308 - - - Q200510070002

Scientific Notebook No. 711: Np Sorption on Consolidated with Simulated Waters (with and without calcium) Over a pH Range. Identified as CAL (consolidated alluvium) Series 5 and 6 - Continued in Scientific Notebook No. 733 (06/06/2005 through 08/30/2005)

LABORATORY NOTEBOOK

711

CNWRA/SwRI

NOTEBOOK NO. 711
ISSUED TO BRADLEY WERLING 210-522-6565
ON _____ 20____
DEPARTMENT _____
RETURNED _____ 20____

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RADIONUCLIDE TRANSPORT < SZ 20.06002.01.242
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6 JUNE 2005

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INVESTIGATOR	SIGNATURE	INITIALS
BRADLEY WERLING	Bradley Werling	BAW
Marla Roberts	Marla Roberts	MR

This notebook contains work for Radionuclide Transport (RT) in both the saturated zone and unsaturated zone. RT is concerned with identification of key geochemical processes that may control radionuclide transport at Yucca Mountain. Radionuclide retardation is dependent on the hydrochemistry and mineralogy along the groundwater flow paths from the proposed repository. Typical activities associated with these investigations would include conducting laboratory batch sorption experiments, collecting field samples, and analyses of field samples.

Current experimental activities are focused on laboratory batch sorption experiments that incorporate materials (groundwater and sonic corings) from the Yucca Mountain region and materials (simulated groundwater) based on the chemistry of the Yucca Mountain region materials. The objective for a sorption experiment is to determine the sorption capability (Kd or sorption coefficient) for a particular radionuclide, solid material, and solution typically over a range of pH values. Tasks typically include the following:

- Preparation of reference solutions.
- Cation (Div 01), inorganic carbon (CNWRA) and pH analyses (CNWRA) of reference solutions.
- Preparation of experimental solutions
- Liquid Scintillation Analyses (CNWRA) of experimental solutions.

Further details of these tasks are recorded in initial and in-process entries.

This scientific notebook (SN) is a continuation of SN 696. That notebook concludes with the pH analyses of groundwater that will be used in the CAL series 5 and 6 sorption experiments. The first entries in SN 711 are for the CAL series 5 and 6 sorption experiment. The initial entry for the CAL series 5 and 6 sorption experiment is in 696/170-171. Notebook 711 will start with an updated version of this initial entry.

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Initial Entry - Np sorption on consolidated alluvium with Simulated waters (with and without calcium) over a pH range. Identified as CAL (consolidated alluvium) series 5 and 6.

Jon Brown and Bradley Werling are the individuals initially assigned to work on this experiment

Objective: To investigate Np sorption on a consolidated alluvium sample using two types of simulated water (with and without calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB over a range of pHs.

Conditions:
- $\sum$Np = 200 ppb
- equilibrium with lab CO_2(g); $pCO_2 = 10^{-3.42}$
- solution volume = 30 ml
- mass of solid = 0.1 g
- M/V (g/L) = 3.33

Samples
- 19PB Simulated water with Ca - 696/14 and 32
- 19PB Simulated water without Ca – 696/14 and 32
- Consolidated alluvium sample (<35 mesh size fraction) NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66.

Reagents
Neptunium Spike # 61A-1 (369/100) - 60 ppm Np-237 at pH of 5.63
pH buffer 4 (Fisher SB98 lot # 041614)
pH buffer 7 (Fisher SB108 lot # 044065)
pH buffer 10 (Fisher SB 116 lot # 044060)
Ultima-Gold AB liquid scintillation cocktail (Packard 6013309 lot # 91-050201)
0.02 M HNO_3 (SN 696/29-30)
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Equipment:
Orion meter 920A (sn 039518)
Thermo Orion combination electrode 8103BN (ID 3G)
New Brunswick Scientific Gyratory shaker model G33 (sn 290127990)
Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
Mettler AE240 balance (sn 101237)
Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
Stir Plate

Supplies:
Pipettes and tips
Repipettor for transfer of scintillation cocktail
Polycarbonate centrifuge tubes with caps (50 ml capacity)
7 ml scintillation vials
Weighing paper/boats
10 mL microbeakers
Plasticware and glassware as needed
Squirt bottle for water
Stir bars
Kimwipes

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Basic Outline of Procedure
- Equilibrate simulated water sample with laboratory CO_2.
- Measure the pH of simulated water samples.
- Add solids to experimental solutions.
- Add simulated water to experimental solutions.
- Add pH adjustment solutions to experimental solutions.
- Add neptunium spike to experimental solutions.
- Place experimental solutions on activated gyrator
- During the experiment, completely remove the experimental solution caps during the regular business hours. Loosely cap otherwise.
- Analyze aliquots of experimental solution for pH and neptunium concentration (LSA)

Equilibrium of Simulated Water Samples

Initial Entrys: 696/170-171 + 711/2-3

Aeration of simulated ground waters conducted since Feb 23, 2005 696/14 + 32

pH Analyses of Simulated Water Samples

Initial Entrys: 696/170-171 + 711/2-3

Conducted ~~6 June~~ ~~BW 6-6-05~~ June 05 696/199

Addition of solids to Exp Solns CAL series 5+6

Initial Entries 696/170-171 + 711/2-3

Conducted on 13 May 05 - 696/172-174

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Challange of Balance (Target = 20.0001g)
At start of day = 20.0001g
At end of day = 20.0002g

Addition of Simulated Water to Experimental Solutions
CAL series 5 + 6

Initial Entry: 696/170-171 and 711/2-3

About 30 mL (10 mL pipet) of appropriate simulated water was added to the experimental solution pc tube. The mass was recorded - 711/4-5
19PB Simulated water with Ca used for CAL series 5
19PB Simulated water without Ca used for CAL series 6

SAMPLE ID	Mass(g) of tube + solid + sim water
CAL5-Np1	53.1652
CAL5-Np2	53.0259
CAL5-Np3	53.1417
CAL5-Np4	53.0458
CAL5-Np5	52.8339
CAL5-Np6	52.9243
CAL5-Np7	52.7298
CAL5-Np8	52.2943
CAL5-Np9	52.6110
CAL5-Np10	52.5695
CAL5-Np11	52.7011
CAL5-Np12	52.6784
CAL6-Np1	52.5446
CAL6-Np2	52.4533
CAL6-Np3	52.3547
CAL6-Np4	52.3399
CAL6-Np5	52.3186

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CONT

SAMPLE ID	Mass(g) of tube + solid + sim water
CAL6-Np6	52.2161
CAL6-Np7	52.3224
CAL6-Np8	52.4740
CAL6-Np9	52.5104
CAL6-Np10	52.3146
CAL6-Np11	52.3437
CAL6-Np12	52.2549

Addition of pH adjustment solutions to CAL series 5 + 6 Exp Solns

Initial Entries: 696/170-171 and 711/2-3

Adjusted pH by ~~addit~~ ~~6-6~~ BW ~~6-6~~ BW 6-7-06 adding acid (0.4M HNO_3 from 696/195) or base (0.1M NaOH from 696/30) according to the table below:

ID	Vol (uL) of 0.4 M HNO3	Vol (uL) of 0.1M NaOH
Np1	800	na
Np2	600	na
Np3	500	na
Np4	400	na
Np5	300	na
Np6	200	na
Np7	178	na
Np8	140	na
Np9	105	na
Np10	10	na
Np11	na	517
Np12	na	1622

Assumptions
1. Water at equilibrium with lab CO2(g) = -3.42 (log fugacity)
2. Experimental solutions approximately 30 mL volume
3. GFW of NO3- = 62.0049, GFW of NA = 22.9898

The acid conc is higher than previous exps (0.4M vs 0.1N). We are trying to experimentally determine.

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the approximate amounts to add to create a pH range of 6.5 to 9. These values were chosen based on the CAL series 3+4 and Mort 5+6 values (696/94 + 696/108-109). In order to minimize ~~to~~ BW 6-7-05 the volume of acid added - a stronger conc was used. The "ID" in the table represents the suffix for CAL series 5+6 (CAL5 + CAL6). Used adjustable eppendorfs 10-100µL and 100-1000µL. Recorded mass after pH adjusting solutions added ~~6~~ BW 6-7-05 711/6-7

SAMPLE ID	Mass (g) after pH adjustment	Mass (g) after Np Spike
CAL5 Np1	53.9696	54.0678
CAL5 Np2	53.6252	53.7228
CAL5 Np3	53.6407	53.7324
CAL5 Np4	53.4373	53.5304
CAL5 Np5	53.1242	53.2209
CAL5 Np6	53.1228	53.2203
CAL5 Np7	52.9030	52.9954
CAL5 Np8	52.4348	52.5312
CAL5 Np9	52.7066	52.8034
CAL5 Np10	52.5794	52.6761
CAL5 Np11	53.2114	53.3077
CAL5 Np12	54.2890	54.3841
CAL6 Np1	53.3457	53.4397
CAL6 Np2	53.0458	53.1429
CAL6 Np3	52.8513	52.9482
CAL6 Np4	52.7358	52.8326
CAL6 Np5	52.6162	52.7128
CAL6 Np6	52.4144	52.5106
CAL6 Np7	52.4977	52.5951
CAL6 Np8	52.6127	52.7087
CAL6 Np9	52.6078	52.7001
CAL6 Np10	52.5243	52.4194

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SAMPLE ID	Mass (g) after pH adjustment	Mass (g) after Np spike
CAL6 Np11	52.8517	52.9488
CAL6 Np12	53.8577	53.9533

Addition of Neptunium Spike to CAL series 5+6 Experimental Solutions

Initial entries: 696/170-171 and 711/2-3

Np Spike #61A-1 (369/100)

Added 100µL (100µL eppendorf pipet) to each exp. soln and recorded mass (711/6-7)

Running of CAL series 5+6 Experimental Solns

Initial Entries: 696/170-171 and 711/2-3
Each exp soln (711/6-7) was vortexed for about 30 secs and then placed on activated gyrator with loose caps (RPM ~ 100)

8 June 05 BAW

Running of CAL series 5+6 Sorption Experiment

Initial Entries 696/170-171 + 711/2-3
Experimental solutions (711/6-7) uncapped and placed on activated gyratory shaker (RPM ~ 100) at 0855. At 1652 exp solutions were tightly capped and each one vortexed for about 10 seconds. Exp solutions loosely capped and placed on activated gyratory shaker again (RPM ~ 100).

