant at the 90% (one-sided) confidence level. Such values, and their corresponding % Change values, are shown in boldface.

(d) Analyte not detected.

(e) The values for Si should be veiwed with caution because of the high process blank.

Table 6. Dilute Hydroxide Washing of C-106 Sludge: Analysis of the Composite Wash Solution[a]

Analyte	Direct	Adjusted[b]	Amount (µCi or µg) in Wash Solutions
Ag	(1.3)	(1.2)	393
Al	75.9	71.1	22937
Ba	(0.16)	(0.15)	48
Ca	(5.0)	(4.7)	1511
Cd	<0.2	<0.2	60
Co	<0.3	<0.3	91
Cr	(0.78)	(0.73)	236
Cu	(1.2)	(1.1)	363
Fe	(1.2)	(1.1)	363
Hg	Not Measured	--	--
K	<20	<19	6044
La	<0.3	<0.3	91
Mg	(1.9)	(1.8)	574
Mn	<0.1	<0.1	30
Mo	<0.3	<0.3	91
Na	16500	15453	4986241
Ni	(2.3)	(2.2)	695
P	17.8	16.7	5379
Pb	<0.6	<0.6	181
Si[c]	25.6	24.0	7736
Ti	(0.1)	(0.1)	25
U	9.39	8.8	2838
Zn	<0.2	<0.2	60
Zr	<0.3	<0.3	91
TOC	Not Measured	--	--
TIC	Not Measured	--	--
Cl^-	Not Measured	--	--
F^-	Not Measured	--	--
NO_3^-	Not Measured	--	--
SO_4^{2-}	Not Measured	--	--
PO_4^{3-}	Not Measured	--	--
$C_2O_4^{2-}$	Not Measured	--	--
CN^-	Not Measured	--	--
NH_3	Not Measured	--	--

Table 6. Contd.

Analyte	Direct	Adjusted[b]	Amount (μCi or μg) in Wash Solutions
^{137}Cs	3.41	3.19E+00	1.03E+03
^{90}Sr	Not Measured	--	--
^{99}Tc	4.40E-04	4.12E-04	1.33E-01
$^{241}Am(\gamma)$	< 4E-03	< 4E-03	< 1E+00
$^{241}Am(\alpha)$	Not Measured	--	--
^{154}Eu	1.55E-03	1.45E-03	4.68E-01
^{155}Eu	< 4E-03	< 4E-03	< 1E+00
^{14}C[c]	Not Measured	--	--
^{129}I	Not Measured	--	--
^{235}U	Not Measured	--	--
^{238}U	Not Measured	--	--
^{237}Np	Not Measured	--	--
^{238}Pu	Not Measured	--	--
^{239}Pu	Not Measured	--	--
^{240}Pu	Not Measured	--	--
$^{239+240}Pu$	Not Measured	--	--
$^{243+244}Cm$	Not Measured	--	--
^{242}Cm	Not Measured	--	--
Total Alpha	9.81E-04	9.19E-04	2.96E-01

(a) Concentrations for radionuclides are in units of μCi/g; all other components are in units of μg/g. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%.
(b) Value adjusted for the 6.3% loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(c) The process blank had a Si content of 32 μg/g, so the Si data for the sample is not reliable.

Table 7. Dilute Hydroxide Washing of C-106 Sludge: Analysis of the Non-Magnetic Fraction of the Washed Solids[a]

Analyte	KOH Fusion					Na_2O_2 Fusion					Amount (µCi or µg) in C106-AQ-8[b]
	C106-AQ-8	C106-AQ-8DUP	Mean	Std Dev.	% RSD	C106-AQ-8	C106-AQ-8DUP	Mean	Std Dev.	% RSD	
Ag	289	285	287	3	1	591	391	491	141	29	582
Al	70500	64200	67350	4455	7	62800	54200	58500	6081	10	136626
Ba	548	496	522	37	7	432	387	410	32	8	1059
Ca[c]	13900	15800	14850	1344	9	16400	12900	14650	2475	17	30125
Cd	(87)	(88)	(88)	1	1	(58)	(40)	(49)	13	26	(178)
Co	(68)	(68)	(68)	0	0	< 50	< 50	< 50	--	--	(138)
Cr	1200	1210	1205	7	1	982	948	965	24	2	2444
Cu	524	513	519	8	2	415	390	403	18	4	1052
Fe	217000	214000	215500	2121	1	181000	157000	169000	16971	10	437163
Hg	369	332	351	26	7	--	--	--	--	--	711
K[d]	--	--	--	--	--	(3800)	(5300)	(4550)	1061	23	(9230)
La	(150)	(160)	(155)	7	5	(120)	(120)	(120)	0	0	(314)
Mg	2840	3180	3010	240	8	4240	2710	3475	1082	31	6106
Mn	5060	5110	5085	35	1	3940	3810	3875	92	2	10315
Mo	< 60	< 60	< 60	--	--	< 60	< 60	< 60	--	--	< 122
Na[e]	57400	52100	54750	3748	7	--	--	--	--	--	111066
Ni	--	--	--	--	--	1260	1050	1155	148	13	2343
P	3820	4960	4390	806	18	(890)	(1400)	1145	361	31	8906
Pb	3270	3260	3265	7	0	3200	2750	2975	318	11	6623
Si	126000	93700	109850	22840	21	108000	104000	106000	2828	3	222842
Ti	2740	2760	2750	14	1	2670	2500	2585	120	5	5579
U	178	173	176	4	2	--	--	--	--	--	356
Zn	(280)	(310)	(295)	21	7	(240)	(250)	245	7	3	(598)
Zr	3420	3320	3370	71	2	--	--	--	--	--	6836
TOC	31300	28300	29800	2121	7	--	--	--	--	--	60452
TIC	6410	6470	6440	42	1	--	--	--	--	--	13064
Cl^-	12000	5800	8900	4384	49	--	--	--	--	--	18055
F^-	300	360	330	42	13	--	--	--	--	--	669
NO_3^-	3000	1300	2150	1202	56	--	--	--	--	--	4361
SO_4^{2-}	< 400	< 400	< 400	--	--	--	--	--	--	--	811
PO_4^{3-}	< 400	< 400	< 400	--	--	--	--	--	--	--	811
$C_2O_4^{2-}$	20300	9700	15000	7495	50	--	--	--	--	--	30429
CN^-	10.4	18.5	14.5	5.7	40	--	--	--	--	--	29
NH_3	3.6	4.3	4.0	0.5	13	--	--	--	--	--	8

Table 7. Contd.

Analyte	KOH Fusion C106-AQ-8	KOH Fusion C106-AQ-8DUP	KOH Fusion Mean	KOH Fusion Std Dev.	KOH Fusion % RSD	Na_2O_2 Fusion C106-AQ-8	Na_2O_2 Fusion C106-AQ-8DUP	Na_2O_2 Fusion Mean	Na_2O_2 Fusion Std Dev.	Na_2O_2 Fusion % RSD	Amount (µCi or µg) in C106-AQ-8
^{137}Cs	401	3.54E+02	3.78E+02	3.32E+01	9	--	--	--	--	--	766
^{90}Sr	935	9.16E+02	9.25E+02	1.31E+01	1	--	--	--	--	--	1877
^{99}Tc	1.70E-02	2.04E-02	1.87E-02	2.40E-03	13	--	--	--	--	--	3.79E-02
^{241}Am(γ)	2.74	2.60E+00	2.67E+00	9.90E-02	4	--	--	--	--	--	5.42
^{241}Am(α)	2.42	2.22E+00	2.32E+00	1.41E-01	6	--	--	--	--	--	4.71
^{154}Eu	3.67	3.39E+00	3.53E+00	1.98E-01	6	--	--	--	--	--	7.16
^{155}Eu	2.41	2.33E+00	2.37E+00	5.66E-02	2	--	--	--	--	--	4.81
^{14}C$^{(f)}$	7.73E-03	--	7.73E-03	--	--	--	--	--	--	--	1.57E-02
^{129}I	4.43E-03	3.35E-03	3.89E-03	7.64E-04	20	--	--	--	--	--	7.89E-03
^{235}U	4.18E-06	5.28E-06	4.73E-06	7.78E-07	16	--	--	--	--	--	9.60E-06
^{238}U	9.72E-05	8.77E-05	9.25E-05	6.72E-06	7	--	--	--	--	--	1.88E-04
^{237}Np	1.70E-03	1.81E-03	1.76E-03	7.78E-05	4	--	--	--	--	--	3.56E-03
^{238}Pu	5.19E-01	4.96E-01	5.08E-01	1.63E-02	3	--	--	--	--	--	1.03E+00
^{239}Pu	3.11E+00	2.87E+00	2.99E+00	1.70E-01	6	--	--	--	--	--	6.07E+00
^{240}Pu	7.59E-01	8.74E-01	8.17E-01	8.13E-02	10	--	--	--	--	--	1.66E+00
$^{239+240}$Pu	2.84E+00	2.72E+00	2.78E+00	8.49E-02	3	--	--	--	--	--	5.64E+00
$^{243+244}$Cm	5.18E-02	6.15E-02	5.67E-02	6.86E-03	12	--	--	--	--	--	1.15E-01
^{242}Cm	5.83E-03	3.34E-03	4.59E-03	1.76E-03	38	--	--	--	--	--	9.30E-03
Total Alpha	5.84	5.50E+00	5.67E+00	2.40E-01	4	--	--	--	--	--	11.50

(a) Concentrations for radionuclides are in units of µCi/g dry solids; all other components are in units of µg/g dry solids. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%. TIC/TOC and cyanide analyses was performed directly on the washed solids. Anion (IC) analysis was done on a water leachate of the washed solids, so this does not accurately represent the anions present in the solids.

(b) Because the values obtained from the Na_2O_2 fusion method are generally lower than those obtained by the KOH fusion method, the mean values from the KOH fusions were used to determine the amount of each component in the washed solids (see discussion in the text). Nickel and K are exceptions, because these were only available from the Na_2O_2 fusion.

(c) The process blank for the Na_2O_2 fusion had a Ca content of ~2,500 µg/g.

(d) The process blank for the Na_2O_2 fusion had a K content of ~4,100 µg/g, so the reported K concentration values in the sample can be largely attributed to the sample preparation.

(e) The process blank for the KOH fusion had a Na content of ~2,500 µg/g.

(f) Sample recoveries for the ^{14}C analysis were low and not reproducible; thus, ^{14}C data are suspect.

Table 8. Dilute Hydroxide Washing of C-106 Sludge: Analysis of the Magnetic Fraction of the Washed Solids[a]

Analyte	C106-AQ-8B1 KOH Fusion	C106-AQ-8B1 Na_2O_2 Fusion	Mean	Std Dev.	% RSD	Amount (µCi or µg) in C106-AQ-8B1[b]
Ag	496	338	417	112	27	229
Al	7770	6950	7360	580	8	3580
Ba	(110)	(100)	(105)	(7)	7	(51)
Ca[c]	4640	6950	5795	1633	28	2138
Cd	(29)	<35	<32	--	--	(13)
Co	(80)	(65)	(73)	(11)	15	(37)
Cr	384	(370)	(377)	(10)	3	177
Cu	(190)	(200)	(195)	(7)	4	(88)
Fe	508000	446000	477000	43841	9	234086
Hg	Not Measured	--	--	--	--	--
K	--	<4400	<4400	--	--	<2028
La	(45)	<55	<50	<7	14	(21)
Mg	(1700)	(1900)	(1800)	(141)	8	(783)
Mn	3320	2950	3135	262	8	1530
Mo	(56)	<70	<63	--	--	(26)
Na[d]	7880	--	7880	--	--	3631
Ni	--	(620)	(620)	--	--	286
P	(1600)	(980)	(1290)	(438)	34	(737)
Pb	(1000)	(1200)	(1100)	(141)	13	(461)
Si	12800	9030	10915	2666	24	5898
Ti	1430	1300	1365	92	7	659
U	221	--	221	--	--	102
Zn	(240)	(250)	(245)	(7)	3	(111)
Zr	(360)	--	(360)	--	--	(166)
TOC	Not Measured	--	--	--	--	--
TIC	Not Measured	--	--	--	--	--
Cl^-	Not Measured	--	--	--	--	--
F^-	Not Measured	--	--	--	--	--
NO_3^-	Not Measured	--	--	--	--	--
SO_4^{2-}	Not Measured	--	--	--	--	--
PO_4^{3-}	Not Measured	--	--	--	--	--
$C_2O_4^{2-}$	Not Measured	--	--	--	--	--
CN^-	Not Measured	--	--	--	--	--
NH_3	Not Measured	--	--	--	--	--

Table 8. Contd.

Analyte	C106-AQ-8B1 KOH Fusion	Na$_2$O$_2$ Fusion	Mean	Std Dev.	Rel% Error	Amount (μCi or μg) in C106-AQ-8B1
^{137}Cs	8.08E+01	--	8.08E+01	--	--	3.72E+01
^{90}Sr	1.23E+02	--	1.23E+02	--	--	5.67E+01
^{99}Tc	< 9E-03	--	< 9E-03	--	--	< 4E-03
^{241}Am(γ)	4.35E-01	--	4.35E-01	--	--	2.00E-01
^{241}Am(α)	4.10E-01	--	4.10E-01			
^{154}Eu	5.80E-01	--	5.80E-01	--	--	2.67E-01
^{155}Eu	5.40E-01	--	5.40E-01	--	--	2.49E-01
^{14}C$^{(e)}$	Not Measured	--	--	--	--	--
^{129}I	7.74E-04	< 2E-04	7.74E-04	3.82E-04	49	3.57E-04
^{235}U	2.64E-05	3.06E-05	2.85E-05	2.97E-06	10	1.31E-05
^{238}U	1.28E-04	1.37E-04	1.33E-04	6.36E-06	5	6.11E-05
^{237}Np	< 7E-04	< 7E-04	< 7E-04	< 0E+00	0	< 3E-04
^{238}Pu	2.00E-01	--	2.00E-01	--	--	9.22E-02
^{239}Pu	8.80E-01	8.12E-01	8.46E-01	4.81E-02	6	3.90E-01
^{240}Pu	3.68E-01	3.45E-01	3.57E-01	1.63E-02	5	1.64E-01
$^{239+240}$Pu	7.43E-01	--	7.43E-01	--	--	3.42E-01
$^{243+244}$Cm	3.16E-02	--	3.16E-02	--	--	1.46E-02
^{242}Cm	2.49E-03	--	2.49E-03	--	--	1.15E-03
Total Alpha	1.39E+00	--	1.39E+00	--	--	0.64

(a) Concentrations for radionuclides are in units of μCi/g dry solids; all other components are in units of μg/g dry solids. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%. Anion (IC) analysis was done performed on this sample.

(b) Because the values obtained from the Na$_2$O$_2$ fusion method are generally lower than those obtained by the KOH fusion method, the mean values from the KOH fusions were used to determine the amount of each component in the washed solids (see discussion in the text). Nickel and K are exceptions, because these were only available from the Na$_2$O$_2$ fusion.

(c) The process blank for the Na$_2$O$_2$ fusion had a Ca content of ~2,500 μg/g.

(d) The process blank for the KOH fusion had a Na content of ~2,500 μg/g.

(e) Sample recoveries for the ^{14}C analysis were low and not reproducible; thus, ^{14}C data are suspect.

Table 9. Concentrations in The Washed and Untreated Solids and the Relative Amount of Each Component Removed by Dilute Hydroxide Washing

	µg/g dry solids or µCi/g dry solids					
Analyte	Washed Solids[a]	Pseudo 95% C.I (if %RSDs=10)[b]	Original Sample[c]	Pseudo 95% C.I (if %RSDs=10)[b]	Removed,%[d]	Pseudo 95% C.I (if %RSDs=10)[b]
Ag	381	44r	51	5.2r	33	7.3r
Al	65825	9078r	6949	846r	14	3.3r
Ba	521	70r	49	6.4r	4	1.0r
Ca	15147	2010r	1439	183r	4	1.1r
Cd	90	12r	11	1.2r	24	5.5r
Co	82	10r	11	1.2r	34	7.7r
Cr	1231	163r	122	15r	8	1.9r
Cu	535	70r	64	7.1r	24	5.5r
Fe	315141	36409r	28606	3303r	0	0.0r
Hg[f]	334	47r	30	4r	--	--
K	5285	642r	737	78r	35	7.9r
La	157	21r	18	2.1r	21	4.9r
Mg	3234	412r	318	38r	8	1.8r
Mn	5561	700r	506	63r	0	0.1r
Mo	69	8r	10	1.1r	38	8.6r
Na[e]	53848	7382r	217267	42482r	98	27r
Ni	1234	158r	142	15r	21	4.8r
P	4527	595r	640	71r	36	8.2r
Pb	3326	442r	309	40r	2	0.6r
Si	107390	14806r	10072	1345r	3	0.8r
Ti	2928	376r	267	34r	0	0.1r
U	215	25r	140	24r	86	23r
Zn	333	41r	33	3.8r	8	1.8r
Zr	3287	454r	302	41r	1	0.3r
TOC[f]	28381	4014r	2575	364r	--	--
TIC[f]	6133	867r	556	79r	--	--
Cl^{-}[f]	8476	1199r	769	109r	--	--
F^{-}[f]	314	44r	29	4.0r	--	--
NO_3^{-}[f]	2048	290r	186	26r	--	--
SO_4^{2-}[f]	381	54r	35	4.9r	--	--
PO_4^{3-}[f]	381	54r	35	4.9r	--	--
$C_2O_4^{2-}$[f]	14286	2020r	1296	183r	--	--
CN^{-}[f]	14	2r	1.2	0.2r	--	--
NH_3[f]	4	1r	0.3	0.05r	--	--

Table 9. Contd.

Analyte	Washed Solids[a]	Pseudo 95% C.I. (if %RSDs=10)[b]	Original Sample[c]	Pseudo 95% C.I. (if %RSDs=10)[b]	Removed,%[d]	Pseudo 95% C.I. (if %RSDs=10)[b]
	µg/g dry solids or µCi/g dry solids					
^{137}Cs	3.77E+02	5.10E+01r	7.81E+01	9.92E+00r	56	13r
^{90}Sr[g]	9.08E+02	1.25E+02r	8.24E+01	1.13E+01r	--	--
^{99}Tc	1.96E-02	2.55E-03r	7.45E-03	1.16E-03r	76	19r
$^{241}Am(\gamma)$	2.64E+00	3.60E-01r	2.88E-01	3.41E-02r	17	4.0r
$^{241}Am(\alpha)$[g]	2.21E+00	3.12E-01r	2.00E-01	2.83E-02r	--	--
^{154}Eu	3.49E+00	4.76E-01r	3.36E-01	4.34E-02r	6	1.4r
^{155}Eu	2.37E+00	3.20E-01r	2.67E-01	3.08E-02r	19	4.5r
^{14}C[f]	7.36E-03	1.47E-03r	6.68E-04	1.34E-04r	--	--
^{129}I[g]	3.87E-03	5.25E-04r	3.51E-04	4.76E-05r	--	--
^{235}U[g]	1.07E-05	1.39E-06r	9.68E-07	1.26E-07r	--	--
^{238}U[g]	1.17E-04	1.37E-05r	1.06E-05	1.24E-06r	--	--
^{237}Np[g]	1.83E-03	2.38E-04r	1.66E-04	2.16E-05r	--	--
^{238}Pu[g]	5.27E-01	6.89E-02r	4.78E-02	6.25E-03r	--	--
^{239}Pu[g]	3.03E+00	4.04E-01r	2.75E-01	3.67E-02r	--	--
^{240}Pu[g]	8.55E-01	1.11E-01r	7.75E-02	1.01E-02r	--	--
$^{239+240}Pu$[g]	2.81E+00	3.76E-01r	2.55E-01	3.41E-02r	--	--
$^{243+244}Cm$[g]	6.08E-02	7.75E-03r	5.52E-03	7.03E-04r	--	--
^{242}Cm[g]	4.91E-03	6.27E-04r	4.45E-04	5.69E-05r	--	--
Total Alpha	5.70E+00	7.66E-01r	5.30E-01	6.95E-02r	2	0.6r

(a) The concentration in the washed solids was determined by summing the quantity found in the non-magnetic and the magnetic fractions and dividing by the total weight (2.13 g) of the washed solids.
(b) Pseudo 95% Confidence Intervals (C.I.) were approximated using propagation of error techniques for the case where the %RSD of all analytical measures used is 10% and all measures are independent The reader can review other potential %RSD values by multiplying the cell value by r, where r is %RSD/10.
(c) The concentration in the untreated solids was determined by summing the quantity found in the wash solution, the non-magnetic and the magnetic solid fractions and dividing by the total weight (23.4777 g) of sample used.
(d) The percent removed was determined by the following formula: %Removed= $100*F_w/(F_w+F_{nm}+F_m)$; where F_w is the fraction in the wash solution, F_{nm} is the fraction in the non-magnetic solids, and F_m is the fraction in the magnetic solids.
(e) The values for Na are not corrected for Na added as NaOH during the washing process.
(f) Only the material in the non-magnetic solids are accounted for in these values.
(g) In calculating the concentration in the untreated solids, only the material in the washed solids are accounted for in these values.

Table 10. Caustic Leaching of C-106 Sludge: Analysis of the Leaching Solution and the Composite Wash Solution[a]

Analyte	Leach Solution C106-OH-3			Composite Wash Solution C106-OH-9		
	Direct	Adjusted[b]	Amount (µCi or µg)	Direct	Adjusted[c]	Amount (µCi or µg)
Ag	(2.5)	(1.6)	(167)	(0.53)	(0.50)	(107)
Al	778	509	52936	66.6	62.4	13464
Ba	(0.1)	(0.1)	(8)	<0.2	<0.2	<40
Ca	(2.4)	(1.6)	(163)	<5	<5	<1011
Cd	<0.2	<0.1	<13.6	<0.3	<0.3	<61
Co	<0.3	<0.2	<20.4	<0.5	<0.5	<101
Cr	11.0	7.2	748	(0.96)	(0.90)	(194)
Cu	(2.6)	(1.7)	(174)	(0.90)	(0.84)	(182)
Fe	9.81	6	667	(1.7)	(1.6)	(344)
Hg	Not Measured	--	--	Not Measured	--	--
K	(66)	(43)	(4491)	<40	<37	<8086
La	<0.3	<0.2	<20.4	<0.5	<0.5	<101
Mg	<1	<1	<68	<2	<2	<404
Mn	(0.09)	(0.06)	(6)	<0.1	<0.1	<20
Mo	(0.87)	(0.57)	(59)	<0.6	<0.6	<121
Na	62400	40796	4245746	15900	14904	3214381
Ni	(0.38)	(0.25)	(26)	<0.6	<0.6	<121
P	78.1	51	5314	(14)	(13)	(2830)
Pb	16.9	11.0	1146	<2	<2	<404
Si[d]	350	228	23780	49.9	46.8	10088
Ti	(0.13)	(0.08)	(9)	<0.1	<0.1	<20
U	35.3	23.1	2402	0.534	0.501	108
Zn	(2.3)	(1.5)	(159)	<0.4	<0.4	<81
Zr	(0.6)	(0.4)	(38)	<0.5	<0.5	<101
TOC	Not Measured	--	--	Not Measured	--	--
TIC	Not Measured	--	--	Not Measured	--	--
Cl^-	Not Measured	--	--	Not Measured	--	--
F^-	Not Measured	--	--	Not Measured	--	--
NO_3^-	Not Measured	--	--	Not Measured	--	--
SO_4^{2-}	Not Measured	--	--	Not Measured	--	--
PO_4^{3-}	Not Measured	--	--	Not Measured	--	--
$C_2O_4^{2-}$	Not Measured	--	--	Not Measured	--	--
CN^-	Not Measured	--	--	Not Measured	--	--
NH_3	Not Measured	--	--	Not Measured	--	--

Table 10. Contd.

Analyte	Leach Solution C106-OH-3 Direct	Leach Solution C106-OH-3 Adjusted[b]	Leach Solution C106-OH-3 Amount (µCi or µg)	Composite Wash Solution C106-OH-9 Direct	Composite Wash Solution C106-OH-9 Adjusted[c]	Composite Wash Solution C106-OH-9 Amount (µCi or µg)
^{137}Cs	1.78E+01	1.16E+01	1.21E+03	1.81E+00	1.70E+00	3.66E+02
^{90}Sr	Not Measured	--	--	Not Measured	--	--
^{99}Tc	1.84E-03	1.20E-03	1.25E-01	9.01E-05	8.45E-05	1.82E-02
$^{241}Am(\gamma)$	< 8E-03	< 5E-03	< 5E-01	< 3E-03	< 3E-03	< 6E-01
$^{241}Am(\alpha)$	Not Measured	--	--	Not Measured	--	--
^{154}Eu	7.00E-04	4.58E-04	4.76E-02	2.00E-04	1.87E-04	4.04E-02
^{155}Eu	8.00E-03	5.23E-03	5.44E-01	3.00E-03	2.81E-03	6.06E-01
^{14}C[e]	Not Measured	--	--	Not Measured	--	--
^{129}I	Not Measured	--	--	Not Measured	--	--
^{235}U	Not Measured	--	--	Not Measured	--	--
^{238}U	Not Measured	--	--	Not Measured	--	--
^{237}Np	Not Measured	--	--	Not Measured	--	--
^{238}Pu	Not Measured	--	--	Not Measured	--	--
^{239}Pu	Not Measured	--	--	Not Measured	--	--
^{240}Pu	Not Measured	--	--	Not Measured	--	--
$^{239+240}Pu$	Not Measured	--	--	Not Measured	--	--
$^{243+244}Cm$	Not Measured	--	--	Not Measured	--	--
^{242}Cm	Not Measured	--	--	Not Measured	--	--
Total Alpha	7.82E-03	5.11E-03	5.32E-01	1.29E-03	1.21E-03	2.61E-01

(a) Concentrations for radionuclides are in units of µCi/g; all other components are in units of µg/g. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%.
(b) Value adjusted for the 34.6% loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(c) Value adjusted for the 6.3% loss in sample weight that occurred before analysis; this weight loss was assumed to be due to evaporation.
(d) The process blank had a Si content of ~60 µg/g.

Table 11. Caustic Leaching of C-106 Sludge: Analysis of the Non-Magnetic Fraction of the Leached Solids[a]

	KOH Fusion					Na_2O_2 Fusion					Amount (μCi or μg)
Analyte	C106-OH-8	C106-OH-8DUP	Mean	Std Dev.	% RSD	C106-OH-8	C106-OH-8DUP	Mean	Std Dev.	% RSD	in C106-OH-8[b]
Ag	285	327	306	30	10	639	1460	1050	581	55	1991
Al	38600	31600	35100	4950	14	29500	28300	28900	849	3	228382
Ba	384	324	354	42	12	290	263	277	19	7	2303
Ca[c]	16200	8920	12560	5148	41	11700	10400	11050	919	8	81723
Cd	(38)	(37)	(38)	1	2	(31)	(37)	(34)	4	12	(244)
Co	< 45	< 50	< 48	--	--	< 50	< 51	< 51	--	--	< 309
Cr	(260)	(280)	(270)	14	5	(300)	(340)	(320)	28	9	(1757)
Cu	(130)	(140)	(135)	7	5	(140)	(250)	(195)	78	40	(878)
Fe	74200	85300	79750	7849	10	87300	99100	93200	8344	9	518901
Hg	153	121	137	23	17	--	--	--	--	--	891
K[d]	--	--	--	--	--	< 3850	< 4065	< 4065	--	--	< 26449
La	(74)	(94)	(84)	14	17	(71)	(72)	(72)	1	1	(547)
Mg	2490	2930	2710	311	11	2630	(1900)	(2265)	516	23	17633
Mn	1890	1850	1870	28	2	1830	2120	1975	205	10	12167
Mo	< 55	< 60	< 58	--	--	< 60	< 61	< 61	--	--	< 374
Na[e]	160000	183000	171500	16263	9	--	--	--	--	--	1115882
Ni	--	--	--	--	--	841	951	896	78	9	5830
P	(1300)	(1500)	(1400)	141	10	(680)	(970)	(825)	205	25	(9109)
Pb	1140	1650	1395	361	26	1550	1800	1675	177	11	9077
Si	134000	102000	118000	22627	19	68800	59400	64100	6647	10	767779
Ti	1810	2010	1910	141	7	2050	1960	2005	64	3	12428
U	144	208	176	45	26	--	--	--	--	--	1145
Zn	(110)	(120)	(115)	7	6	(120)	(160)	(140)	28	20	(748)
Zr	651	1780	1216	798	66	--	--	--	--	--	7909
TOC	150000	121000	135500	20506	15	--	--	--	--	--	881644
TIC	1400	< 1700	< 1700	--	--	--	--	--	--	--	< 11061
Cl^-	< 8000	< 8000	< 8000	--	--	--	--	--	--	--	< 52053
F^-	< 8000	< 8000	< 8000	--	--	--	--	--	--	--	< 52053
NO_3^-	< 15000	< 15000	< 15000	--	--	--	--	--	--	--	< 97599
SO_4^{2-}	< 15000	< 15000	< 15000	--	--	--	--	--	--	--	< 97599
PO_4^{3-}	< 15000	< 15000	< 15000	--	--	--	--	--	--	--	< 97599
$C_2O_4^{2-}$	355000	434000	394500	55861	14	--	--	--	--	--	2566854
CN^-	5	4	4.5	0.7	16	--	--	--	--	--	29
NH_3	9.5	13.2	11.4	2.6	23	--	--	--	--	--	74

Table 11. Contd.