6-9-05 BW

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Running of CAL series 5+6 Sorption Experiment

Initial Entries: 696/170-171 + 711/2+3
Exp Solns from 711/6-7

Followed procedure on 8 Jun 05 (711/7)

Uncapped at 0903 Capped at 1655

~~9 BW 6-10-05~~ 10 June 05 BAW

Running of CAL series 5+6 Sorption Experiment

Initial Entries: 696/170-171 + 711/2-3
Exp Solutions from 711/6-7

Followed procedure on 8 Jun 05 (711/7)

Uncapped at 12:15 Loosely capped at 1657

11 JUNE 05 BAW

Running of CAL series 5+6 Sorption Experiment

Initial Entries: 696/170-171 + 711/2-3
Exp Solns from 711/6-7

Followed procedure on 8 Jun 05 (711/7)

Uncapped at 13:15 Loosely capped at 19:05

6-11-05 BAW

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Initial Entry – CAL Series 7, 8, 9, and 10 Sorption Experiments – Np-237 on consolidated alluvium with Simulated waters (with and without calcium) over a pH range performed in duplicate.

Personnel initially assigned to task: Bradley Werling

Objective: To investigate Np sorption on a consolidated alluvium from NC-EWDP-19PB using two types of simulated water (with and without calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB over a range of pHs.

Conditions:
- $\sum$Np = 200 ppb
- equilibrium with lab CO_2(g); $pCO_2 = 10^{-3.42}$
- solution volume = 30 ml
- mass of solid = 0.1 g
- M/V (g/L) = 3.33

Samples
19PB Simulated water with Ca - 696/14 and 32
19PB Simulated water without Ca – 696/14 and 32
NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66. (<35 mesh size fraction)

Reagents
Neptunium Spike # 61A-1 (369/100) - 60 ppm Np-237 at pH of 5.63
pH buffer 4 (Fisher SB98 lot # 041614)
pH buffer 7 (Fisher SB108 lot # 044065)
pH buffer 10 (Fisher SB 116 lot # 044060)
Ultima-Gold AB liquid scintillation cocktail (Packard 6013309 lot # 91-050201)
0.02 M HNO_3 (SN 696/29-30) for LSA matrix
0.4 M HNO3 (696/195) for pH adjustment
0.1 M NaOH (696/30) for pH adjustment
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Equipment:
Orion meter 920A (sn 039518)
Thermo Orion combination electrode 8103BN (ID 3G)
New Brunswick Scientific Gyratory shaker model G33 (sn 290127990)
Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
Mettler AE240 balance (sn 101237)
Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
Stir Plate

Supplies:
Pipettes and tips
Repipettor for transfer of scintillation cocktail
Polycarbonate centrifuge tubes with caps (50 ml capacity)
7 ml scintillation vials
Weighing paper/boats
10 mL microbeakers
Plasticware and glassware as needed
Squirt bottle for water
Stir bars
Kimwipes

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Basic Outline of Procedure

- Equilibrate simulated water sample with laboratory CO_2.
- Measure the pH of simulated water samples.
- Add solids to experimental solutions.
- Add simulated water to experimental solutions.
- Add pH adjustment solutions to experimental solutions.
- Add neptunium spike to experimental solutions.
- Place experimental solutions on activated gyrator
- During the experiment, completely remove the experimental solution caps during the regular business hours. Loosely cap otherwise.
- Analyze aliquots of experimental solution for pH and neptunium concentration (LSA)

Addition of solids to CAL Series 7, 8, 9, and 10 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To prepare the experimental solutions by adding the solid substrate.

Initial Entry: 711/9-10

Sample info NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66. (<35 mesh size fraction)

Procedure

Four sets of twelve polycarbonate centrifuge tubes were labeled as follows: CAL7-Np1 to 12, CAL8-Np1 to 12, CAL9-Np1-12, and CAL10-Np1-12. The mass of each tube (with cap) was measured and recorded on 711/11 to 12. An individual experimental tube was uncapped and a funnel made from weighing paper was inserted in the top. Approximately 0.1 g of solid was transferred to a piece of weighing paper that was tared on the balance. The solid was transferred into the experimental tube and the tube capped. This "solid addition" step was repeated until all experimental tubes contained consolidated alluvium. The mass of each experimental tube was measured again and recorded on 711/11 to 12.

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SAMPLE ID	Mass(g) of Empty tube	Mass(g) of tube + solid
CAL7-Np1	22.1925	22.2927
CAL7-Np2	21.9720	22.0720
CAL7-Np3	22.0031	22.1033
CAL7-Np4	22.4663	22.5664
CAL7-Np5	22.4850	22.5856
CAL7-Np6	22.0423	22.1424
CAL7-Np7	22.1750	22.2747
CAL7-Np8	22.4743	22.5736
CAL7-Np9	21.9193	22.0197
CAL7-Np10	22.2300	22.3302
CAL7-Np11	22.1289	22.2288
CAL7-Np12	22.3713	22.4714
CAL8-Np1	22.2777	22.3790
CAL8-Np2	22.0792	22.1792
CAL8-Np3	22.5760	22.6753
CAL8-Np4	22.2298	22.3297
CAL8-Np5	22.1210	22.2226
CAL8-Np6	22.1410	22.2407
CAL8-Np7	22.3828	22.4826
CAL8-Np8	22.3254	22.4257
CAL8-Np9	22.0951	22.1953
CAL8-Np10	22.2696	22.3703
CAL8-Np11	22.1089	22.2086
CAL8-Np12	22.3604	22.4616
CAL9-Np1	21.9896	22.0892
CAL9-Np2	22.0265	22.1268
CAL9-Np3	22.0846	22.1852
CAL9-Np4	22.0217	22.1224
CAL9-Np5	BAW 6-11-05 ~~22.~~ 21.9852	22.0857
CAL9-Np6	21.9552	22.0543
CAL9-Np7	22.0599	22.1604
CAL9-Np8	22.0016	22.1016

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SAMPLE ID	Mass (g) of empty tube	Mass (g) of tube + solid
CAL9-Np9	22.0243	22.1243
CAL9-Np10	22.0670	22.1677
CAL9-Np11	22.0712	22.1717
CAL9-Np12	21.9860	22.0864
CAL10-Np1	22.1140	22.2144
CAL10-Np2	21.9784	22.0787
CAL10-Np3	22.0492	22.1503
CAL10-Np4	21.9684	22.0699
CAL10-Np5	22.0506	22.1518
CAL10-Np6	22.0189	22.1193
CAL10-Np7	21.9681	22.0689
CAL10-Np8	22.2430	22.3426
CAL10-Np9	21.9691	22.0690
CAL10-Np10	22.0913	22.1917
CAL10-Np11	22.1444	22.2441
CAL10-Np12	21.9755	22.0760

Challenge of balance (Target = 20.0001g)
At start of day = 20.0002g
At end of day = 20.0002g

13 June 05 BAW

Running of CAL series 5+6 Sorption Experiment

Initial Entries: 696/170-171 + 711/2+3
Exp Solns - from 711/6-7

Followed procedure on 8 Jun 05 (711/7)

Uncapped at 11:15 Loosely capped at 17:45

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Liquid Scintillation and pH Analyses of CAL series 5 and 6 – Np-237 with Consolidated Alluvium in two types of simulated groundwater over a pH range

Bradley Werling will be performing this task

Objective: To determine the neptunium concentration and pH of experimental solutions and use in calculations for determining the sorption coefficient (Kd) under specified conditions

Initial Entry 696/170-171 and 711/2-3

Sample Info Two experiments based on consolidated alluvium substrate from NC-EWDP-19PB and two simulated waters (with and without calcium) based on 19PB shallow string groundwater.

Procedure

LSA vials (size 7 mL) were labeled for experimental solutions, blank solution, and neptunium spikes solutions. Experimental solutions were analyzed in duplicate and labeled to identify the sorption solid phase and series, the radionuclide, the experimental solution number, and the a1/b1 duplicate designator (example – CAL7-Np1a1 and CAL7-Np1b1). One simulated water blank solution was analyzed and labeled as w CA with scientific notebook page reference. The neptunium spike was analyzed in duplicate and labeled Spike 61A1-1 and 61A1-2.

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.5 mL (500µL eppendorf pipet) of 0.02 M HNO_3 added. The mass of all of the LSA vials with acid was recorded (See table 1 column 1).

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (See table 2, column 1). The tubes containing the experimental solutions were placed in a tared 30mL glass beaker when being weighed.

For experimental solutions, 0.5 mL (500µL eppendorf pipet) of the experimental solution was transferred into the appropriate LSA vial. For the simulated water blank, 0.5 mL (500µL eppendorf pipet) of simulated water was added to the LSA vial. For the neptunium spike solutions, 0.1 mL (100µL eppendorf pipet) of the spike was added into each LSA vial. The mass of all of the LSA vials was recorded after the addition of the appropriate sample solution (See table 1 column 2).

The pH of the experimental solutions was taken. Fresh pH buffers of 4, 7 and 10 (60mL bottles with stir bars) were used to calibrate the pH meter (using stir plate). The temperature, calibration setpoints and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

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A 10 mL aliquot (pipet with disposable 10 mL tip) of experimental solution was transferred into a 10 mL microbeaker with stir bar. A stir plate was used when measuring the pH of the experimental solutions. The pH was recorded (See table 2, column 2). pH meter challenged with pH 10 (from calibration). The solutions in the microbeakers were transferred back to into the original experimental solution.

All LSA vials had 5 mL (Repipet bottle top dispenser) of LSA cocktail added. The LSA vials were tightly capped and rigorously shaken by hand.