Analyte	KOH Fusion C106-OH-8	KOH Fusion C106-OH-8DUP	KOH Fusion Mean	KOH Fusion Std Dev.	KOH Fusion % RSD	Na_2O_2 Fusion C106-OH-8	Na_2O_2 Fusion C106-OH-8DUP	Na_2O_2 Fusion Mean	Na_2O_2 Fusion Std Dev.	Na_2O_2 Fusion Rel% Error	Amount (μCi or μg) in C106-OH-8
^{137}Cs	9.90E+01	1.12E+02	1.06E+02	9	9	--	--	--	--	--	686
^{90}Sr	2.66E+02	3.20E+02	2.93E+02	38	13	--	--	--	--	--	1906
^{99}Tc	<9E-03	<9E-03	--	--	--	--	--	--	--	--	<6E-02
$^{241}Am(\gamma)$	9.76E-01	1.04E+00	1.01E+00	0.05	4	--	--	--	--	--	6.56
$^{241}Am(\alpha)$	8.34E-01	8.34E-01	8.34E-01	0.00	0	--	--	--	--	--	5.43
^{154}Eu	1.09E+00	1.38E+00	1.24E+00	0.21	17	--	--	--	--	--	8.04
^{155}Eu	6.48E-01	9.08E-01	7.78E-01	0.18	24	--	--	--	--	--	5.06
$^{14}C^{(f)}$	1.76E-03	5.95E-04	1.18E-03	--	--	--	--	--	--	--	7.66E-03
^{129}I	1.01E-03	1.44E-03	1.23E-03	3.04E-04	25	--	--	--	--	--	7.97E-03
^{235}U	5.08E-06	5.94E-06	5.51E-06	6.08E-07	11	--	--	--	--	--	3.59E-05
^{238}U	8.50E-05	1.09E-04	9.70E-05	1.70E-05	17	--	--	--	--	--	6.31E-04
^{237}Np	<7E-04	<2E-03	--	--	--	--	--	--	--	--	<1E-02
^{238}Pu	1.69E-01	1.88E-01	1.79E-01	1.34E-02	8	--	--	--	--	--	1.16E+00
^{239}Pu	1.54E+00	8.74E-01	1.21E+00	4.71E-01	39	--	--	--	--	--	7.85E+00
^{240}Pu	4.83E-01	2.07E-01	3.45E-01	1.95E-01	57	--	--	--	--	--	2.24E+00
$^{239+240}Pu$	1.50E+00	8.33E-01	1.17E+00	4.72E-01	40	--	--	--	--	--	7.59E+00
$^{243+244}Cm$	2.23E-02	3.08E-02	2.66E-02	6.01E-03	23	--	--	--	--	--	1.73E-01
^{242}Cm	1.63E-03	1.09E-03	1.36E-03	3.82E-04	28	--	--	--	--	--	8.85E-03
Total Alpha	2.23E+00	1.89E+00	2.06E+00	0.240	12	--	--	--	--	--	13.4

(a) Concentrations for radionuclides are in units of μCi/g dry solids; all other components are in units of μg/g dry solids. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%. TIC/TOC and cyanide analyses was performed directly on the washed solids. Anion (IC) analysis was done on a water leachate of the leached solids, so this does not accurately represent the anions present in the solids.

(b) Because the values obtained from the Na_2O_2 fusion method are generally lower than those obtained by the KOH fusion method, the mean values from the KOH fusions were used to determine the amount of each component in the washed solids (see discussion in the text). Nickel and K are exceptions, because these were only available from the Na_2O_2 fusion.

(c) The process blank for the Na_2O_2 fusion had a Ca content of ~2,500 μg/g.

(d) The process blank for the Na_2O_2 fusion had a K content of ~4,100 μg/g, so the reported K concentration values in the sample can be largely attributed to the sample preparation.

(e) The process blank for the KOH fusion had a Na content of ~2,500 μg/g.

(f) Sample recoveries for the ^{14}C analysis were low and not reproducible; thus, ^{14}C data are suspect.

Table 12. Caustic Leaching of C-106 Sludge: Analysis of the Magnetic Fraction of the Leached Solids[a]

Analyte	C106-OH-8B1 KOH Fusion	C106-OH-8B1 Na_2O_2 Fusion	Mean	Std Dev.	% RSD	Amount (µCi or µg) in C106-OH-8B1[b]
Ag	719	(290)	(505)	303	60	1462
Al	6210	6810	6510	424	7	12630
Ba	(110)	(150)	(130)	(28)	22	(224)
Ca[c]	2680	5160	3920	1754	45	5451
Cd	(33)	<30	(33)	--	--	(67)
Co	(90)	<50	<100	--	--	(183)
Cr	392	(380)	(386)	(8)	2	797
Cu	(200)	(170)	(185)	(21)	11	(407)
Fe	472000	402000	437000	49497	11	959954
Hg	Not Measured	--	--	--	--	--
K	--	<4000	<4000	--	--	<8135
La	(54)	<50	<60	--	--	(110)
Mg	(700)	(840)	(770)	(99)	13	(1424)
Mn	3110	2700	2905	290	10	6325
Mo	<60	<60	<60	--	--	<122
Na[d]	15000	--	15000	--	--	30507
Ni	--	719	719	--	--	1462
P	(1200)	(980)	(1090)	(156)	14	(2441)
Pb	(1470)	1420	(1445)	(35)	2	(2990)
Si	22200	33100	27650	7707	28	45150
Ti	1040	1100	1070	42	4	2115
U	210	--	210	--	--	427
Zn	(340)	(350)	(345)	(7)	2	(691)
Zr	(110)	--	(110)	--	--	(224)
TOC	Not Measured	--	--	--	--	--
TIC	Not Measured	--	--	--	--	--
Cl^-	Not Measured	--	--	--	--	--
F^-	Not Measured	--	--	--	--	--
NO_3^-	Not Measured	--	--	--	--	--
SO_4^{2-}	Not Measured	--	--	--	--	--
PO_4^{3-}	Not Measured	--	--	--	--	--
$C_2O_4^{2-}$	Not Measured	--	--	--	--	--
CN^-	Not Measured	--	--	--	--	--
NH_3	Not Measured	--	--	--	--	--

Table 12. Contd.

Analyte	C106-OH-8B1 KOH Fusion	Na_2O_2 Fusion	Mean	Std Dev.	Rel% Error	Amount (μCi or μg) in C106-OH-8B1
^{137}Cs	3.25E+01	--	3.25E+01	--	--	6.61E+01
^{90}Sr	1.32E+02	--	1.32E+02	--	--	2.68E+02
^{99}Tc	< 9E-03	--	0.0085	--	--	< 2E-02
$^{241}Am(\gamma)$	3.47E-01	--	3.47E-01	--	--	7.06E-01
$^{241}Am(\alpha)$	2.69E-01	--	2.69E-01	--	--	5.46E-01
^{154}Eu	2.89E-01	--	2.89E-01	--	--	5.88E-01
^{155}Eu	2.00E-01	--	2.00E-01	--	--	4.07E-01
$^{14}C^{(e)}$	Not Measured	--	--	--	--	--
^{129}I	< 3E-04	--	< 3E-04	--	--	5.13E-04
^{235}U	7.70E-06	--	7.70E-06	--	--	1.57E-05
^{238}U	1.30E-04	--	1.30E-04	--	--	2.64E-04
^{237}Np	< 7E-04	--	< 7E-04	--	--	< 1E-03
^{238}Pu	5.25E-02	--	5.25E-02	--	--	1.07E-01
^{239}Pu	3.77E-01	--	3.77E-01	--	--	7.67E-01
^{240}Pu	1.61E-01	--	1.61E-01	--	--	3.27E-01
$^{239+240}Pu$	3.03E-01	--	3.03E-01	--	--	6.16E-01
$^{243+244}Cm$	5.45E-03	--	5.45E-03	--	--	1.11E-02
^{242}Cm	4.86E-04	--	4.86E-04	--	--	9.87E-04
Total Alpha	6.30E-01	--	6.30E-01	--	--	1.28E+00

(a) Concentrations for radionuclides are in units of μCi/g dry solids; all other components are in units of μg/g dry solids. Values in parentheses are within 10 times the analytical detection limit, and thus have a potential measurement uncertainty >15%. Anion (IC) analysis was done performed on this sample.

(b) Because the values obtained from the Na_2O_2 fusion method are generally lower than those obtained by the KOH fusion method, the mean values from the KOH fusions were used to determine the amount of each component in the washed solids (see discussion in the text). Nickel and K are exceptions, because these were only available from the Na_2O_2 fusion.

(c) The process blank for the Na_2O_2 fusion had a Ca content of ~2,500 μg/g.

(d) The process blank for the KOH fusion had a Na content of ~2,500 μg/g.

(e) Sample recoveries for the ^{14}C analysis were low and not reproducible; thus, ^{14}C data are suspect.

Table 13. Concentrations in The Leached and Untreated Solids and the Relative Amount of Each Component Removed by Caustic Leaching

	µg/g dry solids or µCi/g dry solids					
Analyte	Leached Solids[a]	Pseudo 95% C.I. (if %RSDs=10)[b]	Original Sample[c]	Pseudo 95% C.I. (if %RSDs=10)[b]	Removed,%[c]	Pseudo 95% C.I. (if %RSDs=10)[b]
Ag	409	48r	151	17r	7	1.3r
Al	28512	3833r	12445	1384r	22	4.3r
Ba	299	39r	104	13r	2	0.4r
Ca	10313	1373r	3577	470r	1	0.3r
Cd	37	4.4r	(16)	1.6r	19	3.8r
Co	58	6.7r	<25	2.5r	20	3.9r
Cr	302	35r	142	13r	27	5.1r
Cu	152	18r	66	6.3r	22	3.7r
Fe	174950	24315r	59908	8321r	0	0.01r
Hg[f]	105	15r	36	7r	--	--
K	4091	483r	< 1909	181r	27	4.7r
La	78	10r	<31	3.4r	16	3.1r
Mg	2254	297r	791	102r	2	0.5r
Mn	2188	253r	750	86r	0	0.03r
Mo	59	6.9r	<27	2.6r	27	4.7r
Na[e]	135619	18683r	348411	43587r	87	16.5r
Ni	863	103r	301	35r	2	0.4r
P	1366	163r	797	74r	41	7.2r
Pb	1427	168r	<551	58r	11	2.2r
Si	96170	12890r	34280	4416r	4	0.8r
Ti	1720	214r	590	73r	0	0.04r
U	186	22r	165	21r	61	14.1r
Zn	170	21r	<68	7.2r	14	2.6r
Zr	962	132r	<335	45r	2	0.3r
TOC[f]	104300	14750r	35691	7138r	--	--
TIC[f]	1309	185r	448	90r	--	--
Cl^{-}[f]	6158	871r	2107	421r	--	--
F^{-}[f]	6158	871r	2107	421r	--	--
NO_3^{-}[f]	11546	1633r	3951	790r	--	--
SO_4^{2-}[f]	11546	1633r	3951	790r	--	--
PO_4^{3-}[f]	11546	1633r	3951	790r	--	--
$C_2O_4^{2-}$[f]	303662	42944r	103912	20782r	--	--
CN^{-}[f]	3	0.5r	1.2	0.2r	--	--
NH_3[f]	9	1.2r	3.0	0.6r	--	--

Table 13. Contd.

Analyte	Leached Solids(a)	Pseudo 95% C.I. (if %RSDs=10)(b)	Original Sample(c)	Pseudo 95% C.I. (if %RSDs=10)(b)	Removed,%(d)	Pseudo 95% C.I. (if %RSDs=10)(b)
	µg/g dry solids or µCi/g dry solids					
^{137}Cs	8.90E+01	1.16E+01r	9.42E+01	1.10E+01r	68	13r
^{90}Sr(g)	2.57E+02	3.25E+01r	8.80E+01	1.11E+01r	--	--
^{99}Tc	8.59E-03	1.01E-03r	8.73E-03	1.08E-03r	66	14r
^{241}Am(γ)	8.59E-01	1.11E-01r	3.39E-01	3.85E-02r	13	2.4r
^{241}Am(α)(g)	7.07E-01	9.17E-02r	2.42E-01	3.14E-02r	--	--
^{154}Eu	1.02E+00	1.35E-01r	3.53E-01	4.63E-02r	1	0.2r
^{155}Eu	6.47E-01	8.52E-02r	2.68E-01	2.99E-02r	17	3.1r
^{14}C(f)	9.06E-04	1.28E-04r	3.10E-04	6.20E-05r	--	--
^{129}I(g)	1.00E-03	1.34E-04r	3.43E-04	4.58E-05r	--	--
^{235}U(g)	6.09E-06	7.05E-07r	2.09E-06	2.41E-07r	--	--
^{238}U(g)	1.06E-04	1.23E-05r	3.63E-05	4.20E-06r	--	--
^{237}Np(g)	1.48E-03	1.88E-04r	5.06E-04	6.44E-05r	--	--
^{238}Pu(g)	1.50E-01	1.96E-02r	5.13E-02	6.71E-03r	--	--
^{239}Pu(g)	1.02E+00	1.33E-01r	3.49E-01	4.54E-02r	--	--
^{240}Pu(g)	3.04E-01	3.83E-02r	1.04E-01	1.31E-02r	--	--
$^{239+240}$Pu(g)	9.71E-01	1.28E-01r	3.32E-01	4.37E-02r	--	--
$^{243+244}$Cm(g)	2.17E-02	2.90E-03r	7.44E-03	9.93E-04r	--	--
^{242}Cm(g)	1.16E-03	1.50E-04r	3.98E-04	5.13E-05r	--	--
Total Alpha	1.74E+00	2.26E-01r	6.27E-01	7.76E-02r	5	1.0r

(a) The concentration in the leached solids was determined by summing the quantity found in the non-magnetic and the magnetic fractions and dividing by the total weight (8.453 g) of the leached solids.

(b) Pseudo 95% Confidence Intervals (C.I.) were approximated using propagation of error techniques for the case where the %RSD of all analytical measures used is 10% and all measures are independent The reader can review other potential %RSD values by multiplying the cell value by r, where r is %RSD/10.

(c) The concentration in the untreated solids was determined by summing the quantity found in the leach and wash solutions, the non-magnetic and the magnetic solid fractions and dividing by the total weight (24.7022 g) of sample used.

(d) The percent removed was determined by the following formula: %Removed= $100*(F_l+F_w)/(F_l+F_w+F_{nm}+F_m)$; where F_l is the fraction in the leach solution, F_w is the fraction in the wash solution, F_{nm} is the fraction in the non-magnetic solids, and F_m is the fraction in the magnetic solids.

(e) The values for Na are not corrected for Na added as NaOH during the leaching process.

(f) Only the material in the non-magnetic solids are accounted for in these values.

(g) In calculating the concentration in the untreated solids, only the material in the washed solids are accounted for in these values.

Table 14. C-106 Washing/Leaching: Comparison to Previous Studies

	"Water-Washed"				Caustic-Leached Solids					
	µg/g dry solids		µg/g Fe		µg/g dry solids			µg/g Fe		
Analyte	Lumetta 1996	Lumetta 1999	Lumetta 1996	Lumetta 1999	Lumetta 1996	Brooks 1997	Lumetta 1999	Lumetta 1996	Brooks 1997	Lumetta 1999
Ag	1260	381	7171	1208	630	3020	409	3369	22206	2335
Al	86400	65825	491747	208874	69100	49100	28512	369519	361029	162972
Ba	580	521	3301	1653	630	398	299	3369	2926	1709
Ca	3900	15147	22197	48064	6505	5480	10313	34786	40294	58947
Cd	110	< 90	626	284	125	78	(37)	668	574	210
Co	Not Reported	82	Not Reported	260	Not Reported	Not Reported	< 58	Not Reported	Not Reported	333
Cr	1300	1231	7399	3905	1046	711	302	5594	5228	1727
Cu	Not Reported	535	Not Reported	1697	Not Reported	58	152	Not Reported	426	869
Fe	175700	315141	--	--	187000	136000	174950	--	--	--
K	< 4685	< 5285	26665	16771	6710	Not Reported	< 4009	35882	Not Reported	22913
La	< 150	157	854	499	229	Not Reported	< 78	1225	Not Reported	444
Mg	858	3234	4883	10264	1060	1320	2254	5668	9706	12886
Mn	4215	5561	23990	17647	4235	2850	2188	22647	20956	12505
Mo	Not Reported	< 69	Not Reported	220	Not Reported	Not Reported	< 59	Not Reported	Not Reported	336
Na	101700	53848	578828	170871	89700	113000	135619	479679	830882	775187
Ni	2140	1234	12180	3916	2020	1090	863	10802	8015	4931
P	1460	4527	8310	14365	2553	3480	1366	13652	25588	7810
Pb	4020	3326	22880	10554	5107	3310	1427	27310	24338	8159
Si	86500	107390	492316	340767	87400	66600	96170	467380	489706	549702
Ti	640	2928	3643	9293	787	995	1720	4209	7316	9834
U	191	215	1087	682	613	Not Reported	186	3278	Not Reported	1063
Zn	315	333	1793	1056	155	Not Reported	170	829	Not Reported	974
Zr	3860	3287	21969	10432	6210	1050	962	33209	7721	5499

(a) Lumetta 1996 = PNNL-11381; Brooks 1997 = PNNL-11432; Lumetta 1999 = current work.

Appendix A. Test Plan

Work Place Copy

PNNL Test Plan	**Document No.:** BNFL-TP-29953-8 **Rev. No.:** 0

Title: Determination of the Solubility of HLW Sludge Solids

Work Location: RPL/SAL	Page 1 of 21
Author: GJ Lumetta **Use Category Identification:** Mandatory	**Effective Date:** **Supersedes Date:** New
Identified Hazards: __ Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** X Technical Reviewer __ Building Manage __ Radiological Control __ ES&H X Quality Engineer X Other: Client X Other: Project Manager X Other: RPL Manager

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 ___ No
Does the OJT package associated with this procedure require revision to reflect procedure changes? __ Yes__ No__ N/A

Approval:

	Signature	Date
Author	Gregg J. Lumetta	1/13/99
Technical Reviewer	Brian M. Rapko	1-14-99
RPL Manager	[signature]	1/19/99
Project Manager	D E Kurath	1/14/99
RPG QE	[signature]	1/15/98
BNFL	[signature] BNFL QA: [signature]	22/1/98

Applicability

This test plan is to be used to determine 1) the aqueous-insoluble fraction of BNFL HLW sludge samples, 2) the caustic-insoluble fraction of BNFL HLW sludge samples, and 3) the effect of temperature on the solubility of solids in the BNFL HLW sludge samples. The work will be conducted in the SAL hot cells. The work will be conducted by Radiochemical Processing Group staff. This work is being done as part of the Technical Support to BNFL for Phase 1B project.

Test Objectives

Justification: This activity supports confirmation of the process sequence, equipment performance and design basis for the HLW entrained solids removal process. BNFL must complete research and testing activities conducted to confirm system design bases before 14 April 1999.

Objective: The purpose of this task is to obtain the information needed in the filtration and washing of the Envelope D material. The specific objective of this test is to determine the relative mass and composition of the water-insoluble solids and of the caustic-insoluble solids (at 85°C) and to determine the components in the liquid portion of the HLW sample at 30, 40, and 50°C and their concentrations.

Definitions

BNFL	British Nuclear Fuels Ltd.
HDPE	High-density polyethylene
HLW	High-level waste
RPL	Radiochemical Processing Laboratory

Emergency Response

In the event of building audible alarms (e.g., fire or criticality) personnel should proceed in accordance with the RPL Building Emergency Procedure. If time permits, ensure that test materials are secured from spilling prior to exiting the area.

Quality Control

Quality assurance for work conducted under this Test Plan is governed by the Standards-Based Management System (SBMS). The quality control for each analysis indicated in Table 1 will be established per Quality Assurance Plan MCS-033. MCS-033 specifies the minimum calibration and verification requirements for analytical systems, as well as batch processing quality control samples to monitor preparations (i.e., blanks, duplicates, matrix spikes, and laboratory control standards).

A work place copy of this document shall be present at the work location. Specific information regarding each test (e.g., sample numbers) will be recorded on the work place copy and kept as project records.

As discussed in the Prerequisites section, calibrated balances must be used in performing this test. Likewise, a calibrated temperature controller is required. The calibration ID, date of calibration, and calibration expiration date must be recorded on the work place copy for each balance used and for the temperature controller.

Measured weights will be recorded on the work place copy at the indicated spot in the work instructions.

Hand written changes or corrections made to the work place copy will be made by means of a single line-out. Such changes or corrections shall be initialed and dated by the staff member making the change and by the cognizant scientist.

Equipment Description

A standard laboratory hot plate/magnetic stirrer will be used for this test. An aluminum heating block will be placed on the hot plate/stirrer to heat the sample. The apparatus will be equipped with two thermocouples. One of the thermocouples will be connected to a temperature controller, while the other will be connected to an over-temperature shut-off device. The latter will be used to ensure the sample is not over heated, which could result in lose of sample.

Prerequisites

Staff performing the work must read and understand the entire test plan prior to beginning work.

The following are items that should be staged prior to start of the test.

- Wide-mouth HDPE bottle; size to be determined (2)
- 30-mL HDPE bottle
- 20-mL HDPE vial (8)
- 30- to 40-mL glass vials (2)
- Hot plate/stirrer
- Aluminum heating block
- Temperature controller with temperature read-out
- Over-temperature shut-off device
- 0.45-μm nylon syringe filters (6)
- 5-mL syringes (6)
- 0.45-μm nylon disposable filter units (8)
- Adjustable 5-mL pipette
- Boiling water bath
- Small plastic bag

0.1 M NaOH Used ⇒ 0.1014 ± 0.0001 M Prepared by R.G. Swoboda on 7/10/98. Chem Rec-43

0.01 M NaOH prepared by measuring 100 mL of the 0.1 M NaOH solution in a volumetric flask.. this was then transferred to a 1000 mL flask and diluted to the mark with deionized water. M.J. Smith 4/23/99

3.08 ± 0.12 M NaOH
Chem Rec - 29
2/26/97 by RGS

The temperature controller shall be calibrated by maintenance services. Record the following information regarding the temperature controller used.

		Thermocouples
Calibration ID:	02093	02899 / 02900
Calibration Date:	1/12/99	1/99
Expiration Date:	1/2000	1/2001

A calibrated balance is required for this test. Record the following information regarding the balance(s) used.

Calibration ID:	Cell-2 360-06-01-016	Calibration ID:	
Calibration Date:	3/2/99 ex 8/99	Calibration Date:	

Expiration Date: 8/99 Expiration Date: ________

Before beginning work, a routine performance check should be performed and documented in the space below.

Wt., g	Found
1	0.9996
10	9.9997
50	49.9995
100	99.9994
150 (100+50)	149.9997

M.L. 4/20/99

Work Instructions

> **Note**
> Where practical, catch pans should be used when working with the tank waste samples, so that they can be recovered if spilled.

Part 1. Solubility Versus Temperature

4/20/99 1.1. Prepare the sample vials according to the following table. All vials should be HDPE.

Sample ID[a]	Tare wts.
C106 -SOL-30-1	6.4858
C106 -SOL-30-2	6.4995
C106 -SOL-40-1	6.4695
C106 -SOL-40-2	6.4899
C106 -SOL-50-1	6.4988
C106 -SOL-50-2	6.4985

(a) The prefix to the sample IDs should be the tank number; e.g. "C106."

1.2. Label a ~~30~~ 60 M.L. 4/19/99 -mL HDPE bottle as "C106 -SOL-TEST" (______ = tank number) and place a magnetic stir bar in this bottle. Weigh C106-SOL-TEST → 15.6573 g

1.3. ~~Mix the stock HLW sample to give a homogeneous slurry~~ → C106-B1 ~~wt~~ M.L. 4/19/99

Weigh ~~8~~ 10 g of the dry material in C106-B1 into C106-SOL-TEST.

wt. C106-SOL-TEST with sample = 25.7730 g

wt. sample = 25.7730 − 15.6573 = 10.1157 g

Note: the C106 sample ~~was~~ consisted of dry chunks of material.

 M.L. 4/20/99

0.1014 ± 0.0001 M NaOH Prepared 7/10/98 by RG Swoboda Chem Rec. 43

Note

If the HLW sample does not contain a liquid fraction, then add ~5 g of sludge to 25 mL of 0.1 M NaOH.

4/20/99

1.4. ~~Transfer approximately 25 mL of the homogenized HLW slurry to C106 -SOL-TEST~~ Add 50 mL of 0.1 M NaOH to C106-SOL-TEST, then weigh. wt. C106-SOL-TEST = 75.1999 g

1.5. Place C106 -SOL-TEST into an aluminum heating block thermostatted at 30°C wt. 0.1 M NaOH: 75.1999 − 25.7730 = 49.4269

1.6. Stir the contents of C106 -SOL-TEST

1.7. Once the temperature has equilibrated at 30°C, stir the sample for 1 h (Stir bar = 1.4366 g removed) (Stir bar = 3.1818 g installed) larger stir bar added.

Note: We had problems with the stir bar stopping (this happened during the night.) Changing to a larger stir bar seemed to help, but stirring did stop on occasion.

Start date/time: ~~1517~~ 1517 / 4-20-99 (4-21-99/1034)

Stop date/time: 4/21/99 13:25

4/21/99

1.8. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

1.9. Withdraw a ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-30-1 Slurry filtered easily. wt. C106-SOL-30-1 = 10.9521

wt. sample = 10.9521 − 6.4858 = 4.4663 g

1.10. Withdraw a second ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-30-2 wt. C106-SOL-30-2 = 10.7155

wt. sample = 10.7155 − 6.4995 = 4.2160

1.11. Adjust the temperature of aluminum heating block assembly to 40°C

1.12. Once the temperature has equilibrated at 40°C, stir the sample for 1 h Sample stirred OK all night.

Start date/time: 4/21/99 13:35

Stop date/time: 4/22/99 8:05

4/22/99

1.13. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

1.14. Withdraw a ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-40-1 → wt = 10.7522

wt. sample = 10.7522 − 6.4695 = 4.2827

1.15. Withdraw a second ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-40-2 → wt. = 10.6778

wt. sample = 10.6778 − 6.4899 = 4.1879

1.16. Adjust the temperature of aluminum heating block assembly to 50°C

1.17. Once the temperature has equilibrated at 50°C, stir the sample for 1 h

Start date/time: 4/22/99 8:15

Stop date/time: 4/22/99 8:20

4/23/99

1.18. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

1.19. Withdraw a ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-50-1 → wt = 10.04~~6~~4 M.L.L. 4/23/99

wt sample = 10.0464 − 6.4988 = 3.5476

Note: Although it filtered fine at first, the filter plugged after about 3 mL had filtered. This might have been because more solids were pulled in this aliquot.

S.J. [illegible] 4/23/99

4/23/99

1.20. Withdraw a second ~~2~~ 4-mL aliquot of the slurry and filter into vial C106 -SOL-50-2 → wt = 10.8641

wt. sample = 10.8641 − 6.4985 = 4.3656

1.21. The samples collected during the test are to be submitted for the analyses listed in Table 1. The cognizant scientist will prepare the required ASR.

wt. C106-SOL-TEST = 47.5759 ⇒ wt. slurry remaining = 47.5759 − 15.6573 (original tare) − 1.4366 (original stir bar) + 3.1818 (new stir bar) = 30.1734 g

S.J. [signature]

Part 2. Determination of Aqueous-Insoluble Fraction

2.1. Homogenize the stock HLW sample by stirring

2.2 Label a disposable filter unit (0.45-μm nylon) as C106 -AQ-1

2.3 Weigh C106 -AQ-1

Wt. C106 AQ-1 = ________ g (2.3A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = ________ g (2.3B)

2.4 Connect C106 -AQ-1 to the vacuum line, but do not yet apply vacuum

2.5 Transfer enough of the homogenized HLW sample to give ~25 g solids to the filter funnel of C106 -AQ-1

2.6 Apply vacuum to the filter unit. Disconnect from the vacuum once the liquid has filtered.

2.7 Place the cap on the top of the filter unit and weigh C106 -AQ-1

Wt. C106 -AQ-1 = ________ g (2.7A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = ________ g (2.7B)

2.8 Determine the total weight of the sample

Wt. Sample = 2.7A-2.3A = ________ g (2.8A)

Determine the weight of the filtered liquid

Wt. Liquid = 2.7B-2.3B = ________ g (2.8B)

Determine the weight of the filtered solids

Wt. Solids = 2.8A-2.8B = ________ g (2.8C)

2.9 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Because the C-106 sample is a dry solid, we will just weigh out an appropriate amount (25g). S.J. [signature] 4/23/99

~~Vol. Used = ________ mL ________ (2.9A)~~ M.J. Hewitt 4/23/99

2.10 Label an appropriately sized 125-mL wide-mouthed HDPE bottle as C106-AQ-2

5/3/99

2.11 Weigh C106-AQ-2

Wt. C106-AQ-2 = 31.6504 g (2.11A)

Changes by M.J. Hewitt 4/23/99

2.12 ~~Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 2.9A + 5); transfer this slurry to C106-AQ-2~~ Using a spatula, break up any large chunks in the C-106-B1 sample, then mix the dry sample well using the spatula. Transfer ~25 g of sample from C-106-B1 to C106-AQ-2.