CHALLANGE OF BALANCE (Target = 20.0001g)
At start of day = 20.0002g
At end of day = 20.0002g

TABLE 1 - LSA info

ID	Mass (g) of vial + acid	Mass (g) of vial + acid + sample
WCA 711/13	7.8334	8.3274
Spike 61A1-1	7.8377	7.9281
Spike 61A1-2	7.8114	7.9035
CAL5-Np1a1	7.8592	8.3431
CAL5-Np1b1	7.8452	8.3303
CAL5-Np2a1	7.7891	8.2781
CAL5-Np2b1	7.7911	8.2824
CAL5-Np3a1	7.8185	8.3094
CAL5-Np3b1	7.8753	8.3667
CAL5-Np4a1	7.8913	8.3810
CAL5-Np4b1	7.9377	8.4261
CAL5-Np5a1	7.8650	8.3513
CAL5-Np5b1	7.9049	8.3934
CAL5-Np6a1	7.8310	8.3150
CAL5-Np6b1	7.8361	8.3221
CAL5-Np7a1	7.8886	8.3733
CAL5-Np7b1	7.7975	8.2861

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ID	Mass (g) of vial + acid	Mass (g) of vial + acid + sample
CAL5-Np8a1	7.8316	8.3140
CAL5-Np8b1	7.8335	8.3241
CAL5-Np9a1	7.9115	8.4029
CAL5-Np9b1	7.8849	8.3778
CAL5-Np10a1	7.8903	8.3810
CAL5-Np10b1	7.9089	8.4013
CAL5-Np11a1	7.8700	8.3590
CAL5-Np11b1	7.8508	8.3420
CAL5-~~Np11~~ BW 6/14/02 Np12a1	7.9102	8.3977
CAL5-Np12b1	7.8371	8.3246
CAL6-Np1a1	7.8082	8.3000
CAL6-Np1b1	7.8096	8.3000
CAL6-Np2a1	7.8701	8.3574
CAL6-Np2b1	7.8937	8.3852
CAL6-Np3a1	7.8774	8.3652
CAL6-Np3b1	7.8586	8.3424
CAL6-Np4a1	7.9115	8.3998
CAL6-Np4b1	7.8090	8.3024
CAL6-Np5a1	7.8134	8.3025
CAL6-Np5b1	7.9011	8.3855
CAL6-Np6a1	7.8759	8.3675
CAL6-Np6b1	7.8508	8.3417
CAL6-Np7a1	7.8670	8.3552
CAL6-Np7b1	7.8578	8.3462
CAL6-Np8a1	7.8987	8.3894
CAL6-Np8b1	7.8773	8.3663
CAL6-Np9a1	7.9852	8.4756
CAL6-Np9b1	7.9240	8.4146
CAL6-Np10a1	7.8156	8.3060
CAL6-Np10b1	7.8691	8.3608
CAL6-Np11a1	7.9036	8.3916

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From Page No. Table 1 - LSA Info CONT

ID	Mass (g) of vial + acid	Mass (g) of vial + acid + sample
CAL6-Np11b1	7.8845	8.3741
CAL6-Np12a1	7.8916	8.3804
CAL6-Np12b1	7.8814	8.3661

pH Analyses recorded in Table 2

Temp = 21.4°C Setpts = 4.01, 7.01, 10.05 slope = 99.3%

Challange after analyses using Cal Soln pH
Target = 7.01 Measured = 7.05

TABLE 2 - Experimental Soln Info

ID	Mass (g) of Exp Soln tube before LSA Sampling	pH of Exp Soln
CAL5-Np1	53.5807	2.34
CAL5-Np2	53.2339	2.54
CAL5-Np3	52.8047	2.70
CAL5-Np4	52.8887	2.97
CAL5-Np5	52.1714	3.61
CAL5-Np6	52.2060	7.80
CAL5-Np7	51.8318	8.04
CAL5-Np8	51.7529	8.28
CAL5-Np9	52.4211	8.42
CAL5-Np10	52.1883	8.67
CAL5-Np11	52.6923	8.86
CAL5-Np12	53.6827	9.54
CAL6-Np1	53.0664	2.36
CAL6-Np2	52.7553	2.58
CAL6-Np3	52.3463	2.75

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From Page No. Table 2 - Experimental Soln Info CONT

ID	Mass (g) of Exp Soln before LSA sampling	pH of Exp Soln.
CAL6-Np4	52.3846	3.05
CAL6-Np5	52.1468	4.08
CAL6-Np6	52.0339	BW 6-14-05 7.08
CAL6-Np7	51.7243	8.24
CAL6-Np8	52.1130	8.39
CAL6-Np9	52.3681	8.50
CAL6-Np10	52.0174	8.70
CAL6-Np11	52.5278	8.95
CAL6-Np12	53.4791	9.63

6-14-05

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Equilibration of Simulated Waters used in CAL Series 7, 8, 9, and 10 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To equilibrate with ambient atmosphere the simulated waters used in the experimental solutions.

Initial Entry: 711/9-10

Sample info Two simulated waters used for CAL series 7 to 10

Procedure

Aeration of simulated waters conducted since 23 Feb 2005 – see 696/14 and 32

pH Analyses of Simulated Waters for use in CAL Series 7, 8, 9 and 10 Experimental Solutions.

Bradley Werling will be performing this task.

Objective: To determine the pH of the simulated waters to be used in sorption experiments.

Initial Entry: 711/9-10

Basic Outline of Procedure

Fresh pH buffers 4, 7 and 10 (60mL bottles with stir bars) were used to calibrate the pH meter. (using stir plate). The temperature, calibration setpoints and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

About a 10 mL aliquot of the simulated water was poured out of the carboy into a small glass beaker. This aliquot was transferred into a 10 mL microbeaker with stir bar. This sampling step was repeated for the second simulated water. Each aliquot was placed on a stir plate and then the stir plate was activated. The pH was recorded. pH meter challenged with one of the calibration standards. The pH and solution ID were recorded. The solutions in the microbeakers were not transferred back to into the original experimental solution.

Temp = 21.6°C Setpts = 4.01, 7.01, + 10.05 Slope = 99.3%

ID	
19PB simulated water with Ca =	8.66
19PB simulated water without Ca =	8.72
pH 7 challange (target 7.01) =	7.04

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Addition of Water to Experimental Solutions: CAL Series 7, 8, 9, + 10

Initial Entry 711/9-10

About 30 mL (10mL pipet) of appropriate simulated water was added to the experimental solution pc tube. The mass was recorded on 711/19-20
19PB simulated water with Ca used for CAL series 7 and 9
19PB simulated water without Ca used for CAL series 8 and 10.

Challange of Balance (Target = 20.0001g)
At start of day = 20.0002g
At end of day = 20.0002g

SAMPLE ID	Mass(g) of tube + solid + sim water
CAL7-No1	52.3716
CAL7-No2	52.2594
CAL7-No3	52.2331
CAL7-No4	52.7737
CAL7-No5	52.7919
CAL7-No6	52.3893
CAL7-No7	52.4957
CAL7-No8	52.7754
CAL7-No9	52.2182
CAL7-No10	52.5303
CAL7-No11	52.3746
CAL7-No12	52.6252
CAL8-No1	52.4132
CAL8-No2	52.1820
CAL8-No3	52.6714
CAL8-No4	52.3968
CAL8-No5	52.3937
CAL8-No6	52.3579

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SAMPLE ID	Mass (g) of tube + solid + sim water
CAL8-Np7	52.5225
CAL8-Np8	52.4332
CAL8-Np9	52.1212
CAL8-Np10	52.4523
CAL8-Np11	52.2346
CAL8-Np12	52.5435
CAL9-Np1	52.1741
CAL9-Np2	52.3254
CAL9-Np3	52.1926
CAL9-Np4	52.2746
CAL9-Np5	52.0921
CAL9-Np6	52.2856
CAL9-Np7	52.3030
CAL9-Np8	52.0784
CAL9-Np9	52.1049
CAL9-Np10	52.2931
CAL9-Np11	52.4315
CAL9-Np12	52.1388
CAL10-Np1	52.4016
CAL10-Np2	51.9339
CAL10-Np3	52.2930
CAL10-Np4	52.2301
CAL10-Np5	52.2900
CAL10-Np6	52.2642
CAL10-Np7	52.1844
CAL10-Np8	52.3367
CAL10-Np9	52.2434
CAL10-Np10	52.3391
CAL10-Np11	52.2631
CAL10-Np12	52.1298

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Addition of pH adjustment solutions to CAL series 7, 8, 9, + 10

Initial entry: 711/9-10

Still trying to experimentally determine amounts of pH adjustment solns to use to make pH range of 6.5-9 in exp solns. Based the below take on CAL series 5+6 data (See 711/5 and 16-17). Two slightly different schemes were used: one for series 7+8 and another for series 9+10. Used adjustable eppendorfs 10-100µL and 100-1000µL. Recorded mass after pH ~~BAW~~ 6-17-05 adjusting solutions added. See 711/22-23 for masses.

ID	For CAL series 7 and 8		For CAL series 9 and 10	
	Vol (uL) of 0.4 M HNO3	Vol (uL) 0.1 M NaOH	Vol (uL) of 0.4 M HNO3	Vol (uL) 0.1 M NaOH
Np1	250	na	230	na
Np2	230	na	225	na
Np3	220	na	220	na
Np4	215	na	215	na
Np5	210	na	210	na
Np6	205	na	205	na
Np7	200	na	200	na
Np8	178	na	178	na
Np9	140	na	140	na
Np10	105	na	105	na
Np11	10	na	10	na
Np12	na	517	na	517

Assumptions
1. Water at equilibrium with lab $CO_2(g)$ = -3.42 (log fugacity)
2. Experimental solutions approximately 30 mL volume
3. GFW of NO_3^- = 62.0049, GFW of NA = 22.9898

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SAMPLE ID	Mass (g) after pH adjustment	Mass (g) after Np Spike
CAL7-Np1	52.6185	52.7071
CAL7-Np2	52.4930	52.5830
CAL7-Np3	52.4580	52.5562
CAL7-Np4	52.9947	53.0927
CAL7-Np5	53.0069	53.1040
CAL7-Np6	52.5984	52.6960
CAL7-Np7	52.6948	52.7925
CAL7-Np8	52.9588	53.0564
CAL7-Np9	52.3637	52.4615
CAL7-Np10	52.6412	52.7356
CAL7-Np11	52.3829	52.4803
CAL7-Np12	53.1405	53.2343
CAL8-Np1	52.6053	52.7617
CAL8-Np2	52.4181	52.5121
CAL8-Np3	52.8971	52.9932
CAL8-Np4	52.6118	52.7042
CAL8-Np5	52.6049	52.6999
CAL8-Np6	52.5667	52.6634
CAL8-Np7	52.7266	52.8230
CAL8-Np8	52.6162	52.7088
CAL8-Np9	52.2675	52.3639
CAL8-Np10	52.5674	52.6639
CAL8-Np11	52.2436	52.3398
CAL8-Np12	53.0613	53.1578
CAL9-Np1	52.4098	52.5065
CAL9-Np2	52.5550	52.6509
CAL9-Np3	52.4090	52.5029
CAL9-Np4	52.4943	52.5842
CAL9-Np5	52.3087	52.4013
CAL9-Np6	52.4963	52.5903
CAL9-Np7	52.5068	52.6028

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SAMPLE ID	Mass (g) after pH adjustment	Mass (g) after Np Spike
CAL9-Np8	52.2626	52.3505
CAL9-Np9	52.2498	52.3384
CAL9-Np10	52.4046	52.4999
CAL9-Np11	52.4400	52.5299
CAL9-Np12	52.6509	52.7467
CAL10-Np1	52.6393	52.7348
CAL10-Np2	52.1657	52.2615
CAL10-Np3	52.5064	52.5994
CAL10-Np4	52.4525	52.5477
CAL10-Np5	52.5046	52.6009
CAL10-Np6	52.4785	52.5659
CAL10-Np7	52.3914	52.4865
CAL10-Np8	52.5208	52.6150
CAL10-Np9	52.3911	52.4867
CAL10-Np10	52.4575	52.5451
CAL10-Np11	52.2698	52.3624
CAL10-Np12	52.6435	52.7383

Addition of Neptunium Spike to CAL series 7, 8, 9, + 10 Experimental Solutions

Initial Entry: 711/9-10

Np Spike #61A-1 (369/100)

Added 100µL (100µL eppendorf pipet) to each exp. soln and recorded mass on 711/22-23.

17 June 05 BAW

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Running of CAL series 7, 8, 9, + 10 Sorption Experiment

Initial Entry: 711/9-10

Each experimental soln (711/22-23) after Np 237 spiking (711/23) was vortexed for about 30 seconds and then placed on an activated gyrator (~100 RPM) with loose caps.

20 JUNE 05 BAW

Running of CAL series 7, 8, 9, + 10 Sorption Experiment

Initial Entry 711/9-10

Experiment solns (711/22-23) after Np-237 spiking (711/23) and inital vortexing (711/24) were uncapped and placed on activated gyratory shaker (RPM ~ 100) at 0915. At 16:45 exp solns were tightly capped and each one vortexed for about 10 seconds. Exp solns were loosely capped and placed on activated gyratory shaker again (RPM ~ 100).

21 JUNE 05 BAW

Running of CAL Series 7, 8, 9, + 10 Sorption Experiment

Initial Entry 711/9-10

Followed procedure on 20 June 05 (711/24)

Uncapped at 0905

Loosely capped at 16:30

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Running of CAL Series 7, 8, 9, + 10 Sorption Experiment

Initial entry 711/9-10

Followed procedure on 20 June 05 (711/24)

Uncapped at 12:15 Loosely capped at 17:35

23 JUNE 05 CONT BAW

Running of CAL Series 7, 8, 9, + 10 Sorption Experiments

Initial Entry 711/9-10

Followed procedure on 20 June 05 (711/24)

Uncapped at 0900 Loosely capped at 15:45

24 JUNE 05 BAW

Running of CAL Series 7, 8, 9, + 10 Sorption Experiments

Initial Entry 711/9-10

Followed procedure on 20 June 05 (711/24)

Uncapped: 0830

Capped: 11:40 Experiment ending, pH + LSA sampling initiated

BW 6/24/05

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Liquid Scintillation and pH Analyses of CAL series 7 to 10 – Np-237 with Consolidated Alluvium in two types of simulated groundwater over a pH range

Task initially assigned to Bradley Werling and Marla Roberts.

Objective: To determine the neptunium concentration and pH of experimental solutions and use in calculations for determining the sorption coefficient (Kd) under specified conditions

Initial Entry 711/9-10

Sample Info Two types of experiments run in duplicate for a total of four experiments. Experiements based on consolidated alluvium substrate from NC-EWDP-19PB and two simulated waters (with and without calcium) based on 19PB shallow string groundwater.

Procedure

LSA vials (size 7 mL) were labeled for experimental solutions, blank solution, and neptunium spikes solutions. Experimental solutions were analyzed in duplicate and labeled to identify the sorption solid phase and series, the radionuclide, the experimental solution number, and the a1/b1 duplicate designator (example – CAL7-Np1a1 and CAL7-Np1b1). One simulated water blank solution was analyzed and labeled as w CA with scientific notebook page reference. The neptunium spike was analyzed in duplicate and labeled Spike 61A1-3 and 61A1-4.

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.5 mL (500µL eppendorf pipet) of 0.02 M HNO_3 added. The mass of all of the LSA vials with acid was recorded (See table 1 column 1).

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (See table 2, column 1). The tubes containing the experimental solutions were placed in a tared 30 mL polypropylene beaker when being weighed.

For experimental solutions, 0.5 mL (500µL eppendorf pipet) of the experimental solution was transferred into the appropriate LSA vial. For the simulated water blank, 0.5 mL (500µL eppendorf pipet) of simulated water was added to the LSA vial. For the neptunium spike solutions, 0.1 mL (100µL eppendorf pipet) of the spike was added into each LSA vial. The mass of all of the LSA vials was recorded after the addition of the appropriate sample solution (See table 1 column 2).

The pH of the experimental solutions was taken. Fresh pH buffers of 4, 7 and 10 (60mL bottles with stir bars) were used to calibrate the pH meter (using stir plate). The temperature, calibration setpoints and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

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A 10 mL aliquot (pipet with disposable 10 mL tip) of experimental solution was transferred into a 10 mL microbeaker with stir bar. A stir plate was used when measuring the pH of the experimental solutions. The pH was recorded (See table 2, column 2). pH meter challenged with one of the calibration standards after all of the sample pHs were measured. The challenge solution type and measurement was recorded. The solutions in the microbeakers were transferred back to into the original experimental solution.

All LSA vials had 5 mL (Repipet bottle top dispenser) of LSA cocktail added. The LSA vials were tightly capped and rigorously shaken by hand.

CHALLENGE OF BALANCE (Target = 20.0001g)
At start of day – 20.0002g
At end of day – 20.0002g

TABLE 1 – LSA info.

ID	Mass (g) of vial + acid	Mass (g) of vial + acid + sample
WCA 711/26	7.8726	8.3660
Spike 61A1-3 BW 6-24-05	~~7.9349~~ 7.9505	8.0488
Spike 61A1-4 BW 6-24-05	~~7.8823~~ 7.8068	7.9019
CAL7-Np1a1	7.9349	8.4244
CAL7-Np1b1	7.8823	8.3667
CAL7-Np2a1	7.9265	8.4129
CAL7-Np2b1	7.8852	8.3710
CAL7-Np3a1	7.9022	8.3853
CAL7-Np3b1	7.8387	8.3252
CAL7-Np4a1	7.9528	8.4439
CAL7-Np4b1	7.8995	8.3867
CAL7-Np5a1	7.9184	8.4049
CAL7-Np5b1	7.9706	8.4579
CAL7-Np6a1	7.8391	8.3243
CAL7-Np6b1	7.9603	8.4434
CAL7-Np7a1	7.9191	8.4061

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TABLE 1 - cont - LSA info

ID	Mass(g) of vial + acid	Mass(g) of vial + acid + sample
CAL7-Np7b1	7.9426	8.4296
CAL7-Np8a1	7.9085	8.3874
CAL7-Np8b1	7.8408	8.3293
CAL7-Np9a1	7.8998	8.3903
CAL7-Np9b1	7.8470	8.3389
CAL7-Np10a1	7.7220	8.2114
CAL7-Np10b1	7.8912	8.3795
CAL7-Np~~10~~ 11 BW 6-24-05 a1	7.9193	8.4078
CAL7-Np11b1	7.8912	8.3809
CAL7-Np12a1	7.8349	8.3223
CAL7-Np12b1	7.9360	8.4235
CAL8-Np1a1	8.0145	8.5022
CAL8-Np1b1	7.9091	8.3966
CAL8-Np2a1	7.9560	8.4407
CAL8-Np2b1	7.9838	8.4685
CAL8-Np3a1	7.9750	8.4617
CAL8-Np3b1	7.8713	8.3581
CAL8-Np4a1	7.9011	8.3891
CAL8-Np4b1	7.8716	8.3598
CAL8-Np5a1	7.9438	8.4315
CAL8-Np5b1	7.8769	8.3636
CAL8-Np6a1	7.9037	8.3927
CAL8-Np6b1	7.9572	8.4445
CAL8-Np7a1 7.9088	~~7.9836~~ 24 June 05 MR	8.3974
CAL8-Np7b1 7.9297	~~7.8554~~ 24 June 05 MR	8.4183
CAL8-Np8a1	7.8712	8.3601
CAL8-Np8b1	7.9694	8.4599
CAL8-Np9a1	7.9131	8.3934
CAL8-Np9b1	7.8928	8.3789
CAL8-Np10a1 BW 6-24-05	~~7.8367~~	8.3201
	7.8367	