2.13 ~~Repeat step 2.12 four times to ensure complete transfer of the solids to C106-AQ-2~~

2.14 Weigh C106-AQ-2

Wt. C106-AQ-2 = 55.1281 g (2.14A)

2.14a Fill C106-AQ-2 to capacity with 0.01 M NaOH.
Weight C106-AQ-2 = 159.5334 (a) g

Determine the weight of the ~~slurry~~ sample

Wt. ~~Slurry~~ Sample = 2.14A-2.11A = 23.4777 g (2.14B)

wt. slurry = 159.5334 − 31.6504 = 127.8830

(a) When C106-AQ-2 was tared in step 2.11, it was set on an overturned paper cap so that the stir bar would not affect the balance. However we couldn't do this in 2.14 because this would exceed cap. of balance

2.15 Equip C106-AQ-2 with a condenser, then place in an aluminum heating block at 85°C

2.16 Stir the sample in C106-AQ-2 at 85°C for a minimum of 8 hours

Start date/time: 5/3/99 14:00
Stop date/time: 5/4/99 8:15
(18 h 15 min)

~~2.17 Allow to cool to ambient temperature~~ M.J. Hewitt 3/10/99

(a) Per instructions from Mike Johnson of BNFL, steps 2.17 and 2.18 were eliminated so that the solution could be filtered while hot.

~~2.18 Remove the condenser and replace the original cap on ______-AQ-2. Weigh ______-AQ-2.~~

~~Wt. ______-AQ-2 = ______ g (2.18A)~~

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 2.18A-2.14A = ______ g (2.18B)~~

2.19 Label a disposable filter unit (0.45-µm nylon) as C106-AQ-3

2.20 Weigh C106-AQ-3

Wt. C106 AQ-3 = 64.5858 g (2.20A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.7709 g (2.20B)

2.21 Connect C106-AQ-3 to the vacuum line

M.J.H. 5/4/99

5/4/99

Note: There was some chunky solids still present after the wash step. Also there appeared to be considerable magnetic material stuck to the stir bar.

2.22 Filter the ^hot wash slurry D.L.L. 5/10/99

2.23 Disconnect from the vacuum once the liquid has filtered

2.24 Place the cap on the top of the filter unit and weigh C106 -AQ-3

Wt. C106-AQ-3 = ________ g (2.24A)

Could not weigh because it exceeded capacity of balance. D.L.L. 5/4/99

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

(b) Monitor the clarified solution for precipitate formation.
5/4/99 9:30 soln. clear.
5/4/99 ~15:00 still clear
5/6/99 8:30 still clear

Wt. receiving bottle&cap = 144.5274 g (2.24B)

5/10/99 13:00 Some solids had precipitated in C106-AQ-3

2.25 Determine the total weight of the slurry

Wt. Slurry = ~~2.24A~~-2.20A = ________ g (2.25A)

Could not determine (see above)

Determine the weight of the filtered liquid

Wt. Liquid = 2.24B-2.20B = 102.7565 g (2.25B)

Determine the weight of the filtered solids

Wt. Solids = ~~2.25A~~-2.25B = ________ g (2.25C)

Could not determine.

2.26 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 100 mL (estimated) (2.26A)

2.28 Weigh C106 -AQ-2

Wt. C106 -~~AQ-2~~ = ________ g (2.28A)

missed weight.

2.29 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 2.26A ÷ 5); transfer this slurry to C106 -AQ-2 ~10-mL aliquots used. Transfer of solids was somewhat difficult because they were very sticky when ~~wet~~ wet.

2.30 Repeat step 2.29 four times to ensure complete transfer of the solids to C106 -AQ-2

2.31 Weigh C106 -AQ-2

Wt. C106 -AQ-2 = 140.4774 g (2.31A)

Determine the weight of the slurry

Wt. Slurry = 2.31A-~~2.28A~~ 2.11A (31.6504 g) = 108.8270 g (2.31B)

2.32 Equip C106 -AQ-2 with a condenser, then place in an aluminum heating block at 85°C

D.L.L. 5/4/99

2.33 Stir the sample in C106 -AQ-2 at 85°C for a minimum of 8 hours

Start date/time: 5/4/99 9:30 23 h
Stop date/time: 5/5/99 8:30

~~2.34 Allow to cool to ambient temperature~~

~~2.35 Remove the condenser and replace the original cap on ______ AQ-2. Weigh ______ -AQ-2~~

~~Wt. ______ -AQ-2 = ______ g (2.35A)~~

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 2.35A-2.31A = ______ g (2.36B)~~

N.J. [signature] 5/10/99 see note (a) on page 7

2.36 Label a disposable filter unit (0.45-μm nylon) as C106 -AQ-5

2.37 Weigh C106 -AQ-5

Wt. C106 AQ-5 = 64.9164 g (2.37A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.7804 g (2.37B)

2.38 Connect C106 -AQ-5 to the vacuum line

2.39 Filter the hot wash slurry N.J.L. 5/10/99 The big chunks had broken up. There was definitely magnetic material stuck to the stir bar — looked like Fe filings.

2.40 Disconnect from the vacuum once the liquid has filtered

2.41 Place the cap on the top of the filter unit and weigh C106 -AQ-5

Wt. C106 AQ-5 = ______ g (2.41A) Exceeded balance capacity. N.J.L. 5/5/99

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Note: monitor the clarified liquid for the formation of solids.

Wt. receiving bottle&cap = 135.7813 g (2.41B)

5/6/99 solution clear. 5/11/99 13:00 still clear // 5/12/99 8:25 → Precipitate had formed.

2.42 Determine the total weight of the slurry

Wt. Slurry = 2.41A-2.37A = ______ g (2.42A) Could not determine. N.J.L. 5/5/99

Determine the weight of the filtered liquid

Wt. Liquid = 2.41B-2.37B = ~~135.7813~~ 94.0009 g (2.42B) N.J.L. 5/5/99

N.J. [signature] 5/5/99

Determine the weight of the filtered solids

Wt. Solids = 2.42A-2.42B = ________ g (2.42C) Could not determine M.A.L. 5/5/99

5/5/99 2.43 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = 100 mL (estimated) (2.43A)

2.44 ~~Label an appropriately sized wide-mouthed HDPE bottle as C106 -AQ-2~~ step not needed. M.A.L. 4/23/99

2.45 Weigh C106 -AQ-2

Wt. C106 -AQ-2 = ________ g (2.45A) This weight not really needed. M.A.L. 5/5/99

2.46 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 2.43A ÷ 5); transfer this slurry to C106 -AQ-2 Used a spatula to transfer most of the solids to AQ-2. Then used several portions of 0.01 M NaOH to transfer the remainder.

2.47 Repeat step 2.46 four times to ensure complete transfer of the solids to C106 -AQ-2 Then filled AQ-2 to ~80% full. with 0.01 M NaOH.

2.48 Weigh C106 -AQ-2

Wt. C106 -AQ-2 = 144.0396 g (2.48A)

Determine the weight of the slurry

Wt. Slurry = 2.48A-2.~~45~~A = 112.3892 g (2.48B) 2.11A (31.6504)

2.49 Equip C106 -AQ-2 with a condenser, then place in an aluminum heating block at 85°C

2.50 Stir the sample in C106 -AQ-2 at 85°C for a minimum of 8 hours

Start date/time: 5/5/99 9:20 ~27 h
Stop date/time: 5/6/99 13:00

~~2.51 Allow to cool to ambient temperature~~

~~2.52 Remove the condenser and replace the original cap on ________ -AQ-2. Weigh ________ -AQ-2~~

~~Wt. ________ -AQ-2 = ________ g (2.52A)~~

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 2.52A-2.48A = ________ g (2.52B)~~

M.A. ... 3/10/99 See note (a) on page 7

2.53 Label a disposable filter unit (0.45-μm nylon) as C106 -AQ-7

2.54 Weigh C106 -AQ-7

Wt. C106 -AQ-7 = 64.8363 g (2.54A)

M.A. ... 5/6/99

5/6/99

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.5514 g (2.54B)

2.55 Connect C106 -AQ-7 to the vacuum line

2.56 Filter the hot wash slurry M.A.L. 3/10/99 (Filtration was fairly slow.)
Several portions of 0.01M NaOH were used to rinse solids from C106-AQ-2 to the filter funnel.

2.57 Disconnect from the vacuum once the liquid has filtered

2.58 Place the cap on the top of the filter unit and weigh C106 -AQ-7

Wt. ~~C106 AQ-7~~ = ________ g (2.58A) Exceeds cap. of balance. M.A.L. 5/6/99

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Note: Needed to weigh cap & bottle separately
Bottle w/ soln = 153.4088
Cap = 14.0655

Note: Immediately after filtering, the solution looked cloudy.
5/10/99 13:00 ~~Still~~ Solution looked a bit hazy, but no clear precipitate evident.
5/12/99 8:25 Still the same.

Wt. receiving bottle&cap = 167.4743 g (2.58B)

2.59 Determine the total weight of the slurry

Wt. ~~Slurry = 2.58A-2.54A~~ = ________ g (2.59A) Cannot determine 5/6/99

Determine the weight of the filtered liquid

Wt. Liquid = 2.58B-2.54B = 125.9229 g (2.59B)

Determine the weight of the filtered solids

Wt. ~~Solids = 2.59A-2.59B~~ = ________ g (2.59C) Cannot determine M.A.L. 5/6/99

2.60 Label a glass ~~vial~~ jar as C106 -AQ-8 Labeled another jar as C106-AQ-8B (this will be used for the stir bar + magnetic solids)

2.61 Dry C106 -AQ-8 and -8B at 105°C for a minimum of 1 h

2.62 Cool C106 -AQ-8 and -8B to ambient temperature in a desiccator

2.63 Weigh C106 -AQ-8 and -8B

Wt. C106 -AQ-8 = 127.5829 g (2.63A)
wt. C106 -AQ-8B = 92.0791

2.64 Using several portions of deionized water, quantitatively transfer the washed solids from the filter membrane to C106 -AQ-8 and -8B

Put stir bar w/ magnetic solids in -8B.
Took video image of this

2.65 Heat C106 -AQ-8 and -8B at 80°C to evaporate excess water

2.66 Heat C106 -AQ-8 and -8B at 105°C overnight

2.67 Cool C106 -AQ-8 and -8B to ambient temperature in a desiccator

2.68 Weigh C106 -AQ-8 and -8B

Wt. C106 -AQ-8 = 129.6115 g (2.68A)

wt. C106-AQ-8B = 100.0532

2.69 Determine the dry weight of the washed solids

Wt. Dry Solids = 2.68A-2.63A = 2.0286 g(2.69A)

wt. wet solids + stirbar = 7.9741

2.70 Determine the relative amounts of each wash solution needed to prepare the composite liquid sample

Note
If solids have formed in any of the solutions, contact G.J. Lumetta.

102.7565 94.0009 125.9229

Total Wt. Liquids = ~~2.8B~~ + 2.25B + 2.42B + 2.59B = 322.6803 g (2.70A)

~~Wt Fraction AQ-1 = 2.8B/2.70A = ______ (2.70B)~~

Wt Fraction AQ-3 = 2.25B/2.70A = 0.3184 (2.70C)

Wt Fraction AQ-5 = 2.42B/2.70A = 0.2913 (2.70D)

Wt Fraction AQ-7 = 2.59B/2.70A = 0.3902 (2.70E)

2.71 Label a 20-mL HDPE sample vial as C106 -AQ-9 → wt = 6.4782

2.72 Place C106 -AQ-9 on the balance and tare to 0.000g

2.73 ~~Add the following quantity of the solution in bottle C106 -AQ-1 to C106 -AQ-9~~

~~Quantity from C106 -AQ-1 = 10*2.70B = ______ g (2.73A)~~

~~Record the weight of C106 -AQ-9~~

~~Wt. C106 -AQ-9 = ______ g (2.73B)~~

5/10/99 2.74 Place C106 -AQ-9 on the balance and tare to 0.000g

2.75 Add the following quantity of the solution in bottle C106 -AQ-3 to C106 -AQ-9

Quantity from C106 -AQ-3 = 10*2.70C = ~~8~~ 3.184 g (2.75A)

Note
-AQ-3 was mixed by shaking to suspend solids.

Record the weight of C106 -AQ-9

Wt. C106 -AQ-9 = 3.1842 g (2.75B)

2.76 Place C106 -AQ-9 on the balance and tare to 0.000g

2.77 Add the following quantity of the solution in bottle C106 -AQ-5 to C106 -AQ-9

Quantity from C106 -AQ-5 = 10*2.70D = 2.913 g (2.77A)

M.J. Lumetta
5/10/99

Record the weight of C106 -AQ-9

Wt. C106 -AQ-9 = 2.9249 g (2.77B)

2.78 Place C106 -AQ-9 on the balance and tare to 0.000g

2.79 Add the following quantity of the solution in bottle C106 -AQ-7 to C106 -AQ-9

Quantity from C106 -AQ-7 = 10*2.70E = 3.902 g (2.79A)

Record the weight of C106 -AQ-9

Wt. C106 -AQ-9 = 3.9146 g (2.79B)

Total wt. sample = 3.1842 + 2.9249 + 3.9146 = 10.0237 (wt + wt. vial = 10.0237 + 6.4782 = 16.5019)

2.73 The washed solids and the composite wash solution are to be submitted for the analyses listed in Table 1. The cognizant scientist will prepare the required ASR.

Part 3. Determination of Caustic-Insoluble Fraction

3.1. Homogenize the stock HLW sample by stirring

3.2 Label a disposable filter unit (0.45-μm nylon) as C106 -OH-1

3.3 Weigh C106 -OH-1

Wt. C106 -OH-1 = ________ g (3.3A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = ________ g (3.3B)

3.4 Connect C106 -OH-1 to the vacuum line, but do not yet apply vacuum

3.5 Transfer enough of the homogenized HLW sample to give ~25 g solids to the filter funnel of C106 -OH-1

3.6 Apply vacuum to the filter unit. Disconnect from the vacuum once the liquid has filtered.

3.7 Place the cap on the top of the filter unit and weigh C106 -OH-1

Wt. C106 -OH-1 = ________ g (3.7A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Wt. receiving bottle&cap = ________ g (3.7B)

Steps not needed. [illegible] 5/6/99

3.8 Determine the total weight of the sample

Wt. Sample = 3.7A-3.3A = ________ g (3.8A)

Determine the weight of the filtered liquid

Wt. Liquid = 3.7B-3.3B = ________ g (3.8B)

Determine the weight of the filtered solids

Wt. Solids = 3.8A-3.8B = ________ g (3.8C)

5/10/99 A.L.L. 3.9 Measure out the appropriate volume of 3 M NaOH as instructed by the cognizant scientist into a plastic bottle

A.L.L. 5/10/99 100mL Used

Vol. Used = ________ mL (3.9A)

Steps not used A.L. Fumetta 5/6/99

3.10 Label an appropriately sized wide-mouthed HDPE bottle as C106 -OH-2

3.11 Weigh C106 -OH-2

Wt. C106 -OH-2 = 31.8218 g (3.11A)

3.12 ~~Slurry the filtered solids using a portion of 3 M NaOH (volume = 3.9A ÷ 5); transfer this slurry to C106 -OH-2~~ Transfer remaining material in C-106-B1 into C106-OH-2.

3.13 ~~Repeat step 3.12 four times to ensure complete transfer of the solids to C106 -OH-2~~

3.14 Weigh C106 -OH-2

Wt. Bottle with 100mL 3M NaOH = ~~147.622~~ 150.2024 A.L.L. 5/10/99

Wt. C106 -OH-2 = 56.5240 g (3.14A)

Wt. Bottle after NaOH transferred to C106-OH-2 = ~~44.4~~ 42.1190

Determine the weight of the ~~slurry~~ sample

Wt. 3M NaOH = 108.08 g

Wt. ~~Slurry~~ Sample = 3.14A-3.11A = 24.7022 g (3.14B)

3.15 Equip C106 -OH-2 with a condenser, then place in an aluminum heating block at 85°C

3.16 Stir the sample in C106 -OH-2 at 85°C for a minimum of 8 hours

Start date/time: 5/10/99 14:20 ~20h
Stop date/time: 5/11/99 9:35

~~3.17 Allow to cool to ambient temperature.~~

~~3.18 Remove the condenser and replace the original cap on ________ -OH-2.~~
~~Weigh ________ -OH-2~~

~~Wt. ________ -OH-2 = ________ g (3.18A)~~

A.L. Fumetta 5/10/99 see note (a) on p.7

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 3.18A-3.14A = ________ g (3.18B)~~ n.l. [illegible] 5/10/99

5/11/99 3.19 Label a disposable filter unit (0.45-μm nylon) as C106 -OH-3

3.20 Weigh C106 -OH-3

Wt. C106 -OH-3 = 64.8649 g (3.20A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.9018 g (3.20B)

3.21 Connect C106 -OH-3 to the vacuum line

3.22 Filter the hot leaching slurry n.l.h. 5/10/99

3.23 Disconnect from the vacuum once the liquid has filtered

3.24 Place the cap on the top of the filter unit and weigh C106 -OH-3

Wt. C106 ~~-AQ-3~~ = ________ g (3.24A) Exceeded balance capacity. n.l.h. 5/11/99

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh.

Note: "Monitor the clarified solution for precipitate.
5/12/99 8:25 Soln. Clear.
5/14/99 8:40 Soln. Clear
5/20/99 9:00 soln. clear

Wt. receiving bottle&cap = 145.9756 g (3.24B)

3.25 Determine the total weight of the slurry

Wt. Slurry = ~~3.24A-3.20A~~ = ________ g (3.25A)

Could not be determined because we could not determine 3.24A.

Determine the weight of the filtered liquid

Wt. Liquid = 3.24B-3.20B = 104.0738 g (3.25B)

Determine the weight of the filtered solids

Wt. Solids = ~~3.25A-3.25B~~ = ________ g (3.25C)

3.25a Label a 20-mL HDPE sample vial as C106 -OH-3A → wt. = 6.9483

3.25b Transfer ~15 mL of the filtered leachate solution to C106 -OH-3A → wt = 23.4416
23.4416 - 6.9483 = 16.4933g sample

3.26 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = ~100 mL (3.26A)

n.l. [illegible] 5/11/99

5/11/99 3.28 Weigh C106 -OH-2

Did not weigh (not critical). S.A.L. 5/11/99

Wt. ~~C106~~ -OH-2 = ________ g (3.28A)

3.29 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 3.26A ÷ 5); transfer this slurry to C106 -OH-2 Transferred most of the solids with a spatula, then slurried the rest with 0.01 M NaOH.

3.30 Repeat step 3.29 four times to ensure complete transfer of the solids to C106 -OH-2

3.31 Weigh C106 -OH-2

Wt. C106 -OH-2 = 146.6984 g (3.31A)

Determine the weight of the slurry

Wt. Slurry = 3.31A-~~3.28A~~ 31.8218 (3.11A) = 114.8766 g (3.31B)

3.32 Equip C106 -OH-2 with a condenser, then place in an aluminum heating block at 85°C

3.33 Stir the sample in C106 -OH-2 at 85°C for a minimum of 8 hours

Start date/time: 5/11/99 9:20
Stop date/time: 5/12/99 8:20 23 h

~~3.34 Allow to cool to ambient temperature~~

~~3.35 Remove the condenser and replace the original cap on ________ -OH-2. Weigh ________ -OH-2~~

~~Wt. ________ -OH-2 = ________ g (3.35A)~~

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 3.35A-3.31A = ________ g (3.35B)~~

S.A. Lunetta 3/10/99 see note (A) on page 7

5/12/99 3.36 Label a disposable filter unit (0.45-μm nylon) as C106 -OH-5

3.37 Weigh C106 -OH-5

Wt. C106 -OH-5 = 58.5046 g (note: w/o lid on funnel) (3.37A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.4511 g (3.37B)

3.38 Connect C106 -OH-5 to the vacuum line

3.39 Filter the hot wash slurry S.A.L. 3/11/99

3.40 Disconnect from the vacuum once the liquid has filtered

S.A. Lunetta 5/12/99

5/12/99 3.41 Place the cap on the top of the filter unit and weigh C106 -OH-5

Wt. C106 -OH-5 = 160.8283 g (3.41A)

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh. Note: Solution was very colored (darker than -OH-3).

Note: Monitor the clarified solution for precipitate.

5/14/99 Solution hazy. Wt. receiving bottle&cap = 133.1769 g (3.41B)

3.42 Determine the total weight of the slurry

Wt. Slurry = 3.41A-3.37A = 102.3237 g (3.42A)

Determine the weight of the filtered liquid

Wt. Liquid = 3.41B-3.37B = 91.7258 g (3.42B)

Determine the weight of the filtered solids

Wt. Solids = 3.42A-3.42B = 10.5979 g (3.42C)

3.43 Measure out the appropriate volume of 0.01 M NaOH as instructed by the cognizant scientist into a plastic bottle

Vol. Used = ~100 mL (3.43A)

3.45 Weigh C106 -OH-2

~~Wt. C106 -OH-2 = ____ g~~ → Weight not needed. (can use 3.11A) (3.45A)

3.46 Slurry the filtered solids using a portion of 0.01 M NaOH (volume = 3.43A ÷ 5); transfer this slurry to C106 -OH-2 Used a spatula to transfer the bulk of the solids, then slurried the rest with 0.01 M NaOH.

3.47 Repeat step 3.46 four times to ensure complete transfer of the solids to C106 -OH-2

3.48 Weigh C106 -OH-2

Wt. C106 -OH-2 = 143.6860 g (3.48A)

Determine the weight of the slurry

Wt. Slurry = 3.48A-~~3.45A~~ 31.8218 (3.11A) = 111.8642 g (3.48B)

3.49 Equip C106 -OH-2 with a condenser, then place in an aluminum heating block at 85°C

3.50 Stir the sample in C106 -OH-2 at 85°C for a minimum of 8 hours

Start date/time: 5/12/99 9:00

Stop date/time: 5/13/99 12:55 28h

~~3.51 Allow to cool to ambient temperature~~

~~3.52 Remove the condenser and replace the original cap on ________-OH-2. Weigh ________-OH-2~~

~~Wt. ________-OH-2 = ________g~~ ~~(3.52A)~~

~~Determine mass loss due to evaporation~~

~~Wt. Lost = 3.52A-3.48A = ________g~~ ~~(3.52B)~~

D.A. [signature] 3/11/99 see note (9) on page 7

5/13/99 3.53 Label a disposable filter unit (0.45-μm nylon) as C106-OH-7

3.54 Weigh C106-OH-7

Wt. C106-OH-7 = 58.8763 g (without cap) (3.54A)

Also weigh just the bottom part of the filter unit; i.e., the receiving bottle and cap

Wt. receiving bottle&cap = 41.4739g (3.54B)

3.55 Connect C106-OH-7 to the vacuum line

3.56 Filter the hot wash slurry D.A.L. 5/11/99 As in step 2.56, the filtration was slow and the filtrate appeared cloudy.

3.57 Disconnect from the vacuum once the liquid has filtered

3.58 Place the cap on the top of the filter unit and weigh C106-OH-7

Wt. C106-OH-7 = ________g (3.58A) Exceeded cap. of balance

Carefully remove the funnel part of the apparatus from the receiving bottle, place the cap on the receiving bottle and weigh. w/o cap ← ~~151.36~~ 151.3460 (was drifting down) 14.0795 (cap)

Note: Monitor the clarified solution for precipitate 5/14/99 Reddish solids present

Wt. receiving bottle&cap = 165.4255g (3.58B)

3.59 Determine the total weight of the slurry

Wt. Slurry = 3.58A-3.54A = ________g (3.59A) Could not determine

Determine the weight of the filtered liquid

Wt. Liquid = 3.58B-3.54B = 123.9516g (3.59B)

Determine the weight of the filtered solids

Wt. Solids = 3.59A-3.59B = ________g (3.59C)

3.60 Label a glass vial as C106-OH-8 and another as C106-OH-8B

D.A. [signature] 5/14/99

3.61 Dry C106 -OH-8 and C106-OH-8B at 105°C for a minimum of 1 h

3.62 Cool C106 -OH-8 and C106-OH-8B to ambient temperature in a desiccator

3.63 Weigh C106 -OH-8 and C106-OH-8B --

Wt. C106 -OH-8 = 127.3387 g (3.63A)

Wt. C106-OH-8B = 88.1971

5/14/99 most solids transferred with spatula, then slurried the rest with H_2O.

3.64 Using several portions of deionized water, quantitatively transfer the washed solids from the filter membrane to C106 -OH-8 Put stir bar with magnetic solids in C106-OH-8B

3.65 Heat C106 -OH-8 and C106-OH-8B at 80°C to evaporate excess water

3.66 Heat C106 -OH-8 and C106-OH-8B at 105°C overnight

3.67 Cool C106 -OH-8 and C106-OH-8B to ambient temperature in a desiccator

In oven until 5/20/99

Note: White solid around the walls of C106-OH-8.

3.68 Weigh C106 -OH-8 and C106-OH-8B

Wt. C106 -OH-8 = 133.8453 g (3.68A)

Wt. C106-OH-8B = 97.4508

3.69 Determine the dry weight of the washed solids

That's an eight. [initials] 5/20/99

Wt. Dry Solids = 3.68A-3.63A = 6.5066 g(3.69A)

3.70 Determine the relative amounts of each wash solution needed to prepare the composite liquid sample

Note: If any solids have formed in the solutions, contact Cal. Lumetta

Total Wt. Liquids = 3.42B + 3.59B = 215.6774 g (3.70A)

91.7258 123.9516

Wt Fraction OH-5 = 3.42B/3.70A = 0.4253 (3.70B)

Wt Fraction OH-7 = 3.59B/3.70A = 0.5747 (3.70C)

3.71 Label a 20-mL HDPE sample vial as C106 -OH-9

3.72 Place C106 -~~AQ~~ OH-9 on the balance and tare to 0.000g

3.73 Add the following quantity of the solution in bottle C106 -OH-5 to C106 -OH-9

Quantity from C106 -OH-5 = 10*3.70B = 4.253 g (3.73A)

Record the weight of C106 -OH-9

Wt. C106 -OH-9 = 4.2484 g (3.73B)

3.74 Place C106 -~~AQ~~ OH-9 on the balance and tare to 0.000g

3.75 Add the following quantity of the solution in bottle C106 -OH-7 to C106 -OH-9

Quantity from C106 -OH-7 = 10*3.70C = 5.747 g (3.75A)

Record the weight of C106-OH-9

5/14/99
n.1.2.

Wt. C106-OH-9 = 5.8149 g (3.75B)

3.76 The washed solids, the leaching solution, and composite wash solution are to be submitted for the analyses listed in Table 1. The cognizant scientist will prepare the required ASR.

END of Work Instructions

5/27/99

Checked for Solids Again

C106-AQ-5 → Had lots of white solid

AQ-7 → solution hazy, but no bulk solids

OH-3 → Solution Clear

OH-5 → Some precipitate present.

OH-7 → Solids present.

6/24/99 Because we didn't have a tare wt. on vial C106-OH-9, we could not determine if there had be loss to evaporation before analytical work was begun. For this reason, we went back and weighed what remained at this point. (Note: The solution had considerable solids in it; this was after material had been transferred to a glass vial.)

The weight of C106-OH-9 material remaining → 22.7674 g gross
17.5375 g tare
5.2269 g

To this we need to add quantity used for acid digestion.

5.2269
+ 4.2058
9.4327 g → "weight at time of analysis"

Original wt.
4.2484 (3.73B)
+ 5.8149 (3.75B)
10.0633 g

TABLE 1. Sample Matrix

Sample ID	Acid Digestion	KOH Fusion	Na_2O_2 Fusion	ICP/AES	IC (anions)	TOC	TIC	ICP-MS (^{99}Tc)	ICP-MS (Full)[a]	GEA	^{90}Sr	Total Alpha	Laser Fluorimetry (U)	^{214}Am/ $^{243+244}Cm$	^{14}C	Tritium	CVAA Hg	Total CN	Ammonia
SOL-30-1	X			X	X	X	X	X		X	X	X	X						
SOL-30-2	X			X	X	X	X	X		X	X	X	X						
SOL-40-1	X			X	X	X	X	X		X	X	X	X						
SOL-40-2	X			X	X	X	X	X		X	X	X	X						
SOL-50-1	X			X	X	X	X	X		X	X	X	X						
SOL-50-2	X			X	X	X	X	X		X	X	X	X						
AQ-8		X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X
AQ-9	X			X	X	X	X		X	X	X	X	X	X	X	X	X	X	X
OH-3A	X			X	X	X	X		X	X	X	X	X	X	X	X	X	X	X
OH-8		X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X
OH-9	X			X	X	X	X		X	X	X	X	X	X	X	X	X	X	X

(a) Includes Tc-99, I-129, Np-237, U-isotopic, Pu-isotopic

Protocol for Dissolving Sample C106-AQ-8B

Purpose

The purpose of this protocol is to dissolve the magnetic solids stuck to the stir bar from the C-106 washing test so that they can be analyzed.

Instructions

5/11/99

1. Weigh C106-AQ-8B

   Wt. C106-AQ-8B = 99.8543 g (1a)

2. Add 10 mL of concentrated (12 M) HCl (Ultrex-grade) to C106-AQ-8B

3. Place the cap loosely on C106-AQ-8B and stir gently to dissolve the solids After stirring for ~3.5 h. there was still a small amount of solid. Heated gently, ~1 h. Most solids dissolved.

4. Once all solids are dissolved, transfer the solution to a 25-mL volumetric flask. Filtered through 0.45-um filter (~~[illegible] HCl [illegible] hole in nylon membrane~~) No: It's OK

5. Rinse C106-AQ-8B with several small portions of deionized water, transferring the rinse liquid to the volumetric flask. Solids formed in vol. flask as diluted with H_2O.

6. Fill the volumetric flask to the 25-mL mark with deionized water, then mix. 5/12/99 Transferred to a beaker & heated to evap. liquid. (*)

7. Transfer the ~~solution from the volumetric flask~~ solids to a clean vial labeled as C106-AQ-8B1. 0.4608 g of solids collected.