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TABLE 1 - cont - LSA info

ID	Mass(g) of vial + acid	Mass(g) of vial + acid + sample
CAL8-Np10b1	7.8355	8.3204
CAL8-Np11a1	7.8724	8.3563
CAL8-Np11b1	7.9073	8.3907
CAL8-Np12a1	7.9486	8.4303
CAL8-Np12b1	7.8183	8.3048
CAL9-Np1a1	7.9072	8.3942
CAL9-Np1b1	7.8935	8.3799
CAL9-Np2a1	7.8968	8.3862
CAL9-Np2b1	7.8773	8.3663
CAL9-Np3a1	7.8795	8.3663
CAL9-Np3b1	7.8982	8.3846
CAL9-Np4a1	7.9001	8.3832
CAL9-Np4b1	7.8396	8.3277
CAL9-Np5a1	7.9969	8.4846
CAL9-Np5b1	7.9729	8.4621
CAL9-Np6a1	7.9094	8.3955
CAL9-Np6b1	7.9223	8.4083
CAL9-Np7a1	7.8175	8.2977
CAL9-Np7b1	7.8486	8.3265
CAL9-Np8a1	8.0188	8.5070
CAL9-Np8b1	7.9852	8.4700
CAL9-Np9a1	7.8990	8.3856
CAL9-Np9b1	7.9231	8.4088
CAL9-Np10a1	8.0112	8.4988
CAL9-Np10b1	7.8571	8.3445
CAL9-Np11a1	8.0256	8.5131
CAL9-Np11b1	7.8996	8.3862
CAL9-Np12a1	7.9933	8.4791
CAL9-Np12b1	7.8914	8.3733
CAL10-Np1a1	7.8957	8.3865

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ID	Mass(g) of vial + acid	Mass(g) of vial + acid + sample
CAL10-Np1b1	7.9720	8.4625
CAL10-Np2a1	7.8933	8.3801
CAL10-Np2b1	7.8769	8.3661
CAL10-Np3a1	7.8811	8.3695
CAL10-Np3b1	7.9900	8.4782
CAL10-Np4a1	7.8552	8.3438
CAL10-Np4b1	7.9259	8.4125
CAL10-Np5a1	7.8859	8.3730
CAL10-Np5b1	7.7819	8.2666
CAL10-Np6a1	7.8898	8.3778
CAL10-Np6b1	7.8954	8.3764
CAL10-Np7a1	7.9415	8.4316
CAL10-Np7b1	7.9206	8.4114
CAL10-Np8a1	7.9522	8.4401
CAL10-Np8b1	7.8911	8.3821
CAL10-Np9a1	7.8419	8.3261
CAL10-Np9b1	7.9562	8.4414
CAL10-Np10a1	7.8554	8.3339
CAL10-Np10b1	7.8153	8.3002
CAL10-Np11a1	7.9162	8.4028
CAL10-Np11b1	7.8393	8.3245
CAL10-Np12a1	7.8867	8.3707
CAL10-Np12b1	7.8519	8.3379

pH Analyses recorded in Table 2

Temp = 22.7 Setpts = 4.01, 7.00, 10.04 Slope = 99.7

Challenge after analyses using Cal pH soln

Target = 7.00 Measured = 7.01

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Table 2 - Experimental Soln Info

ID	Mass(g) of Exp Soln tube before LSA sampling	pH of exp soln
CAL7-Np1	52.0489	4.85
CAL7-Np2	51.7798	5.92
CAL7-Np3	51.4956	7.08
CAL7-Np4	52.2736	7.27
CAL7-Np5	52.2572	7.46
CAL7-Np6	51.7835	7.59
CAL7-Np7	51.9366	7.77
CAL7-Np8	52.1615	7.96
CAL7-Np9	51.8529	8.22
CAL7-Np10	52.2151	8.37
CAL7-Np11	51.8657	8.66
CAL7-Np12	52.3899	8.87
CAL8-Np1	51.9104	7.34
CAL8-Np2	51.7136	7.71
CAL8-Np3	52.1417	7.84
CAL8-Np4	51.8682	7.97
CAL8-Np5	52.2987	7.98
CAL8-Np6	52.1975	8.01
CAL8-Np7	52.3203	8.04
CAL8-Np8	52.1507	8.18
CAL8-Np9	51.6985	8.35
CAL8-Np10	51.9402	8.46
CAL8-Np11	51.5841	8.70
CAL8-Np12	52.4446	8.93
CAL9-Np1	52.1101	5.77
CAL9-Np2	52.1954	6.75
CAL9-Np3	52.0026	7.38
CAL9-Np4	52.0580	7.29
CAL9-Np5	51.8531	7.37
CAL9-Np6	51.9315	7.57

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TABLE 2- cont- Experimental Soln Info

ID	Mass(g) of Exp Soln tube before LSA Sampling	pH of exp soln
CAL9-Np7	51.9272	7.67
CAL9-Np8	51.6862	7.91
CAL9-Np9	51.9719	8.18
CAL9-Np10	52.0782	8.33
CAL9-Np11	52.0721	8.62
CAL9-Np12	52.2483	8.83
CAL10-Np1	52.1899	7.69
CAL10-Np2	51.6912	7.70
CAL10-Np3	51.9795	7.84
CAL10-Np4	51.9109	7.84
CAL10-Np5	52.2158	7.92
CAL10-Np6	52.1813	7.94
CAL10-Np7	52.0306	7.98
CAL10-Np8	51.9660	8.15
CAL10-Np9	52.0026	8.33
CAL10-Np10	52.0270	8.45
CAL10-Np11	51.8241	8.68
CAL10-Np12	52.1444	8.90

6-27-05 BAW

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Witnessed & Understood by me, | Date | Invented by | Date | Recorded by

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Liquid Scintillation Analyses Results for CAL series 5 + 6 Experimental Solutions

Task initially assigned to Bradley Werling

Objective

To determine the amount of neptunium in the experimental solution at the end of the sorption experiments.

Initial Entry 696/170-171 and 711/2-3

LSA sample preparation 711/13-16

Sample Info

Blank (one sample from simulated water with calcium): w Ca 711/13
Neptunium spikes (two samples) – Spike 61A1-1 and Spike 61A1-2
Two experiments derived from 1 solid and 2 types of simulated waters.
Consolidated Alluvium series 5 (24 samples) using simulated water with calcium
Consolidated Alluvium series 6 (24 samples) using simulated water without calcium

Results

Printout of LSA analyses following:

6-27-05 BW

To Page No.____

Witnessed & Understood by me, | Date | Invented by | Date | Recorded by

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6/23/05 5:25:05 PM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 1

Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

Assay Definition-

Assay Description:

Assay Type: Alpha/Beta
Report Name: Np_Pa_Exp
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\20050620_1811.results
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.035
Assay File Name: C:\Packard\TriCarb\Assays\Pa_Np_Exp_AB.lsa

Count Conditions-

Nuclide: NP/PA
Quench Indicator: SIS
External Std Terminator (sec): n/a
Pre-Count Delay (min): 0.00
Alpha/Beta Standards:
Low Energy: NP/PA
Count Time (min): 240.00
Count Mode: Normal
Assay Count Cycles: 1 Repeat Sample Count: 1
#Vials/Sample: 1 Calculate % Reference: Off

Background Subtract: On - 1st Vial
Low CPM Threshold: Off
2 Sigma % Terminator: On - Any Region

In Use Discriminator: 143

Regions	LL	UL	Bkg Subtract	2Sigma % Terminator
Beta A	0.0	400.0	1st Vial	0.00
Beta B	100.0	400.0	1st Vial	0.00
Alpha	100.0	400.0	1st Vial	2.00

Count Corrections-

Static Controller: On Luminescence Correction: Off
Colored Samples: n/a Heterogeneity Monitor: n/a
Coincidence Time (nsec): 18 Delay Before Burst (nsec): 75

Half Life-

Half Life Correction: Off

Regions	Half Life	Units	Reference Date	Reference Time
Beta A				
Beta B				
Alpha				

IPA Block Data
Software Version IC: 2.11
Software Version EC: 1.31
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 20.40 Date Processed: 6/20/05 6:11:42 PM
14C Chi Square: 18.82 Date Processed: 6/20/05 6:11:42 PM
3H E^2/B (1-18.6 keV): 294.70 Date Processed: 6/20/05 6:11:42 PM
14C E^2/B (4-156 keV): 549.28 Date Processed: 6/20/05 6:11:42 PM
3H Efficiency (0-18.6 keV): 65.41 Date Processed: 6/20/05 6:11:42 PM
14C Efficiency (0-156 keV): 96.52 Date Processed: 6/20/05 6:11:42 PM
IPA Background Date Processed: 6/20/05 6:11:42 PM

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Witnessed & Understood by me,	Date	Invented by	Date
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Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