8. Allow C106-AQ-8B to dry, then weigh

   Wt. C106-AQ-8B = 99.7529 g (8a)

9. Determine the weight of the solids

   Wt. solids = 1a – 8a = 0.1014 g (9a) → this is almost hard to believe. It looked like a lot more than this

10. C106-AQ-8B1 will be submitted for analysis as prescribed in an ASR prepared by the cognizant scientist.

(*) Tried to dissolve in hot HNO_3 → did not completely dissolve.
Tried 1:1 HNO_3/HCl → still did not dissolve completely.
Evaporated acids by heating.

Protocol for Dissolving Sample C106-OH-8B

Purpose

The purpose of this protocol is to dissolve the magnetic solids stuck to the stir bar from the C-106 washing test so that they can be analyzed.

Instructions

1. Weigh C106-OH-8B

5/20/99 Wt. C106-OH-8B = 97.4508 g (1a)

2. Transfer stir bar with solids to a pyrex beaker

3. Add 10 mL of concentrated (16 M) HNO_3 (Ultrex-grade) to the beaker

4. Heat to near boiling to dissolve the solids — Solids did not all dissolve. Added enough conc. HCl to cover the stir bars & solids. Continued to heat. After a couple of hours almost all solids had dissolved.

5. Once all solids are dissolved, transfer the solution to a 25-mL volumetric flask. — Heated to drive off excess acid. → Cooked down to pasty mass.

6. Rinse the beaker with several small portions of deionized water, transferring the rinse liquid to the volumetric flask.

7. Fill the volumetric flask to the 25-mL mark with deionized water, then mix. — Added ~25 mL 0.1 M HCl. Heated/stirred → Solids did not all go back in solution. → Evaporated to dryness. Dried in oven at 105°C

8. Transfer the solution from the volumetric flask to a clean vial labeled as C106-OH-8B1. — Scraped solids into C106-OH-8B1.

9. Transfer the stir bar from the beaker back into C106-OH-8B and allow to dry

10. Weigh C106-OH-8B

Wt. C106-OH-8B = 95.5044 g (10a)

11. Determine the weight of the solids

Wt. solids = 1a – 10a = 1.9464 g (11a)

12. C106-OH-8B1 will be submitted for analysis as prescribed in an ASR prepared by the cognizant scientist.

6/24/99 We need the weight of the dried solids obtained after acid dissolution.

→ Weighed the solids → 1.8242 g

→ To this we will need to add the weight of aliquots used for fusion.

$1.8242 + \underbrace{0.1079}_{\text{KOH fusion}} + \underbrace{0.1017}_{Na_2O_2\text{ fusion}} = 2.0338\ g$

Protocol for Isolating Solid From C106-AQ-3

Purpose

The purpose of this protocol is to isolate the precipitated solids from solution C106-AQ-3 so that they can be analyzed.

Instructions

5/27/99
1. Label a 0.45-μm Nylon filter unit as C106-AQ-3* → white solids.

2. Swirl the solution in C106-AQ-3 to suspend the solids then filter in C106-AQ-3*

3. Disassemble the filter unit and put the cap on the reservoir with the clarified liquid.

4. Label a glass vial as C106-AQ-3Solid and weigh

   Wt. C106-AQ-3Solid = 21.9477 g

5. Using a spatula, transfer the filtered solids to C106-AQ-3Solid.

6. Allow solids to air-dry, then weigh

   Wt. C106-AQ-3Solid = 22.7130 g

   Wt. Dry Solid = 0.765

7. C106-AQ-3Solid will be submitted for analysis as prescribed in an ASR prepared by the cognizant scientist.

Note: There were a few solids left in C106-AQ-3. D.I water was added to see if the white solid was soluble → It was.

Appendix B. Raw Data

579716-8315

Analytical Chemistry Laboratory (ACL) Analytical Services Request (ASR)

(Cover Page ... information applicable to all samples in series)

Requested By: Gregg J. Lumetta (Print Name) | [signature] 5/28/99 (Signature/Date) | 376-6911 (Phone) | P7-25 (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.: W48486

Cost Estimate ($): ______

Protocol Requirement: X None __RCRA __CERCLA, or

Other (specify): ______

Hold Time Requirement: X None __RCRA __CERCLA, or

Other (specify): ______

TPA Support: X No, or

Milestone No.: ______

QA Plan: X MCS-033, or

Other ACL QA Plan (specify): ______

Additional QA Requirements: X No, or

Reference Doc.: ______

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

Sample Storage Requirements: X No __Refrigerate, or

Other (specify): ______

Date Sampled (optional): ______

Time Sampled (optional): ______

Matrix: X 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.: ASR 5275

PCBs Present: X No __Yes

Sample Disposition ... Check with GJL prior to disposal.

Untreated Sample(s): __Return X Dispose __Store, or

Reference Doc.: ______

Prep'd Sample(s): X Dispose __Return __Store, or

Reference Doc.: ______

Additional Instructions: X No, or

Reference Doc.: ______

Date Report Req'd: 7/9/99

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: Hot Cell 5/28/99

Time Delivered (optional): ______

Deliv. By (if known): ______

Received By: V. Hoopes

Resp. ACL Mgr.: M. W. Urie

Signature/Date: [signature]

Job Group (optional): ______

Sample Group (optional): ______

PNL Impact Level: __1 __2 __3

DQ Review Req'd: X No __Yes ACL Waste: X No __Yes

ASR Number: 5397 Revision: __Yes

ACL Numbers: (99-1881) - (99-1894)

MJS, JW, OTF

TABLE 1. Sample Matrix (Revised 6/3/99)

	Sample ID	Acid Digestion	KOH Fusion	Na_2O_2 Fusion	ICP/AES	IC (anions)	TOC	TIC	ICP-MS (^{99}Tc)	ICP-MS (Full)[(a)]	GEA	^{90}Sr	Total Alpha	Laser Fluorimetry (U)	^{241}Am/ $^{243+244}Cm$	^{238}Pu/ $^{239+240}Pu$	^{14}C	Tritium	CVAA Hg	Total CN	Ammonia
99-1881	C106-SOL-30-1	X			X	X	X	X	X		X	X	X	X							
1882	C106-SOL-30-2	X			X	X	X	X	X		X	X	X	X							
1883	C106-SOL-40-1	X			X	X	X	X	X		X	X	X	X							
1884	C106-SOL-40-2	X			X	X	X	X	X		X	X	X	X							
1885	C106-SOL-50-1	X			X	X	X	X	X		X	X	X	X							
1886	C106-SOL-50-2	X			X	X	X	X	X		X	X	X	X							
1887	C106-AQ-3Solid[(b)]				X	X															
1888	C106-AQ-8[(c)]		X	X	X	X	X	X		X	X	X	X	X	X	X	X	(d)	X	X	X
1889	C106-AQ-8B1		X	X	X					X	X	X	X	X	X	X					
1890	C106-AQ-9	X			X						X		X	X							
1891	C106-OH-3A	X			X						X		X	X							
1892	C106-OH-8[(c)]		X	X	X	X	X	X		X	X	X	X	X	X	X	X	(d)	X	X	X
1893	C106-OH-8B1		X	X	X					X	X	X	X	X	X	X					
99-1894	C106-OH-9	X			X						X		X	X							

(a) Includes Tc-99, I-129, Np-237, U-isotopic, Pu-isotopic

(b) This material is water-soluble. Weighed amounts can be dissolved in water for analyses, as appropriate.

(c) Analyses should be performed in duplicate.

(d) Although originally specified in the test specification, tritium analyses will not be performed because any tritium originally present would have been removed during previous drying cycles.

Special Instructions

1. Plastic sample vial should be used whenever practical.

2. All samples should be shaken to mix thoroughly before sub-sampling. (ESPECIALLY SOLIDS)

3. Visual checks for solids should be performed for the liquid samples.

4. Before transferring liquid samples to clean vials for removal from the SAL, gross weights should be obtained on the original samples. This will allow us to assess whether evaporation has occurred.

5. Dilutions should be based on volume. For dilutions made in the SAL, the weight of each aliquot used will be recorded along with the volume used.

6. Solid samples should be dried at 105°C immediately prior to processing.

7. If limited sample quantities require elimination of analyses or a compromise on detection limits, the following priorities are established for this sample set:

    1. ICP/AES
    2. Total Alpha
    3. GEA
    4. Sr-90
    5. TOC/TIC
    6. IC (anions)
    7. ICP-MS (Tc-99)
    8. ICP-MS (Full)
    9. ^{241}Am/ $^{243+244}$Cm
    10. ^{238}Pu/ $^{239+240}$Pu
    11. Laser Fluorimetry (U)
    12. CVAA Hg
    13. Total CN
    14. Ammonia
    15. C-14

ASR 5397: Additional Special Instructions

Recommended sample sizes and final volume & recommended distribution volumes

Sample Lab Number / Sample size to final vol.	Analysis	Vol.
Solutions: 99-1881 thru 99-1886		
Dilution 0.5 mL to 5 mL	TOC/TIC	2 mL
	IC	2 mL
Acid Dig. 2 mL to 20 mL	ICP	7 mL
	ICP/MS (Tc-99)	7 mL
	Rad (Note A)	5 mL
Solutions: 99-1890, 99-1891, 99-1894		
Acid Dig. 4 mL to 20 mL	ICP	7 mL
	ICP/MS (Note B)	8 mL
	Rad (Note C)	5 mL

Note A: Rad = GEA, Sr-90, Gross alpha, and U-laser
Note B: ICP/MS = Tc-99, I-129, Np-237, U-iso, Pu-iso
Note C: Rad = GEA, Gross alpha, and U-laser
Note D: Rad = GEA, Sr-90, Gross alpha, U-laser, Am/Cm, Pu-238, Pu- 239+240

Sample Lab Number / Sample size to final vol.	Analysis	Vol.
Dried Solids: 99-1887		
Dilutions 0.5 g to 10 mL	ICP	4 mL
	IC	3 mL
Dried Solids: 99-1889, 99-1893		
KOH Fusion 0.2 g to 20 mL	ICP	7 mL
Scale Flux to	ICP/MS (Note B)	8 mL
0.2 Sample Size	Rad (Note D)	5 mL
Na2O2 Fusion 0.2 g to 20 mL	ICP	7 mL
Scale Flux to		
0.2 Sample Size		
Dried Solids: 99-1888**, 99-1892 **		
KOH Fusion 0.15 g to 20 mL	ICP	7 mL
Scale Flux to	ICP/MS (Note B)	8 mL
0.15 Sample Size	Rad (Note D)	5 mL
Na2O2 Fusion 0.15 g to 20 mL	ICP	7 mL
Scale Flux to		
0.15 Sample Size		
Water Leach 0.25 g to 10 mL	IC	5 mL
	Ammonia	5 mL
Direct	TOC/TIC	0.1 g
	Mercury	0.1 g
	C-14	0.1 g
	Total CN	0.15 g

**** Perform all analysis in duplicate. More 99-1892 material available, sample sizes may be increased per RT Steele**

If prepared in the SAL: For TOC/TIC, C-14, Hg , and CN attempts should be made to pre-weigh samples in appropriate "containers" prior to distribution (e.g., CN in distillation tubes, TOC/TIC in weigh bottles, etc) Contact MJ Steele for TOC/TIC & C-14, JJ Wagner for Hg, and PK Berry for CN.

If dose levels prove to be too high for safe distribution of samples contact appropriate cognizant analyst for additional dilution levels: JJW for ICP & Hg, OT Farmer for ICP/MS, MJ Steele for IC & TOC/TIC & C-14, CZ Soderquist for Rad & Ammonia; PK Berry for CN

If insufficient quantities of sample are available for processing or distribution contanct MW Urie

	Table 4.2 Analytical Requirements for Filtrate, Washed Solids, and Wash Solutions		
	Analyte	Washed Solids Minimum Reportable Quantity (MRQ) µCi/gm	Filtrate, Wash Solutions Minimum Reportable Quantity (MRQ) µCi/ml
*	Cesium-137	6.0E-02	9.0E+00
*	Strontium-90	7.01E+01	1.5E-01
*	Technetium-99	6E+00 µgm/gm	1.5E-03
	Americium-241	1.2E-03	7.2E-04
	Europium-154	6.0E-02	2.0E-03
	Europium-155	6.0E-02	9.0E-02
*	Total Alpha	1.0E-03	2.3E-01
		µgm/gm	µgm/ml
*	Al	3.3E+02	7.5E+01
	Ag	9.0E+02	1.75E+01
	Ba	6.0E+02	7.8E+01
	Ca	1.8E+02	1.5E+02
	Cd	1.1E+01	7.5E+00
	Co	3.0E+00	3.0E+01
*	Cr	1.2E+02	1.5E+01
	Cu	1.8E+01	1.7E+01
*	Fe	1.4E+02	1.5E+02
	K	1.5E+03	7.5E+01
	La	6.0E+01	3.5E+01
	Mg	5.4E+02	1.5E+02
	Mn	3.0E+02	1.5E+02
	Mo	6.0E+00	9.0E+01
*	Na	1.5E+02	7.5E+01
*	Ni	1.6E+02	3.0E+01
	Pb	6.0E+02	3.0E+02
*	Si	3.0E+03	1.7E+02
	Ti	1.5E+02	1.7E+01
*	U	6.0E+02	6.0E+02
	Zn	6.0E+00	1.65E+01
	Zr	6.0E+02	N/A
*	TOC	6.0E+01	1.5E+03
	TIC	3.0E+01	1.5E+02
	Cl	2.3E+02	3.0E+00
	F	7.5E+03	1.5E+02
*	NO3	4.5E+02	3.0E+03
*	SO4	1.2E+03 (as S)	2.3E+03
*	PO4	6.0E+02 (as P)	2.5E+03

Mike:

Here are the MRQ's specified by BNFL. They don't cover all of the required analytes. For those not covered, do what you think is reasonable, without breaking the bank. Items with a "*" are what I view to be the big hitters.

Greg

Battelle Pacific Northwest National Laboratory
Radiochemical Process Group
Shielded Facility Operations Team

Shielded Analytical Laboratory
C-14

Tank C106 Core(s) N/A

Project Id: 29953 WP Number: W48486

TI/ASR Number: ASR 5397

Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt (g)	Sample Net Wt (g)	Spike Tare Wt (g)	Spike Gross Wt (g)	Spike Net Wt (g)	Spike Ident.
99-1888 C106-AQ-8	42.8714	42.9631	0.0917				
99-1892 C106-OH-8	42.6923	42.8130	0.1207				
99-1892-DUP C106-OH-8	46.0684	46.1971	0.1287				
99-1892-MS1 C106-OH-8	42.5311	42.6554	0.1245				
99-1892-MS2 C106-OH-8	44.8281	44.9428	0.1147				

M&TE: ✓ Cell 2 (360-06-01-016)

____ Cell 5 (360-06-01-019)

____ Denver (360-06-01-040)

____ Mettler AT201 (510-06-01-014)

Other ____

Analyst: [signature] Date: 7-2-99

Reviewer: [signature] Date: 7/2/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operations Team

Shielded Analytical Laboratory Bench Sheet

Client: GJ LUMETTA

WP Number: W48486

TI#/ASR: ASR 5397

Procedure: BENCH INSTRUCTION

C106 Zr FUSION DILUTIONS

SAMPLE IDENTIFICATION

PIPET PERFORMANCE CHECK DATA

SAMPLE PIPET DIW @ 25 °C	DILUENT PIPET
4.9285g $\bar{x}$ = 4.9413 g	5.0061g $\bar{x}$ = 5.0049g
4.9092g s = .0234	5.0029g s = .0035
4.9508g RSD = .47%	5.0098g RSD = .0007
4.9472g Vol = 4.9559 ml.	5.0052g Vol = 5.0197 ml.
4.9708g	5.0004g

SAMPLE ID	DILUENT TARE	DILUENT GROSS	DILUENT NET	SAMPLE TARE	SAMPLE GROSS	SAMPLE NET
99-1888-Zr-PB PROCESS BLANK	18.9430	23.9792	5.0362	34.0869	39.2457	5.1588
99-1888-Zr C106-AQ-8	23.9792	29.0398	5.0606	39.2444	44.4304	5.1860
99-1888-Zr-DUP C106-AQ-8	29.0398	34.1072	5.0674	44.4301	49.6177	5.1876
99-1889-Zr C106-AQ-8B1	19.1188	24.1764	5.0576	34.2467	39.4504	5.2037
99-1892-Zr C106-OH-8	24.1764	29.2299	5.0535	39.4495	44.6395	5.1900
99-1892-Zr-DUP C106-OH-8	29.2299	34.2823	5.0524	44.6384	49.8423	5.2039
99-1893-Zr C106-OH-8B1	15.9148	20.9682	5.0534	26.0020	31.3682	5.3062
SRM 2710-Zr LCS/99-1888/Zr	20.9682	26.0185	5.0503	31.3076	36.4907	5.1831

M&TE: X Cell 2 (360-06-01-016) Mettler AE160 Balance Other ______

____ Cell 5 (360-06-01-019) Sartorius LP4200S Balance

____ Bench (510-06-01-014) Mettler AT201 Balance

X Bench (360-06-01-040) Denver A160 Balance

Analyst: [signature] Date: 6-14-99 Reviewer: [signature] Date: 6/14/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operations Team

Shielded Analytical Laboratory
Bench Sheet

Client: GJ LUMETTA

WP Number: W48486

TI#/ASR: ASR 5397

Procedure: BENCH INSTRUCTION

AQUEOUS SAMPLE DOSE RATES

SAMPLE IDENTIFICATION

Sample Ident.	Open Window @ Contact	Closed Window @ Contact	Open Window @ 6"	Closed Window @ 6"	Open Window @ 12"	Closed Window @ 12"
	CP Readings					
C106-SOL-50-1	700/½"	28	45	3.5	14	1
C106-AQ-9	50 "	4.5	8	1	4	.6
C106-OH-3A	48 "	18	7	2.5	4	1.5
* All Mr/Hr uncorrected						

APPROXIMATE SAMPLE VOLUMES IN mL:

C106-SOL-50-1 2-3 ml
C106-AQ-9 8 solids on bottom
C106-OH-3A 14

M&TE: ____ Cell 2 (360-06-01-016) Mettler AE160 Balance Other ____

____ Cell 5 (360-06-01-019) Sartorius LP4200S Balance

____ Bench (510-06-01-014) Mettler AT201 Balance

____ Bench (360-06-01-040) Denver A160 Balance

Analyst: [signature] Date: 6-3-99 Reviewer: Rick L. Steele Date: 6/9/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operations Team

Shielded Analytical Laboratory
Bench Sheet

Client: GJ LUMETTA

WP Number: W48486

TI#/ASR: ASR 5397

Procedure: BENCH INSTRUCTION

AQUEOUS SAMPLE PRE-PROCESSING WEIGHT DATA

SAMPLE IDENTIFICATION

ACL No.	Sample Identification	Vial Gross Weight (g)
99-1881	C106-SOL-30-1	10.5470
99-1882	C106-SOL-30-2	9.7723
99-1883	C106-SOL-40-1	10.3140
99-1884	C106-SOL-40-2	10.1434
99-1885	C106-SOL-50-1	8.3734 * Looks like some has leaked out.
99-1886	C106-SOL-50-2	10.1754
99-1887	C106-AQ-3SOLID	22.6114
99-1890	C106-AQ-9	15.8656
99-1891	C106-OH-3A	17.7312
99-1894	C106-OH-9	23.4407

M&TE: ✓ Cell 2 (360-06-01-016) Mettler AE160 Balance Other ______

_____ Cell 5 (360-06-01-019) Sartorius LP4200S Balance

_____ Bench (510-06-01-014) Mettler AT201 Balance

_____ Bench (360-06-01-040) Denver A160 Balance

Analyst: [signature] Date: 6-4-99 Reviewer: [signature] Date: 6/4/99

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

PNL-ALO-114

Solubilization of Metals from Solids Using a Na2O2-NaOH Fusion

Client name: GJ LUMETTA
Work Auth. Doc (WAD): ASR 5397
Tank/Core/Project: TANK C-106
Special instructions

Work package number: W48486
Project number: 29953
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Crucible Identifier	Crucible weight (g)	Crucible + sample weight (g)	Sample weight (g)	Spike added Vol. (ml)	Spike added Weight. (g)	Final solution Volume (ml)	Process Factor (1)
1	99-1888-Zr-PB	PROCESS BLANK	B						50	
2	99-1888-Zr	C106-AQ-8	1	34.3016	34.4081	0.1065				469.48
3	99-1888-Zr-DUP	C106-AQ-8	2	35.2822	35.3867	0.1045				478.47
4	99-1889-Zr	C106-AQ-8B1	3	34.8851	34.9766	0.0915				546.45
5	99-1892-Zr	C106-OH-8	4	34.5004	34.6052	0.1048				477.10
6	99-1892-Zr-DUP	C106-OH-8	5	31.5864	31.6854	0.0990				505.05
7	99-1893-Zr	C106-OH-8B1	6	35.1401	35.2418	0.1017				491.64
8	SRM 2710-Zr	LCS/99-1888/Zr	7	34.5435	34.6330	0.0895				558.66
9										
10										
11										
12										
13										
14										

Analyst's sample preparation comments: DOSE RATE:

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
HCl volume added (ml): 1
Solution heated (yes/no): no
Sample filtered (yes/no): no.

(1) Process factor = Final volume (ml) / [Crucible & sample weight (g) - Crucible weight (g)]

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: [signature] 6-14-99 Reviewer/Date: [signature] 6/14/99

Pacific Nor. National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR 5397 PROJECT NO.: 29953 WBS NO.: SAMPLE TYPE: SOLIDS

ISSUED BY: RT STEELE DATE: 6/7/99 PREP TYPE: Ni/KOH FUSION (ALO-115)

ANALYST: [signature] DATE: 6-9-99 CHAIN OF CUSTODY RQD: NO

REVIEW: Rick T. Steele DATE: 6/10/99 QA PLAN: MCS-033 IMPACT LEVEL:

CLIENT: GJ LUMETTA CORE ID: N/A TANK ID: C-106

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(1) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W48486	99-1888-PB	PROCESS BLANK	SEE BELOW				50						
	99-1888	C106-AQ-8		0.1083									
	99-1888-DUP	C106-AQ-8		0.1131									
	99-1889	C106-AQ-8B1		0.1126									
	99-1892	C106-OH-8		0.1129									
	99-1892-DUP	C106-OH-8		0.1013									
	99-1893	C106-OH-8B1		0.1079									
			GEA										
			Sr-90										
			GROSS ALPHA										
			U LASER										
			Am, Cm										
			Pu/AEA										

(1) Specific Gravity of ORIGINAL material. **SEE ASR 5397 TABLE 4.2 FOR MRQs**

Pacific Nor[illegible] National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR 5397 PROJECT NO.: 29953 WBS NO.: ______ SAMPLE TYPE: SOLIDS

ISSUED BY: RT STEELE DATE: 6/7/99 PREP TYPE: Zr/Na2O2 FUSION (ALO-114)

ANALYST: [signature] DATE: 6-14-99 CHAIN OF CUSTODY RQD: NO

REVIEW: [signature] DATE: 6/14/99 QA PLAN: MCS-033 IMPACT LEVEL: ______

CLIENT: GJ LUMETTA CORE ID: N/A TANK ID: C-106

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(1) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W48486	99-1888-Zr-PB	PROCESS BLANK	ICP				50			2.01X	2% HNO3	4.9559	
	99-1888-Zr	C106-AQ-8		0.1065								5.0197	
	99-1888-Zr-DUP	C106-AQ-8		0.1045									
	99-1889-Zr	C106-AQ-8B1		0.0915									
	99-1892-Zr	C106-OH-8		0.1048									
	99-1892-Zr-DUP	C106-OH-8		0.0990									
	99-1893-Zr	C106-OH-8B1		0.1017									
	SRM 2710-Zr	LCS/99-1888/Zr		0.0895									
	99-1888-DB-Zr	DILUENT BLANK											
						D.I.	4.9559 +		5.0197 / 4.9559	= 2.01X			

(1) Specific Gravity of ORIGINAL material. **SEE ASR 5397 TABLE 4.2 FOR MRQs**

PAGE 1 OF 1

Battelle Pacific Northwest National Laboratory
Radiochemical Process Group
Shielded Facility Operations Team

Shielded Analytical Laboratory
WATER LEACH

Tank C106 Core(s) -- N/A

Project Id: 29953 WP Number: W48486

TI/ASR Number: ASR 5397

Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt (g)	Sample Net Wt (g)	DIW Tare Wt (g)	DIW Gross Wt (g)	DIW Net Wt (g)	Spike Volume
99-1888-PB PROCESS BLK	16.8176	16.8176	0	16.8176	25.0317	8.2141	
99-1888 C106-AQ-8	15.2838	15.5407	0.2569	15.5407	25.3000	9.7593	
99-1888-DUP C106-AQ-8	15.2427	15.5037	0.2610	15.5037	25.3444	9.8407	
99-1892 C106-OH-8	15.2463	15.6145	0.3682	15.6145	26.3261	10.7116	
99-1892-DUP C106-OH-8	15.2486	15.6100	0.3614	15.6100	25.9226	10.3126	

Spike Id: ______

M&TE: X Cell 2 (360-06-01-016) Other ______

___ Cell 5 (360-06-01-019)

___ Denver (360-06-01-040)

___ Mettler AT201 (510-06-01-014)

Analyst: [signature] Date: 6-14-99 Reviewer: [signature] Date: 6/15/99

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR 5397 PROJECT NO.: 29953 WBS NO.: SAMPLE TYPE: SOLIDS

ISSUED BY: RT STEELE DATE: 6/7/99 PREP TYPE: Water Leach (ALO-103)

ANALYST: [signature] DATE: 6-15-99 CHAIN OF CUSTODY RQD: NO

REVIEW: [signature] DATE: 6/15/99 QA PLAN: MCS-033 IMPACT LEVEL:

CLIENT: GJ LUMETTA CORE ID: N/A TANK ID: C-106

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(1) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W48486	99-1888-PB	PROCESS BLANK	IC			8.3141							
	99-1888	C106-AQ-8		0.2569		9.7593							
	99-1888-DUP	C106-AQ-8		0.2610		9.8407							
	99-1892	C106-OH-8		0.3682		10.7116							
	99-1892-DUP	C106-OH-8		0.3614		10.3126							

(1) Specific Gravity of ORIGINAL material. **SEE ASR 5397 TABLE 4.2 FOR MRQs**

Pacific Nor... National Laboratory
Radiochemical Processing Laboratory
Shielded Facility Operation Team

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: ASR 5397 PROJECT NO.: 29953 WBS NO.: ______ SAMPLE TYPE: SOLIDS

ISSUED BY: RT STEELE DATE: 6/7/99 PREP TYPE: Water Leach (ALO-103)

ANALYST: [signature] DATE: 6-15-99. CHAIN OF CUSTODY RQD: NO

REVIEW: [signature] DATE: 6/15/99 QA PLAN: MCS-033 IMPACT LEVEL: ______

CLIENT: GJ LUMETTA CORE ID: N/A TANK ID: C-106

WORK PACKAGE NUMBER	ALO NUMBER	SAMPLE IDENTIFICATION	ANALYTE OR ANALYSIS	SAMPLE WT	Sp.G.(1) (g/mL)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
W48486	99-1888-PB	PROCESS BLANK	NH3-ISE			8.2141							
	99-1888	C106-AQ-8		0.2569		9.7593							
	99-1888-DUP	C106-AQ-8		0.2610		9.8467	7 RTS 6-15-99						
	99-1892	C106-OH-8		0.3682		10.7116							
	99-1892-DUP	C106-OH-8		0.3614		10.3126							

(1) Specific Gravity of ORIGINAL material.