3H Background CPM (0-18.6 keV): 14.55 Date Processed: 6/20/05 6:11:42 PM
14C Background CPM (0-156 keV): 21.98 Date Processed: 6/20/05 6:11:42 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMa	alpha2S%	SIS	
MESSAGES									
1	240.00	20.20	2.87	3.34	7.07	0.43	19.61	166.4	w CA 711/13
B									
Missing vial 2.									CAL 5 series
3	70.34	23.14	7.23	9.98	9.03	141.75	2.01	293.7	>Np1 < ql bl
4	69.38	24.04	7.07	10.18	8.97	143.74	2.01	284.4	
5	67.33	23.97	7.18	10.07	9.16	148.13	2.01	286.0	>Np2 < ql bl
6	67.95	24.40	7.05	10.17	9.07	146.73	2.01	288.4	
7	71.25	23.16	7.19	9.32	9.39	139.95	2.01	283.9	>Np3 < ql bl
8	69.32	23.51	7.19	9.92	9.13	143.85	2.01	287.7	
9	69.46	24.17	7.04	10.37	8.87	143.58	2.01	293.3	>Np4 < ql bl
10	69.16	24.91	6.89	11.24	8.44	144.16	2.01	308.6	
11	67.44	28.15	6.36	11.10	8.60	147.86	2.01	278.3	>Np5 < ql bl
12	66.30	28.09	6.42	11.34	8.56	150.43	2.01	283.6	
13	70.16	27.76	6.31	11.34	8.33	142.11	2.01	282.9	>Np6 < ql bl
14	69.75	28.77	6.16	11.52	8.27	142.95	2.01	284.8	
15	73.24	28.83	6.02	11.64	8.03	136.12	2.01	285.7	>Np7 < ql bl
16	72.87	28.80	6.04	11.43	8.14	136.81	2.01	276.2	
17	70.29	26.77	6.48	11.32	8.33	141.86	2.01	285.8	>Np8 < ql bl
18	69.21	27.61	6.38	11.21	8.44	144.13	2.01	292.1	
19	68.05	29.66	6.10	12.92	7.79	146.58	2.01	299.6	>Np9 < ql bl
20	68.62	28.80	6.20	11.92	8.16	145.33	2.01	287.9	
21	67.95	28.38	6.30	11.32	8.47	146.75	2.01	283.2	>Np10 < ql bl
22	68.15	29.51	6.11	12.32	8.01	146.33	2.01	289.4	
23	155.57	13.54	8.11	5.99	9.07	63.85	2.02	296.2	>Np11 < ql bl
24	155.99	14.51	7.63	6.10	8.94	63.67	2.02	292.3	
25	240.00	11.10	8.35	4.52	9.56	21.18	2.86	283.1	>Np12 < ql bl
26	240.00	10.83	8.53	4.67	9.30	21.65	2.83	298.9	
Missing vial 27.									CAL series 6
28	70.41	29.66	6.00	11.96	8.04	141.59	2.01	284.6	>Np1 < ql bl
29	70.00	28.14	6.26	11.33	8.34	142.42	2.01	278.1	
30	68.48	29.04	6.17	12.21	8.04	145.62	2.01	289.5	>Np2 < ql bl
31	66.34	30.50	6.04	13.15	7.79	150.32	2.01	297.9	
32	68.27	29.93	6.04	12.39	7.98	146.06	2.01	289.3	>Np3 < ql bl
33	68.86	29.42	6.10	12.39	7.95	144.79	2.01	293.0	
34	67.34	29.71	6.12	11.94	8.22	148.08	2.01	279.4	>Np4 < ql bl
35	66.21	29.89	6.13	12.96	7.87	150.62	2.01	298.3	
36	67.49	31.94	5.80	13.60	7.57	147.74	2.01	293.1	>Np5 < ql bl
37	67.80	30.73	5.95	12.43	7.99	147.07	2.01	284.6	
38	70.22	35.18	5.31	14.24	7.22	141.99	2.01	281.3	>Np6 < ql bl
39	70.79	33.33	5.50	14.07	7.25	140.86	2.01	287.4	
40	69.56	34.42	5.42	13.67	7.44	143.37	2.01	280.1	>Np7 < ql bl
41	68.75	35.55	5.32	14.16	7.32	145.02	2.01	278.6	
42	69.66	34.98	5.35	14.56	7.15	143.14	2.01	288.3	>Np8 < ql bl
43	69.51	33.72	5.50	13.26	7.58	143.47	2.01	275.5	
44	73.30	33.02	5.45	13.44	7.33	135.99	2.01	282.5	>Np9 < ql bl
45	74.03	34.83	5.22	13.45	7.29	134.73	2.01	274.5	
46	68.43	35.49	5.34	14.33	7.28	145.70	2.01	283.1	>Np10 < ql bl
47	68.53	36.21	5.26	14.64	7.18	145.49	2.01	278.5	
48	71.41	34.89	5.30	14.39	7.12	139.62	2.01	288.3	>Np11 < ql bl
49	72.29	34.15	5.36	13.95	7.21	137.91	2.01	285.9	
50	137.01	24.95	5.16	10.48	6.47	72.55	2.02	287.1	>Np12 < ql bl
51	138.56	24.26	5.25	10.30	6.51	71.74	2.02	289.6	
Missing vial 52.									

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

Project No.
Book No.
TITLE 27 JUNE 05 CONT BAW

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Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

53	1.12	6623.55	2.33	2132.38	4.10	8977.25	1.99	244.7 Spike 61A1-1
54	1.13	6975.37	2.26	2124.10	4.09	8873.90	2.00	237.6 Spike 61A1-2

30 JUNE 05 BAW

Liquid Scintillation Analyses Results for CAL series 7, 8, 9, + 10 Experimental Solutions

Task initially assigned to Bradley Werling

Objective

To determine the amount of neptunium in the experimental solution at the end of the sorption experiments.

Initial Entry 711/9-10

LSA sample preparation 711/26-30

Sample Info

Blank (one sample from simulated water with calcium): w Ca 711/26
Neptunium spikes (two samples) – Spike 61A1-3 and Spike 61A1-4
Four experiments derived from 1 solid and 2 types of simulated waters (7+9 duplicate experiments and 8+10 duplicate experiments).
Consolidated Alluvium series 7 (24 samples) using simulated water with calcium
Consolidated Alluvium series 8 (24 samples) using simulated water without calcium.
Consolidated Alluvium series 9 (24 samples) using simulated water with calcium
Consolidated Alluvium series 10 (24 samples) using simulated water without calcium.

Results

Printout of LSA analyses following:

6-30-05 BAW

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Recorded by

TITLE 30 JUNE 05 CONT BAW
Project No.
Book No.

6/30/05 3:28:28 AM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 1
Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

Assay Definition-

Assay Description:

Assay Type: Alpha/Beta
Report Name: Np_Pa_Exp
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\20050624_2118.results
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.036
Assay File Name: C:\Packard\TriCarb\Assays\Pa_Np_Exp_AB.lsa

Count Conditions-

Nuclide: NP/PA
Quench Indicator: SIS
External Std Terminator (sec): n/a
Pre-Count Delay (min): 0.00
Alpha/Beta Standards:
Low Energy: NP/PA
Count Time (min): 240.00
Count Mode: Normal
Assay Count Cycles: 1 Repeat Sample Count: 1
#Vials/Sample: 1 Calculate % Reference: Off

Background Subtract: On - 1st Vial
Low CPM Threshold: Off
2 Sigma % Terminator: On - Any Region

In Use Discriminator: 143

Regions	LL	UL	Bkg Subtract	2Sigma % Terminator
Beta A	0.0	400.0	1st Vial	0.00
Beta B	100.0	400.0	1st Vial	0.00
Alpha	100.0	400.0	1st Vial	2.00

Count Corrections-

Static Controller: On Luminescence Correction: Off
Colored Samples: n/a Heterogeneity Monitor: n/a
Coincidence Time (nsec): 18 Delay Before Burst (nsec): 75

Half Life-

Half Life Correction: Off

Regions	Half Life	Units	Reference Date	Reference Time
Beta A				
Beta B				
Alpha				

IPA Block Data
Software Version IC: 2.11
Software Version EC: 1.31
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 9.78 Date Processed: 6/24/05 9:18:03 PM
14C Chi Square: 15.90 Date Processed: 6/24/05 9:18:03 PM
3H E^2/B (1-18.6 keV): 261.64 Date Processed: 6/24/05 9:18:03 PM
14C E^2/B (4-156 keV): 505.95 Date Processed: 6/24/05 9:18:03 PM
3H Efficiency (0-18.6 keV): 65.46 Date Processed: 6/24/05 9:18:03 PM
14C Efficiency (0-156 keV): 96.43 Date Processed: 6/24/05 9:18:03 PM
IPA Background Date Processed: 6/24/05 9:18:03 PM

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Recorded by

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Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