Battelle Pacific Northwest National Laboratory
Radiochemical Process Group
Shielded Facility Operations Team

Shielded Analytical Laboratory
TIC/TOC

Tank C106 Core(s) N/A

Project Id: 29953 WP Number: W48486

TI/ASR Number: ASR 5397

Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt (g)	Sample Net Wt (g)	Spike Tare Wt (g)	Spike Gross Wt (g)	Spike Net Wt (g)	Spike Ident.
99-1888 C106-AQ-8	31.7973	31.8586	0.0613				
99-1888-DUP C106-AQ-8	43.3842	43.4630	0.0788				
~~-1888-MS~~ ~~06-AQ-8~~	RGS 6/16/99						
99-1892 C106-OH-8	42.8662	42.8740	.0078				
99-1892-DUP C106-OH-8	33.3527	33.3565	.0038				
99-1892-MS C106-OH-8	34.8528	34.8588	.0060				

M&TE: X Cell 2 (360-06-01-016) Other ______

_____ Cell 5 (360-06-01-019)

_____ Denver (360-06-01-040)

_____ Mettler AT201 (510-06-01-014)

Analyst: [signature] Date: 6-17-99 Reviewer: [signature] Date: 6/18/99

Shielded Analytical Laboratory
Cyanide Analysis

Tank C106 Core(s) N/A

Project Id: 29953 WP Number: W48486

TI/ASR Number: ASR 5397

Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt (g)	Sample Net Wt (g)	Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt. (g)	Sample Net Wt (g)
99-1888 C106-AQ-8	14.5949	14.7484	0.1535	99-1892-MSD C106-OH-8	16.0588	16.2178	0.1590
99-1888-DUP C106-AQ-8	14.7484	14.9051	0.1567				
-1892 C106-OH-8	14.9051	15.0580	0.1529				
99-1892-DUP C106-OH-8	15.7678	15.9174	0.1496				
99-1892-MS C106-OH-8	15.9174	16.0589	0.1415				

M&TE: X Cell 2 (360-06-01-016) Other ______

____ Cell 5 (360-06-01-019)

____ Denver (360-06-01-040)

____ Mettler AT201 (510-06-01-014)

Analyst: Vaughn Hoopes Date: 6-18-99

Reviewer: [signature] Date: 6/18/99

Battelle Pacific Northwest National Laboratory
Radiochemical Process Group
Shielded Facility Operations Team

Shielded Analytical Laboratory
Mercury Digestion

Tank C106 Core(s) N/A

Project Id: 29953 WP Number: W48486

TI/ASR Number: ASR 5397

Sample Ident.	Sample Tare Wt (g)	Sample Gross Wt (g)	Sample Net Wt (g)	Spike Tare Wt (g)	Spike Gross Wt (g)	Spike Net Wt (g)	Spike Ident.
99-1888 C106-AQ-8	27.4612	27.5641	0.1029				
99-1888-DUP C106-AQ-8	27.3695	27.4571	0.0876				
)-1892 :106-OH-8	27.2960	27.4213	0.1253				
99-1892-DUP C106-OH-8	26.8565	27.0019	0.1454				
99-1892-MS C106-OH-8	28.3706	28.4688	0.0982				

M&TE: X Cell 2 (360-06-01-016)

___ Cell 5 (360-06-01-019)

___ Denver (360-06-01-040)

___ Mettler AT201 (510-06-01-014)

Other ___

.ialyst: [signature] Date: 6-17-99 Reviewer: [signature] Date: 6/18/99

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

EXHIBIT I
PAGE 1 of 1

PNL-ALO-128 Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: Gregg Lumetta
Work Auth. Doc (WAD): A-SRO 5397
Tank/Core/Project:
Special Instructions:

Work package number: W48486
Project number: 29953
PNL QA plan: MCS-033
PNL impact level:
Prep. lab (SAL/SRPL/other):
Preparation batch number:

	ACL Sample ID	~~ACL order number or Client sample ID~~ (LPD 6-7-99)	Vial Identifier	* Sample Volume (ml)	~~Spike~~ Volume (ml)	~~Spike added~~ Sample Weight (g)	Final solution Volume (ml)	Process Factor (1)	Visible Solids
1	99-1881	25.5776 / 27.6677	1	2x1ml.		2.0901 g.	20ml		Slight Solids
2	1882	25.3259 / 27.4228	2	↓		2.0969 g.	↓		↓
3	1883	25.4530 / 27.5614	3	↓		2.1084 g.	↓		↓
4	1884	25.4880 / 27.6004	4	↓		2.1124 g.	↓		↓
5	1885	25.4815 / 26.5512	5	1ml.		1.0697 g.	↓		Moderate Solids
6	1886	25.4983 / 27.6142	6	2x1ml.		2.1159 g.	↓		↓
7	1890	25.3597 / 29.5595	7	4x1ml		4.1998 g.	↓		↓
8	1891	25.4301 / 29.9661	8	↓		4.5360 g.	↓		No Solids
9	Dupe-1891	25.3131 / 29.8406	9	↓		4.5275 g.	↓		↓
10	MS-1891	25.3877 / 29.9085	10	↓	0.4ml. Part A 0.4ml. Part B	4.5208 g.	↓		↓
11	99-1894	25.2372 / 29.4430	11	↓		4.2058 g.	↓		Moderate Solids
12	BS-1881	25.5310 —	12	2ml of H_2O	0.4ml. Part A 0.4ml. Part B	—	↓		—
13	Prep Blank	25.3482 —	13	2ml of H_2O		—	↓		—
14									

Analyst's sample preparation comments: * All samples were aliquoted using a 1ml. pipette for volumes of 1ml, 2mls, and 4mls. Spike solutions were scaled down and 0.4ml of Parts A+B were used. Acid volumes were also scaled down for final dilution volumes on 20mls. 0.8ml of each conc. HNO_3 + HCL were used. All samples were clear at completion.

Pipette # 112498 @ 22°c
0.9944 0.9918 } used for
0.9920 } Sample
0.9941 } aliquots
0.9941

Spike source: Standards Lab
PNL spike ID number: 9903055 HCl & A
Anal. balance M&TE: 360-06-01-026
Sample filtered (yes/no):

(1) Process factor = Final volume (ml) / Sample volume (ml)

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: Lori P. Darnell 6-8-99
Reviewer/Date: [illegible signature] 6-9-99

Rev. 2.0 7-28-95 JMR

Radiochemistry Bench Sheet

Client: Gregg Lumetta
Work Order #: W 48486
ASR: 5397

Balance: 360-06-01-037

Procedure: Sub-Sample of Acid Digest for ICP + ICP-MS

Sample ID	Dilutions and Aliquot Sizes	Pipet ID Numbers / Pipet Checks	
99-1881	7 mls	H30754	7.0417
1882		@ 7ml	7.1010
1883		24°C	7.1298
1884			7.1462
1885			7.0960
1886			
1890			
1891			
Dupe-1891			
MS-1891			
99-1894			
BS-1881			
Prep Blank			
*	See attached Acid Digest bench sheet for dilution weighs / Volumes		

Spike, Tracer ID
Isotope
Concentration
Ref Date

Analyst/Date: Lori P. Darnell

Reviewer/Date: [illegible] 6-15-99

Radiochemistry Bench Sheet

Client: Gregg Lumetta
Work Order #: W48486
ASR: 5397

Balance: 360-06-01-026

Procedure: PNL-ALO-103

Pipet ID Numbers
Pipet Checks

Sample ID	Dilutions and Aliquot Sizes		
99-1887	0.5147 gms. → 10 mls	L05722N	10.0454
Prep-Blank	—	@ 10 ml	10.0736
		22°C	10.0369
			10.0696
	The sample material was weighed into a plastic		10.0809

LSC Vial and contacted with 10 mls of DI-H_2O. The solution was ultrasonicated for 1 hr then filtered to 0.45 μm and split for IC & ICP analysis.

Spike, Tracer ID
Isotope
Concentration
Ref Date

Analyst/Date: Lori P. Darnell 6-7-99

Reviewer/Date: LR Greenwood 6-9-99

Radiochemistry Bench Sheet

Client: Gregg Lumetta

U-laser

Work Order #:

ASR:

Balance: 360-06-01-026

Procedure: PNL-ALO-4014

Sample ID	Dilutions and Aliquot Sizes	Pipet ID Numbers / Pipet Checks	
99-1888-PB	0.1ml → 10ml	#47286	.0997
1888		0.1ml	.0996 $\bar{X}$ = 0.0994g ± 0.2%, 1σ
1888-Dup		@23°C	.0992 = 0.0996ml
1889			.0994
1892			.0993
1892-Dup		#H30860	10.0118
1893		@10ml	10.1467 $\bar{X}$ = 10.1166g ± 0.8%, 1σ
	2m	23°C	10.1956 = 10.13ml
	Hotcell KOH Fusion Sample material; HNO_3		10.0590
			10.1698

Spike, Tracer ID:

Isotope:

Concentration:

Ref Date:

Analyst/Date: Lori P. Darnell 6-21-99 Reviewer/Date: C. Soderquist 6-22-99

Radiochemistry Bench Sheet

Client: Lumelta

GEA

Work Order #: W48486

ASR: 5397

Balance: 360-06-01-037

Procedure: PNL-ALO-450

Sample ID	Dilutions and Aliquot Sizes		Pipet ID Numbers / Pipet Checks
99-1888-PB	10 mls	H30754	10.0399
1888		@ 10 ml	10.1240
1888-Dup		25°C	10.1235
1889			10.1212
1892			10.1197
1892-Dup			
1893			

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date: Lori P. Darnell 6-14-99

Reviewer/Date: L R Greenwood 6-16-99

Radiochemistry Bench Sheet

Client: Gregg Lumetta

Work Order #: W484186

ASR: 5397

GEA

Balance: 360-06-01-037

Procedure: PNL-ALO-450

Sample ID	Dilutions and Aliquot Sizes	Pipet ID Numbers	Pipet Checks
99-1881	2 mls	#H30754	2.0070
1882	↓	@ 2ml	2.0234
1883		24°C	2.0324
1884			2.0384
1885			2.0496
1886			
1890			
1891			
Dupe-1891			
MS-1891			
99-1894			
Prep Blank	↓		
*	See acid digestion PNL-ALO-128 prep sheet for sample weights + dilutions		

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date: Lori P. Darnell 6-9-99

Reviewer/Date: LR Greenwood 6-15-99

Radiochemistry Bench Sheet

Gross Alpha

Client: Luonetta

Work Order #: W48486

ASR: 5397

Balance: 360-06-01-037

Procedure: PNL-ALO-420

Sample ID	Dilutions and Aliquot Sizes	Pipet ID Numbers / Pipet Checks		
99-1888-PB	3 ml → (dryness) 3 mls — 1 ml → 9 ml — 0.1 ml	# H30754	2.9904	8.9819
1888	↓	@ 3+9 ml	2.9999	9.0134
1888-Dup	↓	25°C	3.0081	9.0058
1889	↓		3.0193	8.9952
1892	↓		3.0305	9.0237
1892-Dup	↓	#288618	0.9999	
1893	↓	@ 1 ml	1.0021	
1893-Rep	↓	25°C	0.9983	
MS-1893	↓		0.9972	
BS-1888	0.1 ml Pu^{239}		1.0059	
BL-1888	0.1 ml 2M HNO_3	#120737	0.0992	
		@ 0.1 ml	0.0995	
		25°C	0.0996	
			0.0995	
			0.0998	

Spike, Tracer ID: W-215-5

Isotope: Pu^{239}

Concentration: 500.6 ± 7.4 dpm/ml

Ref Date: 5-19-97

0.1 ml Used

Analyst/Date: Lori P. Darnell 6-15-99

Reviewer/Date: LR Greenwood 6-16-99

Radiochemistry Bench Sheet

Gross Alpha

Client: Wagner / Lummetta
Work Order #: K88433 W48486
ASR: 5404 5397

Balance: 360-06-01-037

Procedure: PNL-ALO-420

Sample ID	Dilutions and Aliquot Sizes	Pipet ID Numbers / Pipet Checks		
99-1927	3 mls → 3 mls — 1 ml → 9 ml — 0.1 ml	H30754	3.0082	9.0491
	dryness	@ 3+9 mls	3.0162	9.1449
99-1881	↓	24°C	3.0032	9.1386
1882	↓		2.9969	9.1150
1883	↓		2.9930	9.1611
1884	↓	120737	0.0990	
1885	↓	@ 0.1 ml	0.0992	
1886	↓	24°C	0.1002	
1890	↓		0.0991	
1891	↓		0.0990	
Dupe-1891	↓	288618	1.0027	
1894	↓	@ 1 ml	1.0008	
PB-1891	↓	24°C	1.0017	
BL-1891	0.1 ml 2M HNO_3		0.9995	
BS-1891	0.1 ml Pu239		0.9991	
Rep-1894	3 mls → 3 mls — 1 ml → 9 ml — 0.1 ml			
MS-1894	dryness ↓			

✗ See attached acid digest paperwork for calculations

Spike, Tracer ID: W-215-5
Isotope: Pu239
Concentration: 500.6 ± 7.4 dpm/ml
Ref Date: 5-19-97

} 0.1 ml used

Analyst/Date: Lori P. Darnell 6-11-99
Reviewer/Date: LR Greenwood 6-15-99

Radiochemistry Bench Sheet

Client: Wagner / Lummetta — Gross Alpha
Work Order #: K88433 W48486
ASR: 5404 5397

These dilutions were used for Uranium Analysis

CSoderquist 6-22-99

Balance: 360-06-01-037

Procedure: PNL-ALO-420

Pipet ID Numbers: $\bar{x}$ = 3.0035g ± 0.3%, 1σ = 3.007ml

Pipet Checks

Sample ID	Dilutions and Aliquot Sizes	Pipet ID	Pipet Checks	
99-1927	3 mls → 3 mls (dryness) — 1 ml ↔ 9 ml − 0.1 ml	H30754	3.0082	9.0491
	↓	@ 3+9 mls	3.0162	9.1449 $\bar{x}$ = 9.1217g
99-1881	↓	24°C	3.0032	9.1386 ± 0.5% 1σ
1882	↓		2.9969	9.1150
1883	↓		2.9930	9.1611 = 9.134 ml
1884	↓	120737	0.0990	
1885	↓	@ 0.1 ml	0.0992	
1886	↓	24°C	0.1002	
1890	↓		0.0991	
1891	↓		0.0990	
Dupe-1891	↓	288618	1.0027	
1894	↓	@ 1 ml	1.0008	$\bar{x}$ = 1.0008g
PB-1891	↓	24°C	1.0017	± 0.2%, 1σ
BL-1891	0.1 ml 2M HNO_3		0.9995	= 1.002 ml
BS-1891	0.1 ml Pu^{239}		0.9991	
Rep-1894	3 mls → 3 mls (dryness) — 1 ml ↔ 9 ml − 0.1 ml			
MS-1894	↓			

* See attached acid digest paperwork for calculations

Spike, Tracer ID: W-215-5
Isotope: Pu^{239}
Concentration: 500.6 ± 7.4 dpm/ml
Ref Date: 5-19-97
0.1 ml Used

Analyst/Date: Lori P. Darnell 6-11-99
Reviewer/Date: LRGreenwood 6-22-99

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

99-1881
8/11/1999

Client : Lumetta

Cognizant Scientist: LRGreenwood Date : 8/11/99

Concur : C. Soderquist Date : 8-11-99

Measured Activities (uCi/ml)

ALO ID Client ID	Alpha ± 1s	Co-60 ± 1s	Cs-137 ± 1s	Eu-154 ± 1s	Eu-155 ± 1s	Am-241 ± 1s
99-1881 C106-SOL-30-1	2.25E-3 7%	8.30E-4 10%	4.78E+0 2%	<4E-4	<6E-3	<6E-3
99-1882 C106-SOL-30-2	1.50E-3 8%	7.64E-4 10%	4.84E+0 2%	<6E-4	<6E-3	<6E-3
99-1883 C106-SOL-40-1	1.70E-3 7%	1.08E-3 8%	5.60E+0 2%	<6E-4	<7E-3	<7E-3
99-1884 C106-SOL-40-2	3.09E-3 6%	1.23E-3 7%	5.13E+0 2%	<4E-4	<6E-3	<6E-3
99-1885 C106-SOL-50-1	4.87E-3 6%	1.20E-3 12%	6.26E+0 2%	<1E-3	<1E-2	<1E-2
99-1886 C106-SOL-50-2	3.92E-3 5%	1.22E-3 8%	6.31E+0 2%	<7E-4	<7E-3	<7E-3
99-1890 C106-AQ-9	1.03E-3 7%	1.47E-3 5%	3.58E+0 2%	1.63E-3 9%	<4E-3	<4E-3
PB-1891 Process Blank	<8E-5	<5E-5	<5E-5	<2E-4	<2E-4	<2E-4
99-1891 C106-OH-3A	9.16E-3 2%	9.54E-4 10%	2.04E+1 2%	<8E-4	<9E-3	<9E-3
99-1891 DUP C106-OH-3A	8.56E-3 2%	1.04E-3 8%	1.98E+1 2%	<7E-4	<9E-3	<9E-3
RPD	7%	9%	3%			
99-1891 Rep		1.00E-3 8%	2.02E+1 2%	<7E-4	<9E-3	<9E-3
99-1894 C106-OH-9	1.31E-3 6%	6.00E-4 8%	1.90E+0 2%	<2E-4	<3E-3	<3E-3
99-1894 REP C106-OH-9	1.40E-3 6%					
RPD	7%					
Lab Blank	<2E-6					
Matrix Spike	88%					
Reagent Spike	107%					

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

99-1881
8/11/1999

Client : Lumetta

Cognizant Scientist: SKGreenwood Date : 8/11/99

Concur : C Soderquist Date : 8-11-99

Measured Activities (uCi/g)

ALO ID Client ID	Alpha ± 1s	Co-60 ± 1s	Cs-137 ± 1s	Eu-154 ± 1s	Eu-155 ± 1s	Am-241 ± 1s	Vol, ml	Wt, g
99-1881 C106-SOL-30-1	2.15E-3 7%	7.94E-4 10%	4.57E+0 2%	<4E-4	<6E-3	<6E-3	2.00	2.0901
99-1882 C106-SOL-30-2	1.43E-3 8%	7.28E-4 21%	4.62E+0 2%	<6E-4	<6E-3	<6E-3	2.00	2.0969
99-1883 C106-SOL-40-1	1.61E-3 7%	1.02E-3 9%	5.31E+0 2%	<6E-4	<7E-3	<7E-3	2.00	2.1084
99-1884 C106-SOL-40-2	2.93E-3 6%	1.16E-3 7%	4.86E+0 2%	<4E-4	<6E-3	<6E-3	2.00	2.1124
99-1885 C106-SOL-50-1	4.55E-3 6%	1.12E-3 12%	5.85E+0 2%	<9E-4	<9E-3	<9E-3	1.00	1.0697
99-1886 C106-SOL-50-2	3.71E-3 5%	1.15E-3 8%	5.96E+0 2%	<7E-4	<7E-3	<7E-3	2.00	2.1159
99-1890 C106-AQ-9	9.81E-4 7%	1.40E-3 5%	3.41E+0 2%	1.55E-3 9%	<4E-3	<4E-3	4.00	4.1998
PB-1891 Process Blank	<7E-5	<4E-5	<4E-5	<2E-4	<2E-4	<2E-4	4.00	4.5360
99-1891 C106-OH-3A	8.08E-3 2%	8.41E-4 10%	1.80E+1 2%	<7E-4	<8E-3	<8E-3	4.00	4.5360
99-1891 DUP C106-OH-3A	7.56E-3 2%	9.19E-4 8%	1.75E+1 2%	<6E-4	<8E-3	<8E-3	4.00	4.5275
RPD	7%	9%	3%					
99-1891 Rep		8.85E-4 8%	1.79E+1 2%	<6E-4	<8E-3	<8E-3	4.00	4.5208
99-1894 C106-OH-9	1.25E-3 6%	5.71E-4 8%	1.81E+0 2%	<2E-4	<3E-3	<3E-3	4.00	4.2058
99-1894 REP C106-OH-9	1.33E-3 6%						4.00	4.2058
RPD	7%							
Lab Blank	<2E-6							
Matrix Spike	88%							
Reagent Spike	107%							

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

99-1881
7/9/99

Client : Lumetta

Cognizant Scientist: LR Greenwood Date : 7-9-99

Concur : C. Soderquist Date : 7-9-99

Measured Activities

ALO ID Client ID	Uranium µg/mL ± 1s	Sr-90 µCi/mL ± 1s	Vol, ml	Wt, g
99-1881PB Process Blank	3.89E-1 2%	<2E-4	2.00	2.0901
99-1881 C106-SOL-30-1	2.45E+1 4%	9.87E-2 3%	2.00	2.0901
99-1882 C106-SOL-30-2	2.83E+1 4%	6.70E-2 8%	2.00	2.0969
99-1883 C106-SOL-40-1	3.09E+1 4%	1.05E-1 3%	2.00	2.1084
99-1884 C106-SOL-40-2	2.72E+1 4%	1.51E-1 4%	2.00	2.1124
99-1885 C106-SOL-50-1	3.50E+1 4%	2.26E-1 3%	1.00	1.0697
99-1886 C106-SOL-50-2	3.26E+1 4%	1.15E-1 3%	2.00	2.1159
99-1890 C106-AQ-9	9.86E+0 4%		4.00	4.1998
99-1891 C106-OH-3A	4.03E+1 4%		4.00	4.5360
99-1891 DUP C106-OH-3A	3.97E+1 4%		4.00	4.5275
99-1894 C106-OH-9	5.61E-1 2%		4.00	4.2058
99-1894 REP C106-OH-9			4.00	4.2058
Lab Blank	<2.00E-5	<3E-5		
Matrix Spike		99%		
Reagent Spike		95%		

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

99-1881
7/9/99

Client : Lumetta

Cognizant Scientist: JR Greenwood Date : 7-9-99

Concur : C Soderquist Date : 7-9-99

Measured Activities

ALO ID Client ID	Uranium µg/g ± 1s	Sr-90 µCi/g ± 1s
99-1881PB Process Blank	3.72E-1 2%	<2E-4
99-1881 C106-SOL-30-1	2.34E+1 4%	9.44E-2 3%
99-1882 C106-SOL-30-2	2.70E+1 4%	6.39E-2 8%
99-1883 C106-SOL-40-1	2.93E+1 4%	1.00E-1 3%
99-1884 C106-SOL-40-2	2.58E+1 4%	1.43E-1 4%
99-1885 C106-SOL-50-1	3.27E+1 4%	2.11E-1 3%
99-1886 C106-SOL-50-2	3.08E+1 4%	1.09E-1 3%
99-1890 C106-AQ-9	9.39E+0 4%	
99-1891 C106-OH-3A	3.55E+1 4%	
99-1891 DUP C106-OH-3A	3.51E+1 4%	
99-1894 C106-OH-9	5.34E-1 2%	
99-1894 REP C106-OH-9		
Lab Blank	<2E-5	<1E-2
Matrix Spike		99%
Reagent Spike		95%

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

99-1888
7/9/99

Client : Lumetta

Cognizant Scientist: LR Greenwood Date : 7/9/99

Concur : C. Soderquist Date : 7-12-99

Measured Activities (uCi/g)

ALO ID Client ID	Alpha* Error %	Am-241 Error %	Cm-243/ Cm-244 Error %	Cm-242 Error %	Pu-239/ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Alpha* Sum
99-1888PB Process Blank	1.20E-2 24%	7.20E-4 8%	5.43E-4 9%	<7.E-6	7.28E-4 8%	8.72E-4 7%	<2.E-5	2.86E-3
99-1888 C106-AQ-8	4.89E+0 2%	2.42E+0 5%	5.18E-2 10%	5.83E-3 27%	2.84E+0 4%	5.19E-1 4%	<1.E-3	5.84E+0
99-1888 DUP C106-AQ-8	4.67E+0 2%	2.22E+0 5%	6.15E-2 ~10%	3.34E-3 36%	2.72E+0 4%	4.96E-1 4%	<2.E-3	5.50E+0
RPD	5%	9%	17%	54%	4%	5%		6%
99-1889 C106-AQ-8B1	1.26E+0 2%	4.10E-1 5%	3.16E-2 8%	2.49E-3 23%	7.43E-1 4%	2.00E-1 5%	<3.E-4	1.39E+0
99-1892 C106-OH-8	1.47E+0 2%	8.34E-1 5%	2.23E-2 9%	1.63E-3 30%	1.20E+0 4%	1.69E-1 5%	<4.E-4	2.23E+0
99-1892 DUP C106-OH-8	1.62E+0 2%	8.34E-1 5%	3.08E-2 8%	1.09E-3 38%	8.33E-1 4%	1.88E-1 5%	<5.E-4	1.89E+0
RPD	10%	0%	32%	40%	36%	11%		17%
99-1893 C106-OH-8B1	4.91E-1 3%	2.69E-1 5%	5.31E-3 13%	3.93E-4 45%	3.16E-1 4%	5.54E-2 5%	<2.E-4	6.46E-1
99-1893 REP C106-OH-8B1	4.89E-1 3%	2.68E-1 5%	5.59E-3 8%	5.78E-4 22%	2.90E-1 3%	4.96E-2 4%	<2.E-4	6.14E-1
RPD	0%	0%	5%	38%	9%	11%		5%
Matrix Spike	75%	92%			95%			
Blank Spike	105%	91%			108%			
Blank	<3.E-6	<3.E-4	<2.E-4	<7.E-5	<2.E-5	<2.E-5	<9.E-6	

*Due to alpha self-absorption effects, the sum of the alpha emitters is a more reliable estimate of total alpha activity.

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

99-1888
6/22/99

Client : Lumetta

Cognizant Scientist: LRGreenwood Date : 6/22/99

Concur : T Trang-le Date : 6/23/99

Measured Activities (uCi/g)

ALO ID Client ID	Alpha Error %	Co-60 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Ru/Rh-106 Error %	Ce/Pr-144 Error %
99-1888PB Process Blank	1.20E-2 24%	<5.E-4	<2.E-3	4.67E-3 4%	1.04E-1 2%	<2.E-3	<2.E-3	<2.E-3	<5.E-3	<4.E-3
99-1888 C106-AQ-8	4.89E+0 2%	2.95E-1 3%	3.70E-1 23%	2.82E-2 32%	4.01E+2 2%	3.67E+0 1%	2.41E+0 4%	2.74E+0 5%	<7.E-1	<5.E-1
99-1888 DUP C106-AQ-8	4.67E+0 2%	2.94E-1 3%	4.50E-1 17%	2.52E-2 39%	3.54E+2 2%	3.39E+0 1%	2.33E+0 4%	2.60E+0 5%	<6.E-1	<5.E-1
RPD	5%	0%	20%	11%	12%	8%	3%	5%		
99-1889 C106-AQ-8B1	1.26E+0 2%	7.28E-1 2%	<2.E-1	9.03E-2 7%	8.08E+1 2%	5.80E-1 2%	5.40E-1 6%	4.35E-1 10%	3.89E-1 15%	4.90E-1 16%
99-1892 C106-OH-8	1.47E+0 2%	1.20E-1 3%	1.13E-1 38%	1.58E-2 30%	9.90E+1 2%	1.09E+0 2%	6.48E-1 6%	9.76E-1 6%	<4.E-1	<3.E-1
99-1892 DUP C106-OH-8	1.62E+0 2%	1.42E-1 3%	1.61E-1 30%	3.71E-2 16%	1.12E+2 2%	1.38E+0 2%	9.08E-1 5%	1.04E+0 7%	<4.E-1	<3.E-1
RPD	10%	17%	35%	81%	12%	23%	33%	6%		
99-1893 C106-OH-8B1	4.91E-1 3%	4.55E-2 5%	<1.E-1	<8.E-3	3.25E+1 2%	2.89E-1 3%	2.00E-1 10%	3.47E-1 10%	<2.E-1	<2.E-1
99-1893 REP C106-OH-8B1	4.89E-1 3%									
RPD	0%									
Matrix Spike	75%									
Blank Spike	105%									
Blank	<3.E-6									

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

99-1888
7/9/99

Client : Lumetta

Cognizant Scientist: LR Greenwood Date : 7/9/99

Concur : C Soderquist Date : 7-12-99

ALO ID / Client ID	Uranium ug/g Error +/-	Sr-90 Error +/-
	Measured Activities (uCi/g)	
99-1888PB	1.18E+1	1.25E-1
Process Blank	2%	4%
99-1888	1.78E+2	9.13E+2
C106-AQ-8	2%	3%
99-1888 Rep		9.56E+2
C106-AQ-8		3%
RPD		5%
99-1888 DUP	1.73E+2	9.16E+2
C106-AQ-8	2%	3%
RPD	3%	0%
99-1889	2.21E+2	1.23E+2
C106-AQ-8B1	2%	3%
99-1892	1.44E+2	2.66E+2
C106-OH-8	2%	3%
99-1892 DUP	2.08E+2	3.20E+2
C106-OH-8	2%	3%
RPD	36%	18%
99-1893	2.10E+2	1.32E+2
C106-OH-8B1	2%	3%
Matrix Spike		99%
Blank Spike		95%
Blank		<1.E-2

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

99-1888
8/12/99

Client : Lumetta

Cognizant Scientist: ______________________ Date : ________

Concur : ______________________ Date : ________

Measured Activities (uCi/g)

ALO ID Client ID	C-14 Error +/-
99-1888 C106-AQ-8	7.73E-3 2%
99-1892 C106-OH-8	1.76E-3 4%
99-1892 DUP C106-OH-8	5.95E-4 8%
RPD	99%
Matrix Spike	82%
Matrix Spike Dup	25%
Blank Spike	65%
Blank Spike Dup	41%

Note: Sample recoveries were very low and not reproducible. The duplicate of sample C106-OH-8 disagrees with a high RPD of 99%. The reason for these poor results are not understood.

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329 File
LSO

Date June 25, 1999

To Mike Urie

From Tom Farmer

Subject ICPMS Analysis BNFL samples
(ALO# 99-1881 through 99-1894)

Pursuant to your request, the samples that you submitted for analysis were analyzed on our radioactively-contained ICPMS for the selected analytes; semiquantitative analysis was necessary on certain isotopes for which a standard was not available (see below). The concentration results for the isotopes of interest are displayed on the attached spreadsheets.

Dilutions of Isotope Products standards for ^{129}I, ^{233}U, ^{237}Np and ^{239}Pu, an Amersham ^{99}Tc standard and an NIST isotopic uranium standard (4321B) 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. A duplicate and a spike sample 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. Approximate ^{101}Ru concentrations have been provided for your information.

Interference corrections were performed on the following isotopes: ^{129}I (xenon corrected), ^{239}Pu (Uranium hydride corrected). Printouts of the spreadsheet calculations for these corrections have been provided in the data package.

The results are reported in µg analyte /g (ppm) of original sample material for the fusion samples and ng analyte /g (ppb) of original sample material for the acid digestion samples. The overall uncertainty of the values is conservatively estimated at ±10%, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

Values for the following isotopes were obtained using responses from related isotopes: ^{236}U (obtained from ^{238}U), and ^{240}Pu (obtained from ^{239}Pu). Because standards were not used and the concentrations of the isotopes were determined indirectly, these results should be considered semiquantitative. Printouts of the spreadsheet calculations are provided in the data package.

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

6/28/99

Lumetta Analysis

June 24, 1999

Results are reported in µg/g (ppm) of original sample.
The uncertainty of the results is estimated at ±10%.