3H Background CPM (0-18.6 keV): 16.33 Date Processed: 6/24/05 9:18:03 PM
14C Background CPM (0-156 keV): 23.52 Date Processed: 6/24/05 9:18:03 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMa	alpha2S%	SIS	
MESSAGES									
1	240.00	19.88	2.90	3.50	6.90	0.38	20.85	171.4	w CA 711/26
B									
Missing vial 2.									
									CAL series 7
3	72.72	6.69	20.02	2.93	21.92	137.13	2.01	310.9	Np1
4	74.77	7.20	18.52	2.71	23.05	133.37	2.01	260.2	
5	71.80	8.88	15.66	3.80	17.93	138.92	2.01	294.4	
6	70.99	8.41	16.50	3.36	19.82	140.48	2.01	285.5	Np2
7	68.32	7.96	17.59	3.53	19.41	146.03	2.01	313.4	
8	66.63	7.44	18.88	2.81	23.54	149.71	2.01	271.1	Np3
9	67.29	5.92	23.07	2.58	25.08	148.24	2.01	317.0	
10	68.39	7.30	18.98	3.30	20.44	145.85	2.01	305.3	Np4
11	69.07	8.01	17.43	3.60	19.03	144.43	2.01	308.9	
12	69.91	7.89	17.57	3.28	20.34	142.67	2.01	301.4	Np5
13	69.90	6.64	20.47	2.94	22.20	142.69	2.01	308.0	
14	69.16	8.22	17.03	3.05	21.65	144.24	2.01	250.9	Np6
15	70.91	9.14	15.34	3.51	19.17	140.64	2.01	272.2	
16	70.59	7.12	19.16	3.32	20.09	141.31	2.01	306.6	Np7
17	70.49	9.46	14.94	3.63	18.75	141.49	2.01	286.4	
18	69.47	9.89	14.46	4.15	17.02	143.58	2.01	306.0	Np8
19	68.95	9.01	15.72	3.51	19.42	144.66	2.01	281.6	
20	69.74	9.09	15.54	3.01	21.80	143.01	2.01	256.9	Np9
21	72.57	9.14	15.20	3.45	19.25	137.43	2.01	273.2	
22	71.35	9.17	15.26	4.09	17.01	139.80	2.01	312.4	Np10
23	69.15	10.52	13.74	4.11	17.17	144.27	2.01	279.1	
24	68.83	9.02	15.72	3.59	19.08	144.90	2.01	290.8	Np11
25	163.66	6.10	16.13	2.32	19.29	60.73	2.02	275.3	
26	162.90	6.71	14.79	2.62	17.40	61.02	2.02	278.3	Np12
Missing vial 27.									
									CAL series 8
28	68.31	16.94	9.31	6.72	12.05	146.04	2.01	278.8	Np1
29	68.44	17.91	8.90	6.82	11.92	145.76	2.01	259.0	
30	72.18	13.22	11.13	5.32	13.91	138.17	2.01	284.6	
31	71.93	13.89	10.70	5.36	13.85	138.70	2.01	273.9	Np2
32	71.58	12.97	11.35	5.26	14.06	139.38	2.01	287.0	
33	70.37	14.20	10.61	5.60	13.55	141.75	2.01	265.2	Np3
34	72.27	14.89	10.08	5.69	13.23	137.99	2.01	280.9	
35	72.86	14.58	10.22	5.86	12.90	136.89	2.01	284.3	Np4
36	71.38	14.02	10.65	5.16	14.28	139.71	2.01	252.4	
37	72.23	14.72	10.19	5.59	13.41	138.08	2.01	265.6	Np5
38	72.42	16.15	9.44	5.77	13.09	137.70	2.01	250.4	
39	72.15	15.21	9.92	6.03	12.71	138.26	2.01	279.8	Np6
40	71.13	14.82	10.20	5.56	13.55	140.20	2.01	262.6	
41	70.48	14.93	10.18	5.46	13.79	141.50	2.01	262.2	Np7
42	74.49	14.88	9.96	5.50	13.38	133.86	2.01	276.5	
43	73.25	15.10	9.92	5.46	13.55	136.14	2.01	270.6	Np8
44	71.12	15.44	9.86	5.95	12.90	140.24	2.01	271.8	
45	72.00	15.76	9.65	5.56	13.48	138.51	2.01	259.4	Np9
46	69.85	16.57	9.39	6.04	12.87	142.79	2.01	251.2	
47	69.63	15.39	9.97	6.17	12.70	143.23	2.01	291.2	Np10
48	68.55	17.04	9.25	6.19	12.76	145.50	2.01	266.6	
49	69.87	14.87	10.24	6.52	12.19	142.74	2.01	293.1	Np11
50	75.48	18.53	8.30	6.48	11.82	132.18	2.01	256.2	
51	75.97	17.75	8.57	6.82	11.37	131.25	2.01	267.1	Np12
Missing vial 52.									

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by

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Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

								CAL series 9
53	67.68	18.18	8.84	7.60	11.12	147.39	2.01	290.5 Np1
54	68.55	17.86	8.91	7.34	11.32	145.51	2.01	280.5
55	67.61	17.60	9.07	7.40	11.32	147.52	2.01	280.3 Np2
56	68.17	19.24	8.42	7.61	11.07	146.32	2.01	280.7
57	70.42	19.21	8.31	7.58	10.94	141.62	2.01	275.8 Np3
58	71.01	18.27	8.62	7.25	11.24	140.46	2.01	281.4
59	75.77	15.13	9.76	6.11	12.30	131.60	2.01	287.3 Np4
60	73.64	19.11	8.19	7.10	11.22	135.41	2.01	263.5
61	73.35	17.22	8.91	7.08	11.25	135.96	2.01	277.0 Np5
62	71.87	18.23	8.59	7.77	10.65	138.78	2.01	295.1
63	71.50	18.27	8.59	6.81	11.70	139.49	2.01	265.8 Np6
64	72.21	17.00	9.06	6.93	11.50	138.12	2.01	275.1
65	70.89	18.01	8.72	6.76	11.81	140.68	2.01	269.6 Np7
66	72.01	16.91	9.11	7.22	11.19	138.50	2.01	292.4
67	77.45	17.00	8.80	6.54	11.61	128.80	2.01	273.1 Np8
68	77.48	16.65	8.94	6.39	11.80	128.70	2.01	265.3
69	76.95	15.30	9.61	5.82	12.65	129.57	2.01	271.6 Np9
70	77.86	14.77	9.84	6.07	12.21	128.05	2.01	281.0
71	69.47	16.51	9.43	6.35	12.45	143.58	2.01	266.7 Np10
72	71.16	19.76	8.10	7.68	10.79	140.15	2.01	276.2
73	72.93	17.69	8.74	6.57	11.89	136.73	2.01	272.0 Np11
74	73.91	17.69	8.69	7.03	11.27	134.93	2.01	273.6
75	159.00	10.99	9.58	3.95	12.54	62.51	2.02	257.7 Np12
76	161.32	10.64	9.80	3.81	12.84	61.62	2.02	261.5
Missing vial 77.								
								CAL series 10
78	71.74	18.65	8.44	7.64	10.79	139.02	2.01	276.9 Np1
79	71.44	19.02	8.33	7.30	11.16	139.62	2.01	273.2
80	69.43	23.75	7.10	8.60	10.10	143.65	2.01	266.9 Np2
81	71.47	21.52	7.56	8.65	9.93	139.54	2.01	279.8
82	70.65	22.34	7.38	9.16	9.61	141.17	2.01	286.4 Np3
83	71.64	21.02	7.69	8.30	10.20	139.20	2.01	275.9
84	71.57	24.15	6.92	9.48	9.33	139.38	2.01	273.5 Np4
85	71.66	22.88	7.21	9.52	9.30	139.18	2.01	280.9
86	71.94	19.82	8.04	8.61	9.93	138.62	2.01	292.2 Np5
87	71.78	20.97	7.70	7.73	10.70	138.93	2.01	270.0
88	78.19	20.78	7.47	8.40	9.72	127.54	2.01	291.2 Np6
89	79.94	20.75	7.41	8.00	9.95	124.71	2.01	276.6
90	72.04	23.87	6.96	9.66	9.19	138.44	2.01	277.7 Np7
91	70.34	22.69	7.31	9.23	9.58	141.84	2.01	286.3
92	72.14	23.54	7.03	9.45	9.32	138.25	2.01	284.7 Np8
93	72.43	22.95	7.16	9.00	9.61	137.68	2.01	280.4
94	71.31	22.67	7.27	8.77	9.85	139.88	2.01	275.3 Np9
95	71.51	23.97	6.96	9.96	9.04	139.49	2.01	290.1
96	74.91	23.61	6.90	9.09	9.40	133.12	2.01	272.0 Np10
97	72.70	24.69	6.76	9.54	9.23	137.18	2.01	277.7
98	71.83	24.88	6.75	10.01	8.99	138.83	2.01	276.8 Np11
99	71.00	23.26	7.14	9.48	9.37	140.46	2.01	288.6
100	74.56	25.23	6.58	10.12	8.78	133.75	2.01	283.8 Np12
101	74.32	24.47	6.74	9.49	9.17	134.17	2.01	281.4
Missing vial 102.								
103	0.89	13735.18	1.81	3826.84	3.43	11353.55	1.99	222.1 Spike 61A1-3
104	0.96	12129.08	1.85	3320.46	3.54	10496.49	1.99	219.5 Spike 61A1-4

6-30-05 BW

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From Page No. 30 June 05 MR

Collection of ~~Sample~~ 30 June 05 MR from sonic core composite Aliquot

Task initially assigned to Marla Roberts

Objective: To provide Paul Bertetti ~~a sample~~ an aliquot 1 July 05 MR (at his request) of the 19PB consolidated alluvium used in the CAL series 1 to 10 sorption experiments.

30 June 05 MR ~~Samples:~~
Sample: NC-EWDP-19PB-0305-Sonic Core Composite (696164-66)

Equipment: Mettler AE240 balance, Sn 001439

Supplies: polystyrene bottle
weighing paper
spatula

Procedure: The sample container was gently rolled by hand prior to aliquot removal to avoid a grain settling bias. Using a spatula, about 1 gram of sample was transferred on a taped piece of weighing paper. The mass was recorded and the aliquot was transferred to the labeled 30 June 05 MR ~~polyst~~ polystyrene bottle. The bottle was labeled with the ID, date, SN reference and initials. The ID was CAL19PB. CAL19PB

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From Page No. 30 June 05 MR cont.

was chosen to indicate composite alluvium from well 19PB.

Sample ID	Aliquot ID	Aliquot Mass (g)
NC-EWDP-19PB-Sonic Core Composite	CAL 19PB	1.2737

30 June 05 MR

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Investigation of LSA results of Neptunium Spikes Used in Recent Sorption Experiments

Task initially assigned to Bradley Werling

Objective

To investigate apparent variations in the LSA results for neptunium spikes used in the CAL series 7 to 10 sorption experiments.

Background Information

LSA samples are analyzed in duplicate. Each time a group of samples is analyzed on the LSA, a set of neptunium spikes is also analyzed. The counts for the two neptunium spikes analyzed with the CAL series 7 to 10 sample group were suspicious for two reasons. The both counts were larger than expected based on previous counts and the range between the two counts was large. At Paul Bertetti's suggestion, several old spikes were reanalyzed and some new spikes were prepared and analyzed as well. The new spikes consisted of freshly prepared version of the CAL series 7 to 10 spikes and a version of these spikes that used a more concentrated acid solution for the matrix.