Sample Number	ICP/MS Number	Client Number	*Tc-99 ug/g	•Ru-101 µg/g	I-129 ug/g	U-233 ug/g	U-234 ug/g	U-235 ug/g	†U-236 ug/g	U-238 ug/g	Np-237 ug/g	Pu-239 ug/g	Pu-240 ug/g
1%HNO3	9623a1		<0.5		<1.4	<1	<0.5	<0.5	<0.5	<0.5	<1	<0.5	<0.5
1%HNO3	9623a10		<0.5		<1.4	<1	<0.5	<0.5	<0.5	<0.5	<1	<0.5	<0.5
1%HNO3	9623a23		<0.5		<1.4	<1	<0.5	<0.5	<0.5	<0.5	<1	<0.5	<0.5
99-1888-PB	9623a11	Process Blank	<0.5	0.1	4.8	<1	<0.5	<0.5	<0.5	2.21	<1	<0.5	<0.5
99-1888	9623a15	C106-AQ-8	1.0±0.2	33	24.6	<1	<0.5	1.9±0.8	<0.5	286.	2.4±0.7	50.1	3.3±0.5
99-1888-DUP	9623a16	C106-AQ-8	1.2±0.2	30	18.6	<1	<0.5	2.4±0.8	<0.5	258	2.55	46.3	3.8±0.4
99-1889	9623a17	C106-AQ-8B1	<0.5	1	4.3	<1	<0.5	12±2	<0.5	375	<1	14.2	1.6±0.5
99-1889 (Dup.)	9623a21	C106-AQ-8B1	<0.5	1	<1.3	<1	<0.5	13.9	<0.5	403	<1	13.1	1.5±0.3
99-1892	9623a12	C106-OH-8	<0.5	13	5.6	<1	<0.5	2.31	<0.5	250	<1	24.9	2.1±0.6
99-1892-DUP	9623a13	C106-OH-8	<0.5	15	8.0	<1	<0.5	2.7±0.5	<0.5	320	2.4±0.5	14.1	0.9±0.1
99-1893	9623a19	C106-OH-8B1	<0.5	3	<1.4	<1	<0.5	3.5±1.7	<0.5	382	<1	6.08	0.7±0.2
99-1893 (Spike)	9623a22	C106-OH-8B1	25.1		11.4	9.4±1.2				522	6.96	35.2	
Spike Recovery			108%		108%	101%				110%	101%	79%	
SRM 2710	9623a20	LCS/99-1888	<0.5		<1.4	<1	<0.5	0.5±0.5	<0.5	49.1	<1	0.5±0.4	<0.5
2ppb Tc-99	9622a9		1.92										
2ppb Tc-99	9622a23		2.04										
10ppb Co	9622a24		<0.5										
I-129 2ppb	9623a8				2.20								
I-129 2ppb	9623a24				2.03								
20ppb U	9623a8									20.0			
20ppb U	9623a24									19.4			
Multi CCV	9623a9					0:99±0.13					0.735	5.26	
Multi CCV	9623a26					0.98±0.12					0.789	5.07	
True Value						1.00					0.740	5.00	

*Results are from procedure 9622a.
•Calculated using response of Indium. For information only.
† No standard available, results calculated from response of different isotope.

DATA REVIEW

Reviewed by: O. T. Farmer III

Date: 28Jun99 Pages: 1 of 1

J P Brown
6/28/99

Lumetta Tc-99 Analysis

June 24, 1999

Results are reported in ng/g (ppb) of original sample.
The uncertainty of the results is estimated at ±10%.

Sample Number	ICP/MS Number	Tc-99 ng/g	•Ru-101 ng/g
1%HNO3	9621a1	<1	
1%HNO3	9621a6	<1	
1%HNO3	9621a26	<1	
Prep Blank	9621a8	3±1	1
BS - 1881	9621a20	2±0.5	
99-1881 C106-SOL-30-1	9621a9	118	2700
99-1881 (Dup.)	9621a18	112	2900
99-1882 C106-SOL-30-2	9621a10	82.4	2100
99-1883 C106-SOL-40-1	9621a11	97.3	2200
99-1884 C106-SOL-40-2	9621a12	92.0	2000
99-1885 C106-SOL-50-1	9621a13	117	2800
99-1886 C106-SOL-50-2	9621a14	97.4	2000
99-1886 (Spike)	9621a22	196	
Spike Recovery		104%	
99-1890 C106-AQ-9	9621a15	25.9	400
99-1891 C106-OH-3A	9621a23	108	2100
Dupe - 1891	9621a24	108	2100
MS - 1891	9621a25	109	2100
99-1894 C106-OH-9	9621a19	5.30	50
10ppb Tc-99	9621a7	9.78	
10ppb Tc-99	9621a30	9.16	
10ppb Co	9621a27	<1	
50ppb Co	9621a28	<1	

•Calculated using response of indium. For information only.

To convert from ng/g to µCi/g:

$$\frac{ng}{g} \cdot \frac{g}{10^9 ng} \cdot \frac{0.017 Ci}{g} \cdot \frac{10^6 \mu Ci}{Ci}$$

DATA REVIEW

Reviewed by: D. T. Farmer III

Date: 28 Jun 99 Pages: 1 of 1

Battelle
Putting Technology To Work

Project No. 29953

Internal Distribution

Date July 9, 1999 File/LB

To G. Lumetta

From M. Urie MW Urie

Subject Carbon Analysis Results for C-106 SOL, AQ, and OH Samples

The analysis of the C-106 liquid and solid samples submitted under ASR 5397 was performed by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same weighed sample, with TC being the sum of the TIC and TOC.

The samples were analyzed on June 28-30, and Table 1 below shows the results, rounded 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.

Most liquid sample were analyzed directly (i.e., no preparative or analytical dilution), and are reported in microgram of carbon per milliliter of original sample. Sample C-106-SOL-50-1 appeared to have a high solids content and was diluted approximately 10 fold with DIW prior to carbon analysis. It should be noted that all liquid samples contained visible quantities of solids. Although the liquid samples are reported by volume, the liquid samples were also weighed and the results can be corrected to micrograms of carbon per gram of sample, if required.

Per instructions, sample C-106-AQ-3Solids was dissolved in DIW prior to carbon analysis. The carbon results on the resulting solution are adjusted for the dissolution and reported in micrograms of carbon per gram of sample. All solids samples were analyzed directly and reported in micrograms of carbon per gram of sample.

Inadvertently, carbon analysis of the water leaches of the solids C-106-AQ-8 and C-106-OH-8 was also performed. Although carbon analysis of the water leaches was not requested, the results are reported, as microgram of leached carbon per gram of sample, for information only. Since these carbon analyses were not request, no project funds were use for the analysis.

QC Narrative

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).

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type. The QC system calibration standards were all within acceptance criteria, with the average recovery being 98.9% for TIC and 96.6% for TOC. The calibration blanks were acceptable, averaging 15 µgC for TIC and 39 µgC for TOC.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike recoveries from liquid sample C-106-SOL-30-1 were 99% for TIC and 80% for TOC, both within the acceptance criteria of 75% to 125%. The matrix spike recoveries from solid sample C-106-OH-8 were 131% for TIC and 77% for TOC. Since C-106-OH-8 has very little TIC, the TIC spike should demonstrate nearly 100% recovery. Both the TIC and TOC spikes are added to the sample at the same time. The recovery of the "total carbon" added to C-106-OH-8 was 112%, suggesting a significant matrix effect on the TOC spike. The high variability of the TOC result for C-106-OH-8 also adversely affects the TOC spike recovery.

The precision (estimated by the Relative Percent Difference between duplicates where the carbon concentration is greater than 5 times the method detection limit) was good, with RPDs being well within the acceptance criteria of 20%, except for sample C-106-OH-8. The poor precision on C-106-OH-8 was demonstrated in both the direct solids analysis and on the leach solutions from the solids, suggesting significant sample heterogeneity.

Some results are 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 or mass of sample used for the analysis. The system MDL is based on the attached pooled historical blank data.

Table 1: TIC, TOC, and TC Results

ALO Number	Sample ID	Vol (ml)	Dilution	TIC (µg C/ml)	TIC RPD (%)	TOC (µg C/ml)	TOC RPD (%)	TC (µg C/ml)	TC RPD (%)
99-1881	C106-SOL-30-1	0.10	1.00	1,810		6,740		8,550	
99-1881 Rep	C106-SOL-30-1	0.10	1.00	1,820	1	6,630	2	8,440	1
99-1881 MS	C106-SOL-30-1	0.10		99%		80%		90%	
99-1882	C106-SOL-30-2	0.10	1.00	1,900		6,880		8,780	
99-1882 Rep	C106-SOL-30-2	0.10	1.00	1,940	2	7,780	12	9,710	10
99-1883	C106-SOL-40-1	0.20	1.00	2,140		8,020		10,200	
99-1883 Rep	C106-SOL-40-1	0.20	1.00	2,020	6	7,560	6	9,580	6
99-1884	C106-SOL-40-2	0.20	1.00	1,760		7,120		8,890	
99-1884 Rep	C106-SOL-40-2	0.20	1.00	1,740	1	7,060	1	8,800	1
99-1885	C106-SOL-50-1	0.40	10.86	2,110		8,220		10,300	
99-1885 Rep	C106-SOL-50-1	0.38	10.86	2,240	6	8,260	1	10,500	2
99-1886	C106-SOL-50-2	0.20	1.00	2,150		7,560		9,710	
99-1886 Rep	C106-SOL-50-2	0.10	1.00	2,180	1	7,950	5	10,100	4
ALO Number	**Sample ID**	**Vol (ml)**	**Leach Dilution**	**TIC (µg C/g)**	**TIC RPD (%)**	**~~TIC~~ TOC * (µg C/g)**	**TOC RPD (%)**	**TC (µg C/g)**	**TC RPD (%)**
99-1887 PB	Prep Blank	2.00	20.43	< 140		< 400		< 540	
99-1887	C106-AQ-3Solid	0.20	20.43	2,120		140,000		143,000	
99-1887 Rep	C106-AQ-3Solid	0.20	20.43	1,700	n/a	140,000	0	142,000	1
99-1888	C106-AQ-8 (Leached)	1.00	38.99	< 600		9,580		9,580	
99-1888 Dup	C106-AQ-8 (Leached)	1.00	38.70	< 600	n/a	6,790	n/a	6,790	n/a
99-1892	C106-OH-8 (Leached)	0.10	30.09	< 4500		96,700		96,700	
99-1892 Dup	C106-OH-8 (Leached)	0.10	29.54	< 4500	n/a	122,000	25	122,000	25
ALO Number	**Sample ID**	**Wt (g)**		**TIC (µg C/g)**	**TIC RPD (%)**	**TOC (µg C/g)**	**TOC RPD (%)**	**TC (µg C/g)**	**TC RPD (%)**
99-1888	C106-AQ-8	0.0613		6,410		31,300		37,700	
99-1888 Dup	C106-AQ-8	0.0788		6,470	1	28,300	10	34,700	8
99-1892	C106-OH-8	0.0078		1,400		150,000		151,000	
99-1892 Dup	C106-OH-8	0.0038		<1700	n/a	121,000	22	121,000	23
99-1892 Spike	C106-OH-8	0.0060		131%		77%		112%	

* G.J. Lumetta 7/13/99

Review/Approve: M J Steele 7-9-99

Archive Information:

Files: ASR 5397 Lumetta.doc, ASR 5397 Lumetta.xls

TOC R.L.L. 7/13/99

				TIC	TIC RP	~~TIC~~	TOC RP	TC	TC RPD
ALO Number	Sample ID	wt (g)	Dilution	(ugC/g)	(%)	(ugC/g)	(%)	(ugC/g)	(%)
99-1881	C106-SOL-30-1	0.1042	1.00	1,732		6,471		8,203	
99-1881 Rep	C106-SOL-30-1	0.0977	1.00	1,857	7	6,785	5	8,642	5
99-1881 MS	C106-SOL-30-1	0.1063		99%		79%		90%	
99-1882	C106-SOL-30-2	0.1047	1.00	1,811		6,568		8,379	
99-1882 Rep	C106-SOL-30-2	0.1035	1.00	1,871	3	7,514	13	9,385	11
99-1883	C106-SOL-40-1	0.2276	1.00	1,881		7,051		8,932	
99-1883 Rep	C106-SOL-40-1	0.2074	1.00	1,943	3	7,293	3	9,236	3
99-1884	C106-SOL-40-2	0.2123	1.00	1,660		6,711		8,370	
99-1884 Rep	C106-SOL-40-2	0.2044	1.00	1,704	3	6,904	3	8,608	3
99-1885	C106-SOL-50-1	0.4014	10.86	2,106		8,188		10,294	
99-1885 Rep	C106-SOL-50-1	0.3796	10.86	2,241	6	8,273	1	10,515	2
99-1886	C106-SOL-50-2	0.2114	1.00	2,035		7,155		9,190	
99-1886 Rep	C106-SOL-50-2	0.1047	1.00	2,081	2	7,596	6	9,677	5

HOT PERSUL... .E WORKSHEET

Client G. Lumetta **ASR** 5397 **Analyst** MJ Steele **Date** 6/28 (6/29) (6/2[illegible])

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

TIC STD: Calcium Carbonate CMS-132985 TOC STD: Glucose CSM-53219

	injection #	Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)	comments
6/28	1	Blank	—		—	18.08	53.52	
	2	Blank	—		—	15.98	40.39	
	3	Standard	—	TIC 0.0109	0.0024 TOC	1382	1040	
	4	Standard	—	0.0102	0.0030	1234	1150	
	5	99-1881	C106-S01-30-1	100ul	0.1043	193	690	
	6	↓	↓		0.0977	194	679	
	7	99-1882	C106-S01-30-2		0.1047	202	703	
	8	↓	↓	↓	0.1035	206	790	
	9	99-1883	C106-S01-40-1	200ul	0.2276	438	1589	
	10	↓	↓	↓	0.2312	442	RR	baseline high - Solution changed
	11	Standard	—	0.0129	0.0013	1525	520	
	12	Blank	—		—	14.78	41.31	
6/29	13	Standard	—	0.0125	0.0021	1489	840	
	14	Blank	—		—	15.03	30.03	
	15	99-1883	C106-S01-40-1	200ul	0.2074	413	1500	
	16	99-1884	C106-S01-40-2		0.2123	363	1415	
	17	↓	↓	↓	0.2044	359	1402	
	18	99-1885 (10X)	C106-S01-50-1	400ul	0.4014	91.5	331	
	19	↓	↓	≈380ul	0.3796	92.14	318	
	20	99-1886	C106-S01-50-2	200ul	0.2114	440	1500	
	21	↓	↓	100ul	0.1047	230	807	
	22	99-1887	C106-AQ-3501d	200ul	0.2112	35	1367	
	23	↓	↓	↓	0.2070	31	1361	

HOT PERSUL... E WORKSHEET

	Injection #	Lab ID	Client ID	volume	sample wt (g)	TIC (ug)	TOC (ug)	comments
	24	Standard	—	0.0168	0.0032	1920	1244	
	25	Blank	—		—	12.14	29.49	
6/30	26	Standard	—	0.0216	0.0043	2586	1725	
	27	Blank	—		—	12.89	38.91	
	28	99-1881MS		.0100 / .0027	100ul .1063	1372	1529	
	29	99-1887PB		2X 1000ul	2.003	15.99	40.16	
	30	99-1888	C106-A08	—	0.0613	403	1890	solid weighed in HotCell
	31	99-1888Dup	↓	—	0.0788	519	2170	precleaned Flasks
	32	99-1892	C106-014-8	—	0.0078	2563	1171	↓
	33	99-1892Dup	↓	—	0.0038	2035	481	
	34	99-1892MS		.0128 / .0021	0.0060	2008	1538	
	35	Blank	—		—	12.39	35.05	
	36	Standard	—	.0162 / .0032	—	1940	1308	
	37	99-1888		100ul	1.1022	15.7	NT	Hot cell leach
	38	99-1888Dup		↓	0.1029	17.9	60.18	↓
7/1/99	39	99-1888		1000ul	1.0080	25.11	276	
	40	99-1888Dup		↓	0.9972	28.35	206	
	41	99-1892		100ul	0.1000	26.89	349	
	42	99-1892Dup		↓	0.1036	25.11	437	
	43	Standard	—	.0128 / .0021	—	1516	850	
	44	Blank	—			14.79	40.29	
	45							
	46							
	47							
	48							
	49							
	50							

Battelle
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Project No. 29953

Internal Distribution

Date July 19, 1999

To Gregg Lumetta

From Pam Berry P.K. Berry 7/20/99

Subject Cyanide Results for Samples C106-AQ-8 and C106-OH-9

ALO#	Client ID	CN Results Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Spike Recovery (%)
99-1888	C106-AQ-8	10.4	18.5	56	
99-1892	C106-OH-8	5.0	4.0	23	
99-1892 spike	C106-OH-8 spike	12.0			99
99-1892 spike	C106-OH-8 spike		10.5	---	93

The CN results for two C106 tank samples analyzed on July 2, 1999 per ASR 5397 are reported in the table above. The sample aliquots were weighed in the Shielded Analytical Laboratory and delivered, ready for distillation, to Laboratory 400 in the Radiochemical Processing Laboratory. The samples were distilled with the addition of sulfamic acid to ensure there would be no interference if nitrates were present in the sample. The samples were analyzed using a Lachat QuickChem AE Autoanalyzer (WC36517). The reporting limit is estimated to be 0.2 mg/kg.

An independent calibration check solution run at the beginning and end of each analysis batch gave an average recovery of 109%. Both samples were prepared and analyzed in duplicate. In addition, sample C106-OH-8 was prepared in duplicate for matrix spikes. The spike recoveries were within the control limits (±15%). The solid laboratory control standard (ERA-LSC) recovery was 116%, which is greater than the control limit (85% to 115%). A rerun of that same control standard a week later gave a 113% recovery. The distilled standard recoveries are slightly high, which may indicate a slightly high bias in the reported results. The sample, duplicate, and matrix spike results are reported in the table above.

The matrix spike recoveries, i.e., 99% and 93%, are well within the acceptance criterial of 75% to 125%. The relative percent difference (RPD) between the samples and duplicated exceed the acceptance criteria of 20% for both samples. Both samples, but particularly C106-AQ-8, appears to have significant sample heterogeneity. The C106-AQ-8 duplicate was outside the original calibration curve so it was diluted and rerun. The analysis of the diluted sample verified the initial analytical result.

All sample preparation sheets, standard preparation information, and analytical data are included with this report.

MW Urie   7/20/99
Concur   Date

Memo File: CN ASR 5397 Lumetta.doc	Spreadsheet File: CN ASR 5397 Lumetta.xls

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

WO/Project: **W48486/29953**
Client: **G. Lumetta**

ACL Numbers: 99-1881 through 99-1888, 99-1892

Client ID: C106 SOL and AQ Series

ASR Number: 5397

Total Samples: 6 liquids, 3 solids

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography" (IC).

Analyst: MJ Steele

Analysis Date: June 16-22, 1999

See Chemical Measurement Center 98620: IC File for Calibration and Maintenance Records.

M&TE Number: IC instrument -- WD25214
Mettler AT400 Balance – Cal. No. 360-06-01-031

Analyst: MJ Steele 7/22/99

Approval: MW Urie 7/22/99

Final Results:

Four liquid samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5397. The liquid samples were diluted at the IC workstation up to 1,000-fold to ensure that all anions were within the calibration range. The solids samples were dissolved (C106-AQ-3Solids) or leached (C106-AQ-8 and C106-OH-8) with DIW, and then diluted at the IC workstation similar to the liquid samples. The anion results are presented in the table below.

LIQUIDS

LAB ID	SAMPLE ID	F ug/ml	Cl ug/ml	NO_2 ug/ml	Br ug/ml	NO_3 ug/ml	PO_4 ug/ml	SO_4 ug/ml	C_2O_4 ug/ml
99-1881	C106-SOL-30-1	<250	410	970	<250	<500	<500	<500	21,400
99-1882	C106-SOL-30-2	<250	440	980	<250	<500	<500	<500	22,700
99-1883	C106-SOL-40-1	<250	470	<500	<250	<500	<500	<500	24,300
99-1884	C106-SOL-40-2	<250	450	950	<250	<500	<500	<500	24,300
99-1885	C106-SOL-50-1	<250	470	1,000	<250	<500	<500	<500	25,900
99-1885 MS	....50-1 MS	101%	105%	101%	105%	101%	102%	103%	107%
99-1886	C106-SOL-50-2	<250	510	1,100	<250	<500	<500	<500	28,000

SOLIDS

Prep Fctr	LAB ID	SAMPLE ID	F ug/g	Cl ug/g	NO_2 ug/g	Br ug/g	NO_3 ug/g	PO_4 ug/g	SO_4 ug/g	C_2O_4 ug/g
19.4	99-1887	C106-AQ-3Solid	<5,000	<5,000	<10,000	<5,000	<10,000	<10,000	<10,000	495,000
34.3	99-1888 PB	DIW Leach Blk	<9	10	<17	<9	<17	<17	41	29
39.0	99-1888	C106-AQ-8	300	12,000	<400	<200	3,000	<400	<400	20,300
38.7	99-1888 Dup	C106-AQ-8d	360	5,800	<400	<200	1,300	<400	<400	9,700
		RPD	18%	70%	n/a	n/a	79%	n/a	n/a	71%
30.1	99-1892	C106-OH-8	<8,000	<8,000	<15,000	<8,000	<15,000	<15,000	<15,000	355,000
29.5	99-1892 Dup	C106-OH-8d	<8,000	<8,000	<14,800	<8,000	<15,000	<15,000	<15,000	434,000
		RPD	n/a	n/a	n/a	n/a	n/a	n/a	n/a	20%

RPD = Relative Percent Difference (between sample and duplicate); n/a = sample and/or duplicate less than MDL.

Q.C. Comments:

Following are results of quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan, MCS-033.

Matrix Spiked Sample: The matrix spike recovery for samples C106-SOL-50-1 ranged from 101% to 107%, well within the acceptance criteria of 75% to 125%.

Duplicate: No liquid sample duplicates were identified. Solid sample C106-AQ-8 and C106-OH-8 were leached and analyzed in duplicate. Sample C106-OH-8 barely met the relative percent difference (RPD) acceptance criteria of 20%, and sample C106-AQ-8 demonstrated very poor reproducibility. The duplicates of C106-AQ-8 appear to be different by a factor of two; however, reanalysis of the leach solution verified the concentration of both the sample and the duplicate. Basically, the leach reproducibility of the solids samples appears to be very poor; this was also demonstrated in other analyses (e.g., ICP and TOC/TIC).

System Blank/Processing Blanks: Twenty system blanks were analyzed during the analysis of all of the sample. Only bromide on two system blanks and nitrate on one system blank were measured above the lowest calibration standard. However, no bromide was detected in any of the samples, and the blank nitrate values was the initial system blank and does not effect the sample reported values.

Quality Control Calibration Verification Check Standards: Twelve mid-range verification standards were analyzed throughout the analysis run. For all reported results, except on chloride result, the concentrations of all analytes of interest were recovered within the governing QA Plan acceptance criteria of 90% to 110% for the verification standard. The single chloride failure (80%) had no effect on the reported results; a immediate reanalysis of the chloride standard demonstrated nearly 100% recovery.

Notes:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and 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. Sample-specific precision and bias may be determined on each sample if required.

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Project No. ____________

Internal Distribution

File/LB

Date July 23, 1999

To ~~Mike Urie~~ Gregg Lumetta 9RP 7/28/99

From Chuck Soderquist Chuck Soderquist 7-23-99

Subject Ammonia Analysis

We've completed the ammonia analysis of samples C106 AQ-8 and C106 OH-8. The results are in the attached report.

Ammonia was measured by ion selective electrode in water solutions of the samples (slurries for the solids). The high dissolved solids in these samples required that we measure ammonia by standard addition. The samples were made strongly basic before ammonia measurement, so that all ammonium ion was converted to ammonia and accurately measured. Certain organic amines will interfere and show up as ammonia; if any such amines are present in these samples, then the data is biased high. (Metals complexed by ammonia, such as zinc, cadmium, copper, and mercury, do not interfere in this method.)

The ammonia probe gave a highly linear calibration over the range of 10^{-2} to 10^{-4} molar ammonia, and the slope dropped only slightly down to 10^{-5} molar. The slope went to zero a little below 10^{-6} molar. The detection limits on the report are conservatively based on 10^{-6} molar, although the probe will measure down to about 5×10^{-7} molar with a smaller, non-linear slope.

The ammonia probe's slope was measured before, after, and periodically throughout the analysis. Between 10^{-5} molar and 10^{-2} molar, the slope was reproducible to $\pm$ 5%, 1s. Deionized water blanks were also taken periodically throughout the analysis to confirm that the probe was uncontaminated with ammonia.

Uncertainty estimates were based on the uncertainty in the voltage change from the standard addition and the uncertainty of the electrode slope. The other sources of uncertainty are from volume and weight measurements, which are small compared to the uncertainty in the voltage and slope measurements. 1s uncertainty was calculated by adding 0.2 mV to the voltage change from standard addition and adding 5% to the slope.

The two samples have detectable ammonia, but the accompanying hot cell blank has about the same amount. The two sample solutions, their duplicates, and the accompanying blank solution had between 2.7×10^{-5} and 5.0×10^{-6} molar ammonia, not much above the detection limit of 1×10^{-6} molar. The ammonia concentration for sample C106-AQ-8 is suspiciously close to the hot cell blank ammonia concentration. Sample C106-OH-8 has about 5 times as much ammonia as the hot cell blank.

The duplicates of sample C106-AW-8 agree well, considering how close the results are to the detection limit. The duplicates of sample C106-OH-8, however, are about 3s apart.

JRGreenwood
7-26-99

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

99-1888 NH3
7/22/99

Client: Lumetta

Cognizant Scientist: C - Soderquist 7-23-99

Concur: LR Greenwood 7-23-99

Sample	Lab Number	NH_3 Concentration, µg per g ± 1s		Detection Limit
Hot Cell Preparation Blank	99-1888 PB	2.2	± 20%	0.4
C106-AQ-8	99-1888	3.6	± 20%	0.6
	99-1888 Dup	4.3	± 20%	0.6
C106-OH-8	99-1892	9.5	± 7%	0.5
	99-1892 Dup	13.2	± 6%	0.5

The detection limit is 10^{-6} molar NH_3, corrected for the particular sample's dilution, converted to µg NH_3 per gram sample.

Periodic checks of the electrode slope gave a stability of ± 5% (1s) over the time the samples were analyzed. Each sample was measured by standard addition.

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

Project: 29953
Client: G. J. Lumetta

ACL Number(s): 99-1881 through 99-1894

Client ID: "C106-SOL-30-2" through "C106-OH-9"

ASR Number: 5397

Total Samples: 14

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

Analyst: DR Sanders

Analysis Date (Filename): 7-2-99 (A0532), 7-9-99 (A0534), 7-14-99 (A0535)

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 7-30-99
Reviewed by

8-3-99
Concur

Nine radioactive liquid samples, C106-SOL-30-1 through C106-SOL-50-2, C106-AQ-9, C106-OH-3A and C106-OH-9 (ACL# 99-1881 through 99-1886, 99-1890, 99-1891, and 99-1894), 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 2ml to 4ml of sample (weighed) was processed and diluted to a final volume of 20ml. Most of the liquid samples contained some visible solids before processing. Sample C106-OH-3A (ACL# 99-1891) was completely clear (no visible solids) before processing. After digestion all samples were clear and did not require filtering.

One radioactive solid sample, C106-AQ-3Solid (ACL# 99-1887), was analyzed by ICPAES after preparation by SRPL. Approximately 0.5g of sample was dissolved in water using an ultra-sonic water bath, filtered through 0.45μm membrane filter and diluted to a final volume of 10ml with water.

Four radioactive samples of dried solids: C106-AQ-8, C106-AQ-8B1, C106-OH-8 and C106-OH-8B1 (ACL# 99-1888, 99-1889, 99-1892 and 99-1893), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Approximately 0.1g aliquots were used to prepared samples using both fusion procedures PNL-ALO-114 Na_2O_2/Zr and PNL-ALO-115 KOH/Ni. After samples were fused they were diluted to a final volume of 50 ml. Samples were diluted approximately 2-fold before moving to the analytical laboratory by SAL because of ALARA radiation dose concerns. Additional dilution, up to 10 fold, was performed during ICPAES analysis because of high aluminum, calcium, iron and sodium concentration levels. Duplicate samples were also prepared of C106-AQ-8 and C106-OH-8 using both fusion procedures.

All measurement results reported have been corrected for preparation and analytical dilution. Analytes of interest include Ag, Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Ti, U, Zn and Zr. All results reported are in μg/g including liquid samples as requested by the client. Volumes and weights have been recorded on bench sheets (included with raw data, etc.).

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

<u>Five fold serial dilution:</u>

(Solid samples)	All results are within tolerance limit of ≤ 10% after correcting for dilution.

(Aqueous samples) All results were within tolerance limit of ≤ 10% after correcting for dilution except Al (approximately 11%) in sample C106-SOL-30-2 (ACL# 99-1886). Other analytes in the same dilution were off by about the same percentage. The discrepancy is likely due to a pipetting error. Seven other diluted samples prepared similarly were all within the ≤ 10% tolerance limit.

<u>Duplicate RPD (Relative Percent Difference):</u>

(Solid samples) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD) except for Ag (41%), Ca (23%), Mg (44%) in sample C106-AQ-8 (ACL# 99-1888 Na/Zr fusion) and Si (29%) in the KOH/Ni fusion of the same sample.

Also Ag (78%) in sample C106-OH-8 (ACL# 99-1892 Na/Zr fusion) and Ca (58%), Pb (37%), Si (27%) and Zr (93%) in the KOH/Ni fusion of the same sample exceeded the tolerance limit of ≤ 20% RP.

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

<u>Post-Spiked Samples (Group A):</u>

(Solid samples) All analytes of interest were recovered within tolerance of 75% to 125%.

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

<u>Post-Spiked Samples (Group B):</u>

(Solid samples) All analytes of interest were recovered within tolerance of 75% to 125%.

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

<u>Blank Spike:</u>

(Solid samples) A blank spike is not require for fusion prepared samples.

(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120% except Ag (33%) in sample C106-SOL-30-1 (ACL# 99-1881-BS). Chloride from the sample or from the hydrochloric acid used to prepare the sample using PNL-ALO-128 digestion procedure may have precipitated the silver resulting in low recovery.

Matrix Spiked Sample:

(Solid samples) A matrix spike is not require for fusion prepared samples.

(Aqueous samples) All analytes of interest in the matrix spiked sample C106-SOL-30-1 (ACL# 99-1881-MS) were recovered within tolerance limit of 75% to 125% except Ag (63%). Chloride from the sample or from the hydrochloric acid used to prepare the sample using PNL-ALO-128 digestion procedure may have precipitated the silver resulting in low recovery.

Quality Control Check Standards:

Concentration of all analytes of interest within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV. Calibration Blank (ICP98.0) concentration was less than two times IDL.

High Calibration Standard Check:

Verification of the high-end calibration concentration for all analytes of interest was within tolerance of ± 5% accuracy except for K. Potassium was slightly high, between 6% and 7%, in the high-end calibration check standard measurements of QC_SST. The slightly high measurement results were likely due to sample carry-over from analysis of KOH/Ni fusion reagents. Since potassium is not reported for KOH/Ni fusion prepared samples, measurement results are not affected.

Process Blank:

(Solid samples)	All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except Mn (<12% of sample concentration) and Na (< 32% of sample concentration) in PNL-ALO-115 KOH/Ni fusion prepared samples. No significant blank contribution found for PNL-ALO-114 Na/Zr fusion prepared samples.
(Aqueous samples)	All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration except Si (similar in concentration to the samples) in PNL-ALO-128 acid digestion prepared samples.

Laboratory Control Standard (LCS):

(Solid samples)	All analytes of interest at a concentration equal to or greater than EQL, except Ni, were recovered within tolerance limit of 75% to 125% in both fusion prepared LCS standards. SRM-2710 Montana Soil was used for the LCS in both PNL-ALO-114 and PNL-ALO-115 fusion preparations. Nickel recovery in the Na/Zr (PNL-ALO-114) fusion prepared LCS was unusually high, about four times greater than the process blank concentration. Previous LCS measurement results have all been below EQL for nickel. Contamination during sample preparation of the LCS is suspected since the concentration is so much higher than normal.
(Aqueous samples)	No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. 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	*Multiplier=*	1.0	9.6	19.1	19.0	18.9
	ALO#=	99-1881-PB @ 1	99-1881	99-1882 @2	99-1883 @2	99-1884 @2
	Client ID=	*Preparation Blank*	*C106-SOL-30-1*	*C106-SOL-30-2*	*C106-SOL-40-1*	*C106-SOL-40-2*
	Run Date=	7/2/99	7/2/99	7/2/99	7/2/99	7/2/99
(ug/mL)	(Analyte)	(ug/mL)	ug/g	ug/g	ug/g	ug/g
0.015	Ag	--	1.81	[0.97]	[1.6]	[2.6]
0.060	Al	[0.34]	286	147 --	67.9	333
0.080	As	--	--	--	--	--
0.050	B	2.08	25.6	25.7	27.3	27.3
0.010	Ba	[0.028]	[0.27]	[0.27]	[0.27]	[0.26]
0.005	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[3.2]	--	--	[5.2]
0.015	Cd	--	--	--	--	--
0.100	Ce	--	--	--	--	--
0.025	Co	--	--	--	--	--
0.020	Cr	--	[1.7]	[1.7]	[2.8]	[3.4]
0.015	Cu	--	[0.92]	[0.75]	[1.0]	[1.2]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.066]	[0.77]	--	[1.0]	[3.6]
2.000	K	--	--	--	--	--
0.025	La	--	--	--	--	--
0.005	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.005	Mn	--	--	--	--	--
0.030	Mo	--	[0.82]	[0.73]	[0.76]	[0.81]
0.100	Na	2.70	17,800	16,700	18,200	17,100
0.100	Nd	--	--	--	--	--
0.030	Ni	--	[1.3]	[1.2]	[1.6]	[2.0]
0.100	P	--	43.9	42.2	44.7	45.7
0.060	Pb	--	--	--	--	--
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	--	[3.2]	[3.3]	[3.5]	[3.1]
0.050	Sb	--	--	--	--	--
0.050	Se	--	--	--	--	--
0.100	Si	3.36	29.1	29.2	32.0	29.3
1.000	Sn	--	--	--	--	--
0.005	Sr	--	--	--	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	[0.021]	[0.15]	[0.15]	[0.16]	[0.13]
0.250	Tl	--	--	--	--	--
2.000	U	--	[26]	--	--	--
0.015	V	--	--	--	--	--
0.500	W	--	--	--	--	--
0.010	Y	--	--	--	--	--
0.020	Zn	--	--	--	--	--
0.025	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)	37.4 / 99-1885 @2 / C106-SOL-50-1 / 7/2/99 / ug/g	18.9 / 99-1886 @2 / C106-SOL-50-2 / 7/2/99 / ug/g	19.4 / 99-1887-PB (DRY SOLIDS) / Prep. Blank / 7/2/99 / ug/g	38.9 / 99-1887 @2 / C106-AQ-3 Solid / 7/2/99 / ug/g	9.5 / 99-1890 @2 / C106-AQ-9 / 7/2/99 / ug/g
0.015	Ag	[4.1]	[2.7]	--	[4.4]	[1.3]
0.060	Al	442	66.2	--	[16]	75.9
0.080	As	--	--	--	[10]	--
0.050	B	74.0	40.6	--	[9.2]	14.8
0.010	Ba	[0.68]	[0.35]	--	--	[0.16]
0.005	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[4.9]	--	[40]	[5.0]
0.015	Cd	--	--	--	--	--
0.100	Ce	--	--	--	[6.0]	--
0.025	Co	--	--	--	--	--
0.020	Cr	[4.8]	5.02	--	[1.5]	[0.78]
0.015	Cu	[2.1]	[1.9]	--	[5.0]	[1.2]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[1.2]	--	--	--	[1.2]
2.000	K	--	--	--	[170]	--
0.025	La	--	--	--	[1.4]	--
0.005	Li	--	--	--	[0.98]	--
0.100	Mg	--	--	--	[9.9]	[1.9]
0.005	Mn	--	--	--	[0.35]	--
0.030	Mo	--	[1.1]	--	[1.5]	--
0.100	Na	25,500	23,400	[2.0]	223,000	16,500
0.100	Nd	--	--	--	[5.7]	--
0.030	Ni	[2.6]	[3.3]	--	[6.8]	[2.3]
0.100	P	73.8	63.7	--	265	17.8
0.060	Pb	--	--	--	[5.3]	--
0.300	Pd	--	--	--	[17]	--
0.300	Rh	--	--	--	--	--
0.075	Ru	[4.4]	[4.9]	--	[6.7]	[0.99]
0.050	Sb	--	--	--	[3.7]	--
0.050	Se	--	--	--	[5.0]	--
0.100	Si	47.6	46.1	--	--	25.6
1.000	Sn	--	--	--	--	--
0.005	Sr	--	--	--	[0.42]	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	[0.39]	[0.21]	--	[0.31]	[0.084]
0.250	Tl	--	--	--	--	--
2.000	U	--	[44]	--	[140]	--
0.015	V	--	--	--	[0.76]	--
0.500	W	--	--	--	--	--
0.010	Y	--	--	--	[0.49]	[0.14]
0.020	Zn	--	[0.42]	--	--	--
0.025	Zr	--	--	--	[1.0]	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	8.8	17.7	19.0		
	ALO#=	99-1891 @2	99-1891 DUP @4	99-1894 @4		
	Client ID=	*C106-OH-3A*	*C106-OH-3A*	*C106-OH-9*		
Det. Limit	*Run Date=*	7/2/99	7/2/99	7/2/99		
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g		
0.015	Ag	2.41	[2.5]	[0.53]	--	--
0.060	Al	765	791	66.6	--	--
0.080	As	[1.4]	[1.7]	--	--	--
0.050	B	12.9	12.6	12.0	--	--
0.010	Ba	[0.12]	--	--	--	--
0.005	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[2.4]	--	--	--	--
0.015	Cd	--	--	--	--	--
0.100	Ce	--	--	--	--	--
0.025	Co	--	--	--	--	--
0.020	Cr	10.8	11.2	[0.96]	--	--
0.015	Cu	2.51	[2.6]	[0.90]	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	9.72	9.90	[1.7]	--	--
2.000	K	[67]	[65]	--	--	--
0.025	La	--	--	--	--	--
0.005	Li	[0.13]	[0.12]	--	--	--
0.100	Mg	--	--	--	--	--
0.005	Mn	[0.085]	[0.090]	--	--	--
0.030	Mo	[0.88]	[0.85]	--	--	--
0.100	Na	66,400	58,400	15,900	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.38]	--	--	--	--
0.100	P	77.6	78.6	[14]	--	--
0.060	Pb	16.6	17.1	--	--	--
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	[3.0]	[3.2]	--	--	--
0.050	Sb	--	--	--	--	--
0.050	Se	[0.74]	--	--	--	--
0.100	Si	346	353	49.9	--	--
1.000	Sn	--	--	--	--	--
0.005	Sr	--	--	--	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	[0.13]	[0.12]	--	--	--
0.250	Tl	--	--	--	--	--
2.000	U	[43]	[44]	--	--	--
0.015	V	[0.76]	[0.76]	--	--	--
0.500	W	[7.5]	--	--	--	--
0.010	Y	--	--	--	--	--
0.020	Zn	2.27	[2.4]	--	--	--
0.025	Zr	[0.55]	[0.57]	--	--	--

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

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	1.0 / 99-1888-DB-Zr / Diluent Blank / 7/14/99 / (ug/mL)	1983.6 / 99-1888-PB-Zr @2 / Process Blank / 7/14/99 / ug/g	1887.3 / 99-1888-Zr @2 / C106-AQ-8 / 7/14/99 / ug/g	1923.4 / 99-1888-DUP-Zr @2 / C106-AQ-8 / 7/14/99 / ug/g	2196.7 / 99-1889-Zr @2 / C106-AQ-8B1 / 7/14/99 / ug/g
0.015	Ag	--	--	591	391	338
0.060	Al	--	--	62,800	54,200	6,950
0.080	As	--	--	--	[160]	--
0.050	B	--	--	--	--	--
0.010	Ba	--	--	432	387	[100]
0.005	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[2,500]	16,400	12,900	6,950
0.015	Cd	--	--	[58]	[40]	--
0.100	Ce	--	--	[300]	[250]	--
0.025	Co	--	--	--	--	[65]
0.020	Cr	--	--	982	948	[370]
0.015	Cu	--	--	415	390	[200]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	[190]	181,000	157,000	446,000
2.000	K	--	[4,100]	[3,800]	[5,300]	--
0.025	La	--	--	[120]	[120]	--
0.005	Li	--	[15]	[22]	[29]	[27]
0.100	Mg	--	--	4,240	2,710	[1,900]
0.005	Mn	--	--	3,940	3,810	2,950
0.030	Mo	--	--	--	--	--
0.100	Nd	--	--	[290]	[260]	[290]
0.030	Ni	--	[220]	1,260	1,050	[620]
0.100	P	--	--	[890]	[1,400]	[980]
0.060	Pb	--	--	3,200	2,750	[1,200]
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	--	--	[300]	[310]	[660]
0.050	Sb	--	--	--	[100]	--
0.050	Se	--	[150]	[260]	[250]	[350]
0.100	Si	--	--	108,000	104,000	9,030
1.000	Sn	--	--	[1,900]	[2,000]	[5,600]
0.005	Sr	--	[40]	204	168	[70]
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	--	[11]	2,670	2,500	1,300
0.250	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.015	V	--	--	[61]	[56]	[52]
0.500	W	--	--	--	--	--
0.010	Y	--	--	[47]	[32]	[27]
0.020	Zn	--	--	[240]	[250]	[250]

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

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	1917.9	2030.3	1976.4		
	ALO#=	99-1892-Zr @2	99-1892-DUP-Zr @2	99-1893-Zr @2		
	Client ID=	C106-OH-8	C106-OH-8	C106-OH-8B1		
	Run Date=	7/14/99	7/14/99	7/14/99		
	(Analyte)	ug/g	ug/g	ug/g		
0.015	Ag	639	1,460	[290]	--	--
0.060	Al	29,500	28,300	6,810	--	--
0.080	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	290	263	[150]	--	--
0.005	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	11,700	10,400	5,160	--	--
0.015	Cd	[31]	[37]	--	--	--
0.100	Ce	[200]	--	--	--	--
0.025	Co	--	--	--	--	--
0.020	Cr	[300]	[340]	[380]	--	--
0.015	Cu	[140]	[250]	[170]	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	87,300	99,100	402,000	--	--
2.000	K	--	--	--	--	--
0.025	La	[71]	[72]	--	--	--
0.005	Li	[20]	[20]	[16]	--	--
0.100	Mg	2,630	[1,900]	[840]	--	--
0.005	Mn	1,830	2,120	2,700	--	--
0.030	Mo	--	--	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	841	951	719	--	--
0.100	P	[680]	[970]	[980]	--	--
0.060	Pb	1,550	1,800	1,420	--	--
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	[170]	[190]	[600]	--	--
0.050	Sb	--	--	--	--	--
0.050	Se	[150]	[180]	[230]	--	--
0.100	Si	68,800	59,400	33,100	--	--
1.000	Sn	--	--	[4,400]	--	--
0.005	Sr	176	122	[72]	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	2,050	1,960	1,100	--	--
0.250	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.015	V	[31]	--	[31]	--	--
0.500	W	--	--	--	--	--
0.010	Y	[40]	[43]	--	--	--
0.020	Zn	[120]	[160]	[350]	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	1.0 / 99-1888-DB / Diluent Blank / 7/9/99 / (ug/mL)	1819.8 / 99-1888-PB @2 / Process Blank / 7/9/99 / ug/g	1837.5 / 99-1888 @2 / C106-AQ-8 / 7/9/99 / ug/g	1759.5 / 99-1888-DUP @2 / C106-AQ-8 / 7/9/99 / ug/g	1767.3 / 99-1889 @2 / C106-AQ-8B1 / 7/9/99 / ug/g
0.015	Ag	--	--	289	285	496
0.060	Al	--	[200]	70,500	64,200	7,770
0.080	As	--	--	[160]	[170]	--
0.050	B	--	--	--	[110]	--
0.010	Ba	--	--	548	496	[110]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.100	Ca	[0.23]	[230]	13,900	15,800	4,640
0.015	Cd	--	--	[87]	[88]	[29]
0.100	Ce	--	--	[440]	[470]	[190]
0.025	Co	--	--	[68]	[68]	[80]
0.020	Cr	--	--	1,200	1,210	384
0.015	Cu	--	--	524	513	[190]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	[300]	217,000	214,000	508,000
0.025	La	--	--	[150]	[160]	[45]
0.005	Li	--	[16]	[23]	[25]	[16]
0.100	Mg	--	--	2,840	3,180	[1,700]
0.005	Mn	--	213	5,060	5,110	3,320
0.030	Mo	--	--	--	--	[56]
0.100	Na	--	2,510	57,400	52,100	7,880
0.100	Nd	--	--	[450]	[480]	[280]
.100	P	--	--	3,820	4,960	[1,600]
0.060	Pb	--	[120]	3,270	3,260	[1,000]
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	--	--	[480]	[520]	[1,000]
0.050	Sb	--	--	--	[110]	[89]
0.050	Se	--	[120]	[200]	[200]	[200]
0.100	Si	--	[520]	126,000	93,700	12,800
1.000	Sn	--	--	[2,700]	[2,900]	[6,300]
0.005	Sr	--	[10]	161	120	[30]
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	--	[16]	2,740	2,760	1,430
0.250	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.015	V	--	--	[58]	[65]	[57]
0.500	W	--	--	--	--	--
0.010	Y	--	--	[77]	[82]	[28]
0.020	Zn	--	--	[280]	[310]	[240]
0.025	Zr	--	--	3,420	3,320	[360]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit	Multiplier=	1762.6	1964.5	1844.3		
	ALO#=	99-1892 @2	99-1892-DUP @2	99-1893 @2		
	Client ID=	C106-OH-8	C106-OH-8	C106-OH-8B1		
	Run Date=	7/9/99	7/9/99	7/9/99		
(ug/mL)	(Analyte)	ug/g	ug/g	ug/g		
0.015	Ag	285	327	719	--	--
0.060	Al	38,600	31,600	6,210	--	--
0.080	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	384	324	[110]	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.100	Ca	16,200	8,920	2,680	--	--
0.015	Cd	[38]	[37]	[33]	--	--
0.100	Ce	[190]	[310]	[210]	--	--
0.025	Co	--	--	[90]	--	--
0.020	Cr	[260]	[280]	392	--	--
0.015	Cu	[130]	[140]	[200]	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	74,200	85,300	472,000	--	--
0.025	La	[74]	[94]	[54]	--	--
0.005	Li	[14]	[24]	[23]	--	--
0.100	Mg	2,490	2,930	[700]	--	--
0.005	Mn	1,890	1,850	3,110	--	--
0.030	Mo	--	--	--	--	--
0.100	Na	160,000	183,000	15,000	--	--
0.100	Nd	[200]	[290]	[210]	--	--
.100	P	[1,300]	[1,500]	[1,200]	--	--
0.060	Pb	1,140	1,650	1,470	--	--
0.300	Pd	--	--	[710]	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	[200]	[270]	1,590	--	--
0.050	Sb	--	[130]	[150]	--	--
0.050	Se	[120]	[180]	[300]	--	--
0.100	Si	134,000	102,000	22,200	--	--
1.000	Sn	--	--	[8,000]	--	--
0.005	Sr	182	156	[38]	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	1,810	2,010	1,040	--	--
0.250	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.015	V	[40]	[48]	[59]	--	--
0.500	W	--	--	--	--	--
0.010	Y	[37]	[44]	[24]	--	--
0.020	Zn	[110]	[120]	[340]	--	--
0.025	Zr	651	1,780	[110]	--	--

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 ---Hg Report

WO/Project: W48486/29953
Client: G. Lumetta

ACL Numbers: 99-01888 and 99-01892
ASR Number 5397

Procedure: PNNL-ALO-131, "Mercury Digestion"
PNNL-ALO-201, "Mercury Analysis"

Analyst: J. J. Wagner

Digestion Date: October 21, 1999 **Analysis Date:** October 27, 1999

M&TE: Hg system (WD14126); Mettler AT400 Balance (360-06-01-029) See Chemical Measurement Center 98620 RIDS for Hg File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: Jerry Wagner

Approval: [signature] **Date** 11-5-99

Final Results:

The samples were analyzed by cold vapor atomic absorption spectrophotometry for inorganic mercury as specified in ASR 5397. The solids samples were diluted an additional 250 to 500-fold following sample digestion per procedure ALO-131. The mercury concentration results are presented in the table below.

Lab ID	Solid Sample ID	Solids Grams	Solids Dig Fctr	Solids Anal Fctr	Hg ug/g
99-1888 PB	Solids Process Blank	0.1153	216.8	1	<0.043
99-1888	C106-AQ-8	0.1029	243.0	500	369
99-1888 Dup	C106-AQ-8 Dup	0.0876	285.4	500	332
	RPD(%)				11%
99-1892	C106-OH-8	0.1253	199.5	250	153
99-1892 Dup	C106-OH-8 Dup	0.1454	171.9	250	121
	RPD(%)				23%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
"Sample weight" used for the process blank is an average weight of the samples.

Notes:

1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) The low calibration standard is defined as the estimated quantitation limit (EQL) for the reported results and assumes 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.

Q.C. Comments:

Following are results of quality control checks performed during Hg analyses. In general, quality control checks met the requirements of the governing QA Plan.

Working Blank Spike/Process Blank Spike: Process Blank Spike recovery is 112%, well within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample: A matrix spike was prepared for the samples submitted under this ASR. However, the concentration of the matrix spike processed and analyzed with this batch of sample was too low in concentration relative to the high concentration of mercury in the samples measured. As a result, matrix spike recovery could not be assessed.

Duplicate: Except for one duplicate that demonstrated an RPD of 23%, RPDs were within the acceptance criteria of 20%.

System Blank/Processing Blanks: A system blank was process during the analysis of the sample. All reportable sample concentrations were many times greater than that measured in the system blank or in the processing/dilution blank.

Quality Control Calibration Verification Check Standards: Over 4 mid-range verification standards were analyzed throughout the analysis run. All were within the acceptance criteria of 80% to 120% recovery for the verification standard.

Appendix C. Calculations

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: S.J. [illegible]	Date: 7/28/99	Project: 29953 BNFL Support

Subject: C-106 Washing/Leaching Test: Data Work-up

We will use aluminum for example calculations.

Washing with 0.01 M NaOH

23.4777 g of C-106 solids used (2.14B)

Weight of Wash Solutions:

C106-AQ-3	102.7565	g	(2.25B)
-5	94.0009	g	(2.42B)
-7	125.9229	g	(2.59B)
Total	322.6803	g	

Sample C106-AQ-9 was a composite of these three wash liquids.

Initial wt. sample:

3.1842	(2.75B)
2.9249	(2.77B)
3.9146	(2.79B)
10.0237	

Initial gross wt. C106-AQ-9: $10.0237 + 6.4782 = 16.5019$ g
on 5/10/99 — 6.4782 = tare wt. vial

Gross Sample wt. at time of analysis: 15.8656 g

$15.8656 - 6.4782 = 9.3874$ g sample

We will need to adjust for this weight loss... (which is assumed to be due to evaporation.

ICP analysis of C106-AQ-9 gave a value of 75.9 mg Al/g.

Adjusting:

$$\frac{(75.9\ \text{mg Al/g})(\cancel{15.8656}\ 9.3874\ \text{g})}{10.0237\ \text{g}} = 71.1\ \text{mg Al/g}$$

$\therefore$ Al in wash solution $= (71.1\ \text{mg Al/g})(322.6803\ \text{g}) = 22943$ mg Al

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: S.J. [illegible] | Date: 8/2/99 | Project:

Subject: C106 Wash/Leach

Sample C106-AQ-8 → "Fraction A" nonmagnetic solids → 2.0286 g (2.69 A)

ICP analysis of C106-AQ-8:

Na_2O_2 Fusion	→ 62800 ug/g	Mean	62925 (11% uncertainty)
Na_2O_2 Fusion DUP	→ 54200 ug/g		
KOH Fusion	→ 70500 ug/g	Std. Dev.	6712
KOH Fusion DUP	→ 64200 ug/g		

Al in Fraction A : (2.0286 g)(62925 ug/g) = 127650 ug Al

Sample C106-AQ-8B1 → "Fraction B" magnetic solids stuck to stir bar

0.4608 g of solids collected from acid diss. procedure.
0.1014 g solids initially on stir bar (note: this value seems suspect because visually there appeared to be more than this).

ICP analysis of C106-AQ-8B1:

Na_2O_2 Fusion	→ 6950 ug/g	Mean	7360 ug/g
KOH Fusion	→ 7770 ug/g	Std. Dev.	580

Al in Fraction B : (0.4608 g)(7360 ug/g) = 3391 ug Al

Total Al in washed solids = 127650 + 3391 = 131041 ug

$$\boxed{\text{Al Conc. in Washed solids} = 131041\ \text{ug} / (\underbrace{2.0286}_{\text{Frac A}} + \underbrace{0.1014}_{\text{Frac B}})\ \text{g} = 61522\ \text{ug/g}}$$

Using the summation method to determine Al concentration in the initial sludge ~~solids:~~ sample:

$$\frac{22943 + 131041\ \text{ug}}{23.4777\ \text{g sample}} = \boxed{6559\ \text{ug Al/g solids}}$$

Prepared By: M. J. Burnette (signature) | Date: 8/2/99 | Project:

Subject: C106 Washing / Leaching

Leaching With 3 M NaOH

24.7022 g of C-106 Solids used (3.14B)

wt. leach solution C106-OH-3 = 104.0738 g (3.25B)

wt. wash solutions: C106-OH-5 = 91.7258 g (3.42B)
C106-OH-7 = 123.9516 g (3.59B)
Total = 215.6774 (3.70A)

Sample C106-OH-9 was a composite of C106-OH-5 + -7.

Sample C106-OH-3A (leach solution):

Tare vial = 6.9483
Gross wt = 23.4416
Initial wt. sample = 16.4933 g (on 5/4/99)

Gross wt. at time of analysis = 17.7312 g (6/4/99)

17.7312 − 6.9483 = 10.7829 g wt. sample at time of analysis.

ICP analysis → 765 ug/g C106-OH-3A
791 ug/g C106-OH-3A DUP

Mean: 778 ug/g
Std. Dev: 18

Adjusting:

$$\frac{(778\ \mu g/g)(10.7829\ g)}{16.4933} = 509\ \mu g/g\ \text{Al in leach solution}$$

∴ Al in leach solution = (509 ug/g)(104.0738 g) = 52974 ug Al

Sample C106-OH-9 (wash solution):

Initial wt. sample = 10.0633 g
Sample wt at time of analysis = 9.4327 g

see bottom of p. 20 in test plan

ICP analysis → 66.6 ug Al/g

Adjusting: (66.6 ug/g)(9.4327 g) / 10.0633 g = 62.4 ug/g

∴ Al in wash solution = (62.4 ug/g)(215.6774 g) = 13458 ug Al

Prepared By: S.J. Smith | Date: 8/2/99 | Project:

Subject: C106 Wash/Leach

Sample C106-OH-8 ⇒ "Fraction A" Nonmagnetic solids → 6.5066 g (3.684)

ICP analysis of C106-OH-8:

Na_2O_2 Fusion	: 29500	Mean : 32000 ug/g
Na_2O_2 Fusion DUP	: 28300	Std Dev : 4607
KOH Fusion	: 38600	
KOH Fusion DUP	: 31600	

∴ Al in Fraction A = (6.5066 g)(32000 ug/g) = 208211 ug Al

Sample C106-OH-8B1 ⇒ "Fraction B" magnetic solids → 1.9464 g material stuck to stir bar

⇓

2.0338 g solids obtained from evap of acid solution.

ICP analysis of C106-OH-8B1:

Na_2O_2 Fusion : 6810 ug/g	Mean	6510 ug/g
KOH Fusion : 6210 ug/g	Std. Dev.	424

∴ Al in Fraction B = (2.0338 g)(6510 ug/g) = 13 240 ~~ug/g~~ ug Al

$$\boxed{\text{Al conc in leached solids} = (208211 + 13240)/(6.5066 + 1.9464) = 26,198 \text{ ug/g}}$$

Using the summation method to determine the Al concentration in the initial sludge sample:

$$\frac{52974 + 13458 + 208211 + 13240}{24.7022} = \boxed{11654 \text{ ug/g sample}}$$

note: This is higher than found other washing test.

$$\% \text{ Al removed} = 100\left(\frac{52974 + 13458}{\cancel{208211+}}\right) = 23\%$$

ENGINEERING WORKSHEET

Prepared By: M.A. Lumetta | Date: 8/11/99 | Project:

Title/Subject: C106 Data Work-Up

Solubility Versus Temperature

Sample wts.

Sample ID	Vial Tare	Initial Gross wt	Initial Sample wt.	Final Gross wt.(a)	Final Sample wt.(a)	Correction Factor (c)
C106-SOL-30-1	6.4858	10.9521	4.4663	10.5470	4.0612	0.9093
-30-2	6.4995	10.7155	4.2160	9.7723	3.2728	0.7763
-40-1	6.4695	10.7522	4.2827	10.3140	3.8445	0.8977
-40-2	6.4899	10.6778	4.1879	10.1434	3.6535	0.8724
-50-1	6.4988	10.0464	3.5476	8.3734(b)	1.8746	0.5284
-50-2	6.4985	10.8641	4.3656	10.1754	3.6769	0.8422

(a) Wt. at time analytical work begun.
(b) Evidence that sample had leaked.
(c) = Final sample wt / Initial Sample wt.

Cesium Behavior (an Example Case)

Uncorrected Values; Corrected Values

C106-SOL-30-1 → 4.57 µCi/g × 0.9093 = 4.16 µCi/g
-2 → 4.62 µCi/g × 0.7763 = ~~3.5~~ 3.59 µCi/g M.A.L. 8/11/99
→ Mean = ~~4.03~~ 3.87 µCi/g M.A.L. 8/11/99; Std. Dev. = 0.40

C106-SOL-40-1 → 5.31 µCi/g × 0.8977 = 4.77 µCi/g
-2 → 4.86 µCi/g × 0.8724 = 4.24 µCi/g
→ Mean = 4.50; Std. Dev. = 0.37

C106-SOL-50-1 → 5.85 µCi/g × 0.5284(d) = 3.09 µCi/g
-2 → 5.96 µCi/g × 0.8422 = 5.02 µCi/g
→ Mean = 4.06; Std. Dev. = 1.36

(d) This value is suspect based on footnote (b) above.

30 → 40°C : 100 (4.50 − 3.87)/3.87 = 16% increase in ^{137}Cs concentration

30 → 50°C : 100 (5.02 − 3.87)/3.87 = 30% increase in ^{137}Cs concentration

Discarding C106-SOL-50-1 measurement

Appendix D. Statistical Analysis of the Data

Statistical analyses were performed on the data included in this report. In general, simple summary statistics were provided throughout that included estimates of averages (means), standard deviations (std. dev.) and percent relative standard deviations (%RSD = 100*std. dev./mean). More specific statistical analyses included:

- Solubility versus Temperature Study Regression Analyses
- Solubility versus Temperature Study Tests for Changes due to Temperature
- Washing and Leaching Studies Estimates of Uncertainty for analyte concentrations in the washed and untreated solids and the percent removal

For all of the following analyses it should be kept in mind that all data in each study are taken from one run of the experiment on a single sample. This means that they provide no sense of the additional uncertainty that would result from running different samples or from repeating the experiment on similar samples. The only sources of variability present in these studies are subsampling variability and measurement variability. Consequently, the uncertainty statements developed in this report are likely an underestimate of the variability that will be experienced in the real world application of these conclusions.

Solubility Versus Temperature Study Regression Analyses

The regression analyses performed here are a quantitative assessment of the nature of the relationship between analyte concentrations and temperature. Since there are only three temperature points (30, 40, and 50°C), the maximum model that can be fit as a function of temperature is a quadratic. The two concentration values per temperature provide for estimating subsampling and measurement uncertainty and for testing the lack-of-fit of the linear regression. The general approach taken was to first fit and test a linear regression; i.e., is a linear regression statistically better than no model. This was followed by a test of the lack-of-fit of the linear regression model, or equivalently in this case, whether adding the quadratic term would be useful in describing the solubility-temperature relationship.

The following analyses were done using the evaporation-adjusted concentrations from Tables 1, 2 and 3 with the exception that the C106-SOL-50-1 data were not used because of suspected sample leakage that would render its results unrepresentative. The data were taken from the original Excel® spreadsheet and have additional digits compared to the formatted table values. These analyses were done using the Statistical Analysis System (SAS Institute Inc. Cary, NC).

Table D.1 presents the results of the regression analyses. Included are the estimates of the intercept and slope for the linear regression. Also included are the probabilities (p-values) for the test of the linear regression and for the test of the lack-of-fit of the linear regression. A significance level of 0.10 was used. Those analytes that have a significant linear regression will have a simple linear p-value < 0.10. Those analytes that have a significant lack-of-fit from the linear regression will have a lack-of-fit/quadratic p-value < 0.10. Those analytes that did not have a significant linear regression or had a significant lack-of-fit are grayed out in the table to indicate that their linear regression estimates are not considered useable.

Table D.1. C-106 Solubility Versus Temperature
Data Regression Analysis

Analyte	Estimated Intercept	Estimated Increase per °C	p-value Simple Linear	p-value Lack-of-Fit /Quadratic
Cesium-137	2.14	0.0583	0.055	0.888
Strontium-90	0.0277	0.00160	0.390	0.363
Technetium-99	0.00149	-0.00000174	0.925	0.976
Total Alpha	-0.000810	0.0000750	0.130	0.665
Ag	-0.429	0.0554	0.163	0.858
Al	376	-5.79	0.465	0.711
Ba	0.131	0.00298	0.117	0.284
Ca	1.99	0.0517	0.239	0.175
Cr	-2.75	0.138	0.002	0.739
Cu	-0.601	0.0419	0.024	0.317
Fe	0.509	0.0158	0.855	0.284
Mo	0.265	0.0121	0.122	0.370
Na	7,090	235	0.106	0.455
Ni	-1.48	0.0815	0.013	0.220
P	11.1	0.794	0.052	0.274
Si	3.82	0.648	0.052	0.188
Li	0.0540	0.00219	0.172	0.305
U	13.8	0.254	0.082	0.672
TOC	5,018	26.3	0.278	0.612
TIC	1,232	9.57	0.316	0.815
Cl^-	247	3.82	0.047	0.560
$C_2O_4^{2-}$	10,913	258	0.014	0.672

Radionuclides are in units of µCi/g; all other components are in units of µg/g. The regression estimates are grayed-out (judged unusable) if: the estimated increase is not significantly different from zero (linear p-value > 0.1) or the lack-of-fit of the linear regression is significant (lack-of-fit p-value < 0.1).

Plots for all analytes in Table D.1 are included. The following plotting symbols are used for the data:

- filled diamond— data that was ∃10-times the detection limit
- empty diamond— data that was < 10-times the detection limit
- descending triangle— detection limit

The plots also show the linear regression with a solid line, 90% confidence intervals on the mean with dashed lines, and the quadratic regression with a dotted line. Occasionally a confidence interval is so wide it goes off the plot.

The aliquot variability is surprising for some analytes and, along with small sample numbers, leads to "non-significant" tests for some analytes that appear to show a relationship. Nine of the analytes showed linear p-values < 0.1 and quadratic p-values > 0.1. Three of these had all of their data reported as "()", but none had DL data included.

Solubility Versus Temperature Study Tests for Changes Due to Temperature

Concentration changes in the Solubility versus Temperature study are expressed as the concentration change at each temperature relative to the concentration at 30°C. This is calculated as $100*(C_T-C_{30})/C_{30}$ or equivalently as $100*(C_T/C_{30})-100$, where C_T is the average concentration at temperature = T and C_{30} is the average concentration at 30°C. Table 4 shows these estimates of the change in concentrations (solubility) for detected analytes for all the unadjusted data and Table 5 shows them for the adjusted data without C106-SOL-50-1 (for reasons given previously).

The following method was used to indicate whether the reported changes were significantly different from 0 or could instead simply be an artifact of subsampling and measurement uncertainty, especially with so few data points. There is insufficient data to estimate the variability at any one temperature with any confidence, so a pooled estimate of uncertainty was obtained by pooling the %RSDs at the three temperatures (or only two temperatures for the evaporation adjusted data with the one sample removed). This assumes the RSDs are relatively constant at each temperature. This result in turn was used as input to standard propagation-of-errors calculations for the variance of the estimation formula $100*(C_T/C_{30})-100$. This results in an estimate of the standard deviation of the % Change as $C_T/C_{30}*\text{sqrt}(2)*\text{Pooled }\%\text{RSD}$. The estimate of the percent change at each temperature was then divided by the standard deviation estimate at that temperature. This ratio was compared to a one-sided 90% t-statistic and any ratio that was larger than the appropriate t-statistic is considered strong evidence of a positive change in solubility. These significant changes are bold-faced in Tables 4 and 5. For the unadjusted data there were 3 degrees of freedom in the estimate of variability and the t-statistic value was 1.64. For the adjusted data there were only 2 degrees of freedom in the estimate of variability and the t-statistic value was 1.89.

Washing and Leaching Studies–Estimates of Uncertainty for Analyte Concentrations in the Washed and Untreated Solids and the Percent Removed

The ability to derive estimates of uncertainty for the values reported in Tables 9 and 13 was even more hampered than the percent change estimates discussed in the previous section. The calculation of the concentrations in Washed Solids and Original Sample were made using a number of sample weights and fraction constituent amounts. Only one of these inputs, namely the non-magnetic fraction of the washed solids, had duplicate data that could be used to estimate subsampling and measurement variability. The percent removed calculation in these two tables is even more problematic because of the use of even more terms and because it is the ratio of two other estimates.

In an attempt to get at least some handle on the uncertainty of these estimates the following approach was taken:

- Treat all weights used in the estimation formulas as constants (without error) under the assumption that their uncertainties are much smaller than the uncertainties in the concentration measurements and can be safely ignored.
- Present a "pseudo" 95% confidence interval for at least one value of a %RSD that is assumed to be equal for all measurements that were used in any equation. A %RSD of 10 was chosen as the initial candidate as it appeared to be somewhat lower than the median of %RSDs seen in this study and seems to represent a reasonable lower bound. This reasonable lower bound on the uncertainty can be adjusted to determine the effects of other %RSD values by multiplying the "pseudo" 95% confidence interval values by the ratio of any other practicable %RSD divided by 10.

As input to the "pseudo" 95% confidence intervals it was necessary to again use propagation of errors techniques to develop approximate standard deviations. These standard deviations were then multiplied by 2 (close to 1.96 from a standard normal distribution) to give the confidence interval half widths.

For concentrations in Washed Solids and Original Sample, the calculations are simple additions of fraction amounts divided by the sum of the corresponding fraction weights. The following propagation-of-error rules were used to develop propagation-of-errors formulas for their standard deviations:

- Variance of a mean is the variance of the measurement/n (the number of values used in the mean)
- The variance of a sum is the sum of the variances
- Constants (sample weights in this case) carry through.

This resulted in a general form for these two concentration estimates as:

$$\text{Std.Dev.} = \text{sqrt}\,(\Sigma_f(\text{var}(f)/n_f))/\text{weights},$$

where f= each fraction used in the calculation of the concentration. Each var(f) term in the propagation-of-errors formula can be replaced, by definition, with $(\text{mean}_f*\%\text{RSD})^2$. Also, since the same %RSD is assumed for all measurements, %RSD can be factored out, resulting in the following general formula:

$$\text{Std.Dev.} = \%\text{RSD}*\text{sqrt}\,(\Sigma_f(\text{mean}_f^2/n_f))/\text{weights}$$

The actual version of this general formula used for each analyte for each concentration estimate depends on the fractions that were used to calculate it and the number of subsamples available for each fraction.

For % Removal, the calculations involve 100 times the ratio of two terms, each of which is the sum of fraction amounts. The initial standard propagation-of-errors form of the std. dev. for this ratio of two terms is:

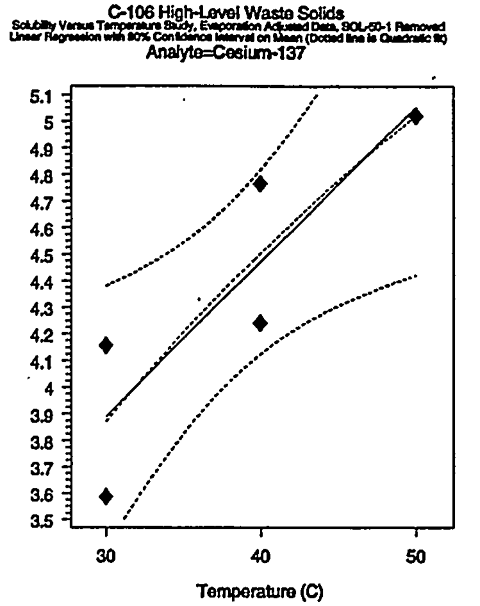
$$\text{std. dev.} = 100*\text{num}/\text{den}*\text{sqrt}(\text{var}(\text{num})/\text{num}^2 + \text{var}(\text{den})/\text{den}^2)$$

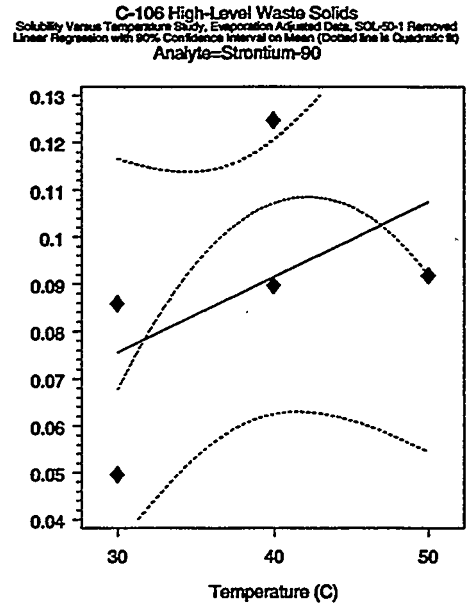
where num = the numerator term, den= the denominator term, and var() is the variance of each. Both the numerator and denominator also need to have propagation-of-errors applied to them. Again, each var() term in their propagation-of-errors formula can be replaced, by definition, with $(\text{mean}*\%\text{RSD})^2$. Also, since the same %RSD is assumed for all measurements, %RSD can again be factored out, resulting in the following general formula:

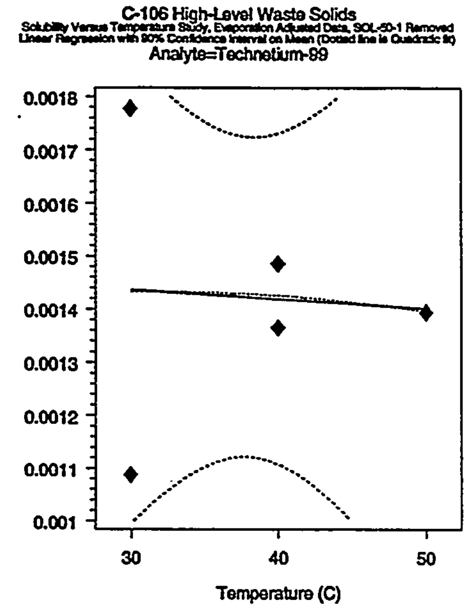
$$\text{Std.Dev.} = 100*\Sigma_f\text{mean}_f/\Sigma_d\text{mean}_d*\%\text{RSD}*$$
$$\text{sqrt}(\Sigma_f(\text{mean}_f^2/n_f)/(\Sigma_f\text{mean}_f)^2 + \Sigma_d(\text{mean}_d^2/n_d)/(\Sigma_d\text{mean}_d)^2)$$

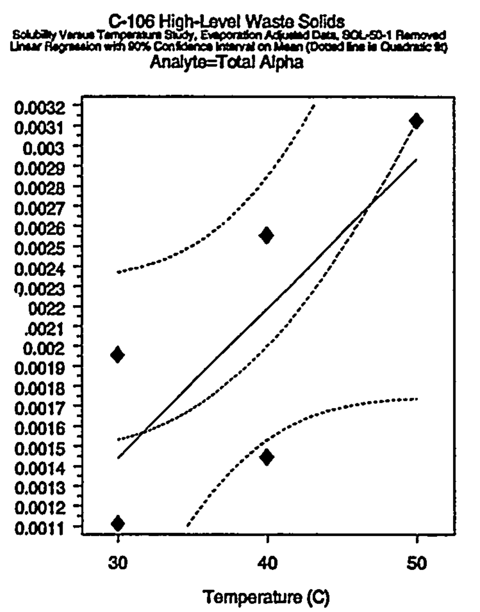
where f = each fraction used in the numerator and d = each fraction used in the denominator.

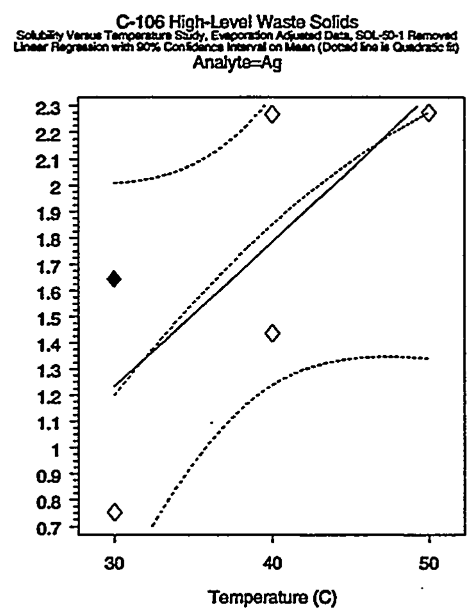
As for the concentration estimates discussed above, the actual version of this general formula used for each analyte depends on the fractions that were used to calculate the numerator and denominator and the number of subsamples available for each fraction.

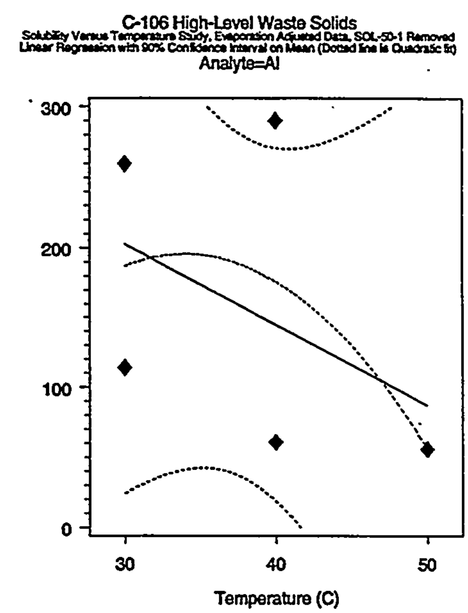

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Cesium-137
Temperature (C)

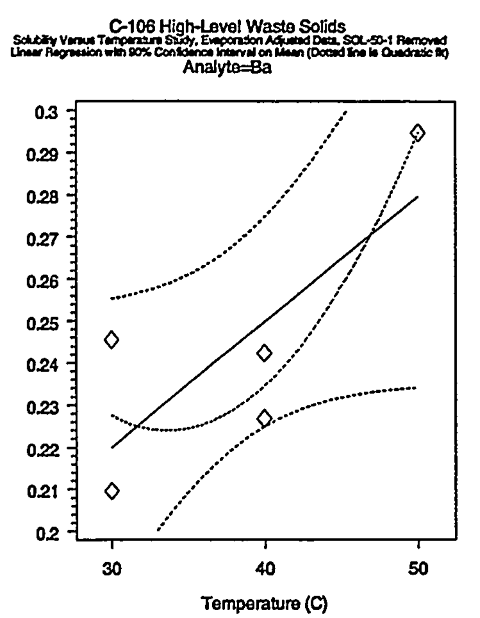

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Strontium-90
Temperature (C)

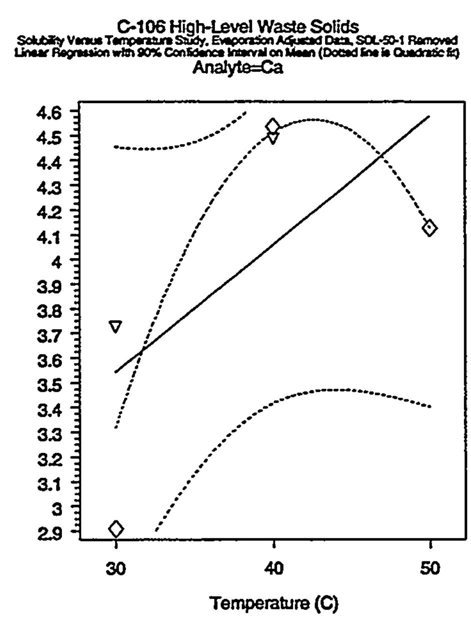

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Technetium-99
Temperature (C)

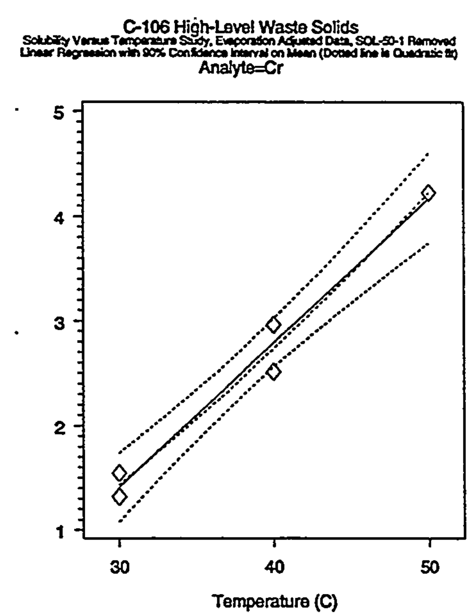

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Total Alpha
Temperature (C)


C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Ag
Temperature (C)

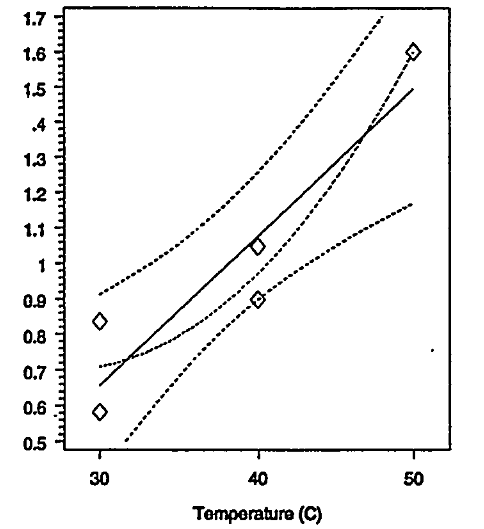

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Al
Temperature (C)


C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Ba
Temperature (C)

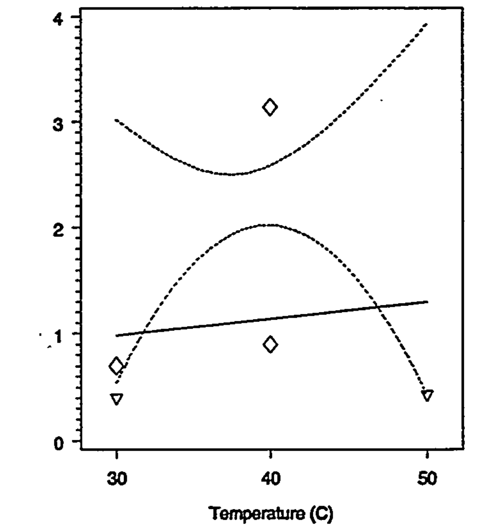

C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Ca
Temperature (C)


C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Cr
Temperature (C)

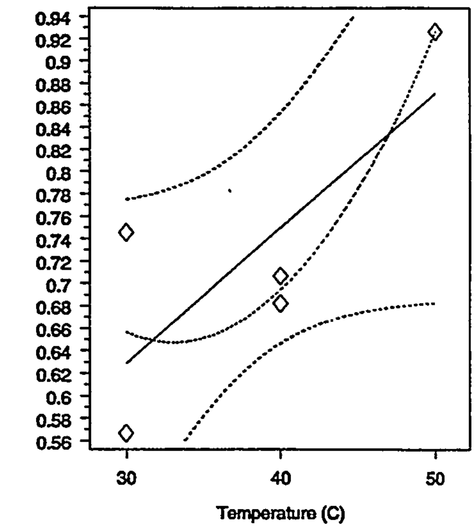
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Cu



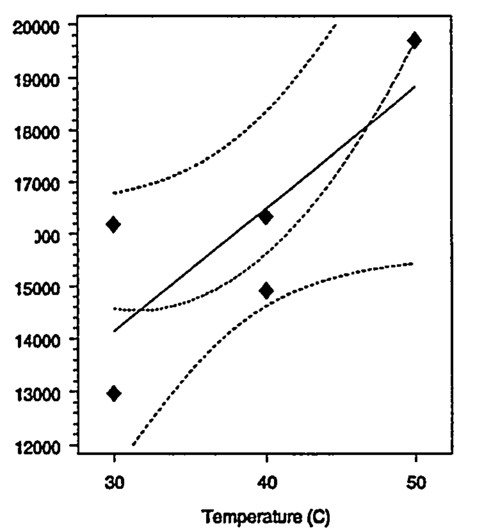
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Fe



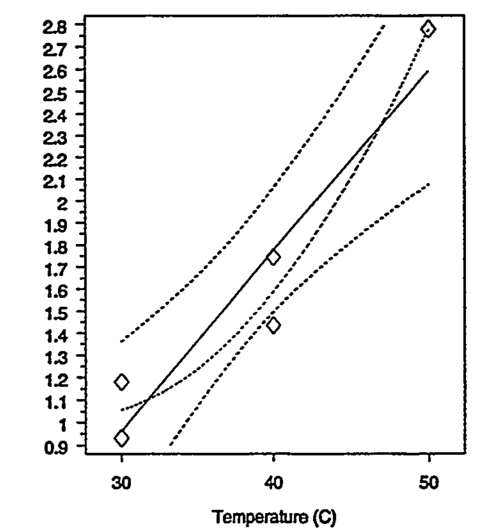
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Mo



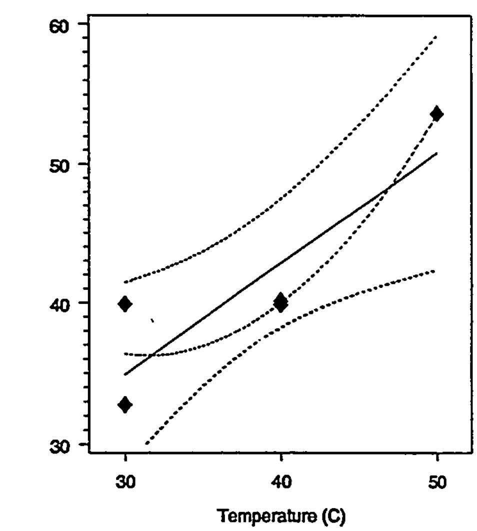
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Na



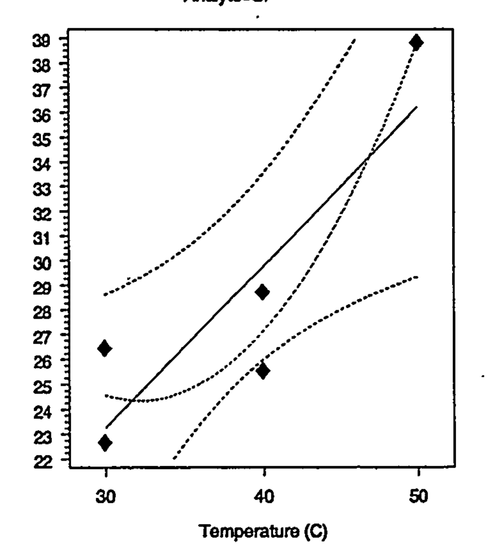
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Ni



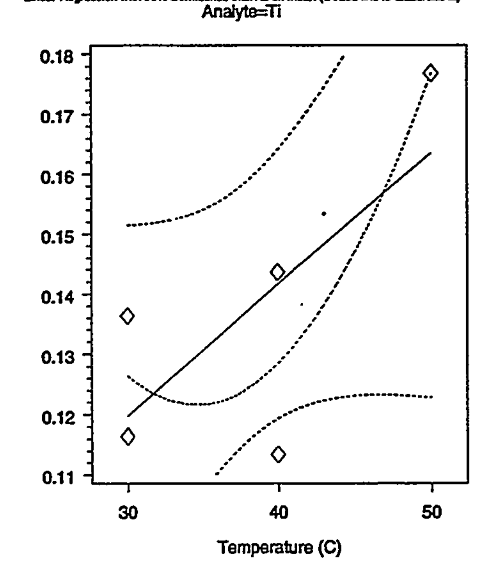
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=P



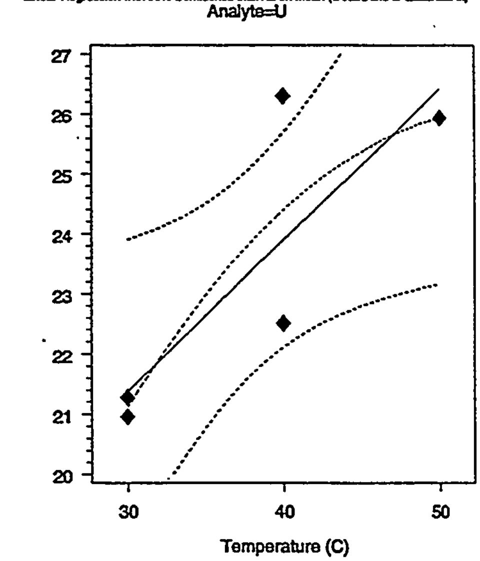
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Si



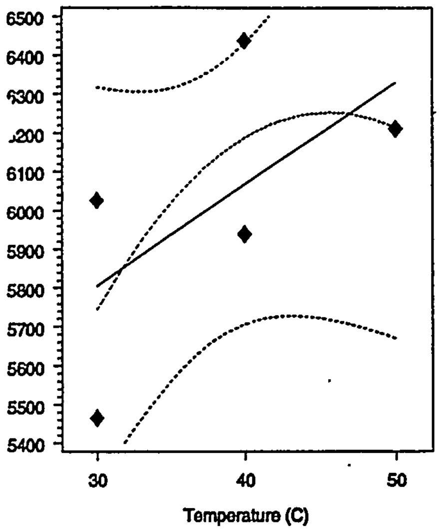
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Ti



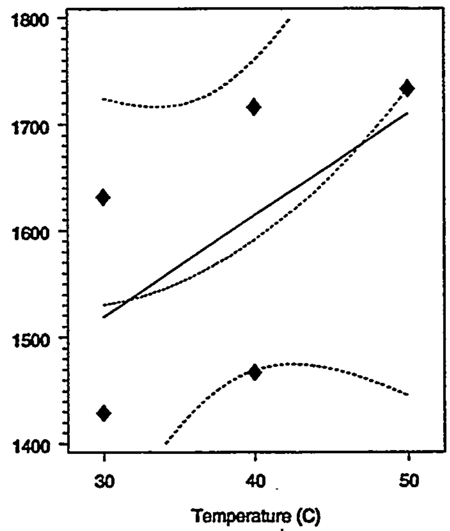
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=U



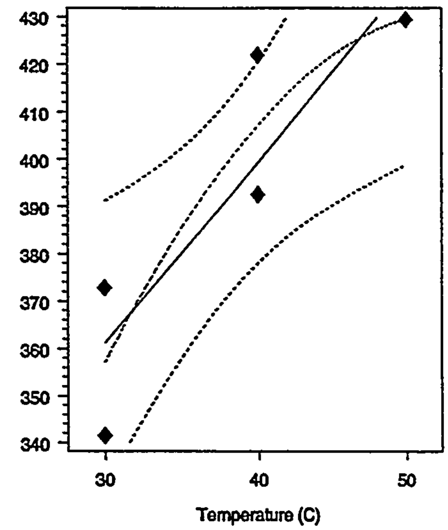
C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=TOC



C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=TIC



C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=Cl-



C-106 High-Level Waste Solids
Solubility Versus Temperature Study, Evaporation Adjusted Data, SOL-50-1 Removed
Linear Regression with 90% Confidence Interval on Mean (Dotted line is Quadratic fit)
Analyte=C2O42-