Sample Info: 17 total samples

Old samples (13) being reanalyzed:

Blank (one): w Ca 711/26 from CAL series 7, 8, 9, and 10
Spike 46A-3 and 4 from AL series 2 (631/40) + AL series 3 (631/49) [used twice]
Spike 46A-5 and 6 from AL series 4 and M series 2 (696/71)
Spike 46A-7 and 8 from AL series 5+6, M series 3+4, and CAL series1+2 (696/100)
Spike 46A-9 and 10 from CAL series 3+4 and M series 5+6 (696/153)
Spike 61A1-1 and 2 from CAL series 5+6 (711/36)
Spike 61A1-3 and 4 from CAL series 7, 8, 9, and 10 (711/39)

New samples (4) being analyzed for the first time:

Spike 61A1-5 and 6
Spike 61A1-7 and 8

Reagents:

Neptunium Spike # 61A1 (369/100) - 60 ppm Np-237 at pH of 5.63
Ultima-Gold AB liquid scintillation cocktail (Packard 6013309 lot # 91-050201)
0.02 M HNO_3 (SN 696/29-30) for LSA matrix (soln normally used for this)
0.4 M HNO3 (696/195) for LSA matrix (soln not normally used for this)
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

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Equipment:

Mettler AE240 balance (sn 101237)
Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)

Supplies:

Pipettes and tips
Repipettor for transfer of scintillation cocktail
7 ml scintillation vials
10 mL microbeakers
Plasticware and glassware as needed

Procedure

The previously analyzed neptunium spikes were gathered. The most recent simulated water background blank was also gathered.

The new samples were prepared using the following procedure.

LSA vials (size 7 mL) were labeled for four neptunium spikes solutions. The neptunium spikes were analyzed in duplicate and labeled Spike 61A1-5, Spike 61A1-6, Spike 61A1-7, and Spike 61A1-8.

Each LSA vial had 0.5 mL (500µL eppendorf pipet) of acid added. Spikes 61A1-5 and 6 had 0.02 M HNO_3 added while spikes 61A1-7 and 8 had 0.4 M HNO_3 added. The mass of all of the LSA vials with acid was recorded. Next, 0.1 mL (100µL eppendorf pipet) of spike 61A1 was added into each LSA vial. The mass of all of the LSA vials was recorded again. All LSA vials had 5 mL (Repipet bottle top dispenser) of LSA cocktail added. The LSA vials were tightly capped and rigorously shaken by hand.

Challange of Balance (Target = 20,000 g)
At start of day = 20,0002 g At end of day = 20,0002 g

ID	Mass (g) of vial + acid	Mass (g) of vial + acid + spike
Spike 61A1-5	7.8236	7.9153
Spike 61A1-6	7.8992	7.9909
Spike 61A1-7	7.9430	8.0415
Spike 61A1-8	7.8806	7.9768

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Liquid Scintillation Analyses Results for Neptunium Spikes Used in Recent Sorption Experiments

Task initially assigned to Bradley Werling

Objective

To investigate apparent variations in the LSA results for neptunium spikes used in the CAL series 7 to 10 sorption experiments.

Initial Entry for Investigation Work 711//42-43

LSA sample preparation for the new samples 711/42-43

Sample Info: 17 Total Samples

Old samples (13) being reanalyzed:

- Blank (one): w Ca 711/26 from CAL series 7, 8, 9, and 10
- Spike 46A-3 and 4 from AL series 2 (631/40) + AL series 3 (631/49) [used twice]
- Spike 46A-5 and 6 from AL series 4 and M series 2 (696/71)
- Spike 46A-7 and 8 from AL series 5+6, M series 3+4, and CAL series1+2 (696/100)
- Spike 46A-9 and 10 from CAL series 3+4 and M series 5+6 (696/153)
- Spike 61A1-1 and 2 from CAL series 5+6 (711/36)
- Spike 61A1-3 and 4 from CAL series 7, 8, 9, and 10 (711/39)

New samples (4) being analyzed for the first time:

- Spike 61A1-5 and 6
- Spike 61A1-7 and 8

Results

Printout of LSA analyses following:

7-1-05 BAW

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6/30/05  6:21:38 PM          QuantaSmart (TM) - 1.31 - Serial# 405314          Page # 1
Protocol# 15 - Pa_Np_Exp_AB.lsa                                                User: Bertetti


  Assay Definition-

  Assay Description:


  Assay Type: Alpha/Beta
  Report Name: Np_Pa_Exp
  Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB
  Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\20050630_1354.results
  Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.037
  Assay File Name: C:\Packard\TriCarb\Assays\Pa_Np_Exp_AB.lsa

  Count Conditions-

  Nuclide: NP/PA
     Quench Indicator: SIS
     External Std Terminator (sec): n/a
     Pre-Count Delay (min): 0.00
  Alpha/Beta Standards:
     Low Energy: NP/PA
  Count Time (min): 240.00
  Count Mode: Normal
  Assay Count Cycles: 1          Repeat Sample Count: 1
  #Vials/Sample: 1               Calculate % Reference: Off

  Background Subtract: On - 1st Vial
  Low CPM Threshold: Off
  2 Sigma % Terminator: On - Any Region

  In Use Discriminator: 143

  Regions          LL       UL  Bkg Subtract  2Sigma % Terminator
  Beta A          0.0    400.0      1st Vial                 0.00
  Beta B        100.0    400.0      1st Vial                 0.00
  Alpha         100.0    400.0      1st Vial                 2.00

  Count Corrections-

  Static Controller: On          Luminescence Correction: Off
  Colored Samples: n/a           Heterogeneity Monitor: n/a
  Coincidence Time (nsec): 18    Delay Before Burst (nsec): 75

  Half Life-

  Half Life Correction: Off
  Regions    Half Life          Units      Reference Date      Reference Time
  Beta A
  Beta B
  Alpha

  IPA Block Data
  Software Version IC: 2.11
  Software Version EC: 1.31
  Instrument Model: Tri-Carb 3100TR
  Instrument Serial Number: 405314
  3H Chi Square: 30.43     Date Processed: 6/30/05 1:54:42 PM
  14C Chi Square: 22.52    Date Processed: 6/30/05 1:54:42 PM
  3H E^2/B (1-18.6 keV): 287.59  Date Processed: 6/30/05 1:54:42 PM
  14C E^2/B (4-156 keV): 544.96   Date Processed: 6/30/05 1:54:42 PM
  3H Efficiency (0-18.6 keV): 65.24  Date Processed: 6/30/05 1:54:42 PM
  14C Efficiency (0-156 keV): 96.22  Date Processed: 6/30/05 1:54:42 PM
  IPA Background Date Processed: 6/30/05 1:54:42 PM
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6/30/05 6:21:38 PM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 2

Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

3H Background CPM (0-18.6 keV): 14.70 Date Processed: 6/30/05 1:54:42 PM
14C Background CPM (0-156 keV): 22.12 Date Processed: 6/30/05 1:54:42 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMa	alpha2S%	SIS	
MESSAGES									
1	240.00	20.00	2.89	3.73	6.68	0.38	20.85	180.9	WCA 711/26
B									
Missing vial 2.									
3	1.14	6837.90	2.27	2332.23	3.88	8844.35	1.99	253.0	SPIKE 61A1-1
4	1.13	7003.01	2.25	2336.97	3.90	8905.81	1.99	251.8	" -2
5	0.89	13501.35	1.83	3860.31	3.41	11338.94	1.99	228.7	" -3
6	0.95	11976.85	1.88	3287.85	3.58	10638.56	1.99	219.4	" -4
7	2.23	3936.51	2.14	1500.75	3.46	4505.45	2.00	279.6	46A-3
8	2.21	4146.07	2.09	1663.69	3.30	4532.20	2.00	289.5	" -4
9	1.12	8089.83	2.10	3435.55	3.23	8980.87	1.99	299.0	" -5
10	1.16	7991.21	2.08	3391.09	3.19	8640.13	2.00	299.6	" -6
11	1.15	8213.05	2.06	3443.22	3.18	8728.31	2.00	294.7	" -7
12	1.16	7779.14	2.11	3177.30	3.30	8685.82	1.99	290.1	" -8
13	1.11	8287.21	2.09	3490.86	3.21	9044.66	2.00	296.8	" -9
14	1.12	8051.43	2.11	3381.98	3.25	8974.62	1.99	295.8	" -10
15	1.14	6781.76	2.28	2338.37	3.88	8865.41	1.99	255.5	61A1-5
16	1.13	6742.84	2.29	2107.77	4.10	8895.19	1.99	238.2	" -6
17	1.14	8343.16	2.05	3482.23	3.18	8856.63	1.99	287.4	" -7
18	1.15	8068.70	2.08	3254.53	3.27	8783.10	1.99	280.7	" -8

In order to compare results, a "mass correct" value was calculated by dividing the counts per minute (cpm) by the mass of the neptunium spike. Also the percent difference between duplicate pairs was calculated.

Table 1 contains the information from the initial analyses of Spikes 46A-3 to 10 and Spikes 61A1-1 to 4. Table 2 contains the information from the reanalyses of the spike from Table 1. Table 3 contains the information from the initial analyses of the new samples Spikes 61A1-5 to 8. Spikes 61A1-5+6 are exactly prepared like Spikes 61A1-1 to 4. Spikes 61A1-7+8 use an acid matrix spike of 0.4M instead of 0.02M. Table 4 compares the initial analyses of nep spikes from Table 1 with the reanalyses from Table 2

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TABLE 1. Initial Analyses of Spikes 46A-3 to 10 + 61A1-1 to 4

sample	Mass (g) of sample	cpm alpha	mass correct (cpm/samp mass)	% difference between dups	Source for LSA results
Spike 46A-3 (from AL2)	0.0504	4448.910	88272	-0.2	631/40
Spike-46A-4 (from AL2)	0.0511	4517.590	88407		631/40
Spike 46A-3 (from AL3)	0.0504	4475.100	88792	-1.3	631/49
Spike-46A-4 (from AL3)	0.0511	4596.440	89950		631/49
Spike 46A-5	0.1005	8707.35	86640	-1.4	696/71
Spike-46A-6	0.0994	8735.25	87880		696/71
Spike 46A-7	0.0994	8828.56	88819	1.9	696/100
Spike-46A-8	0.0996	8674.61	87094		696/100
Spike 46A-9	0.1015	8946.1	88139	-0.4	696/153
Spike-46A-10	0.1015	8979.14	88464		696/153
Spike 61A1-1	0.0904	8977.25	99306	3.0	711/36
Spike 61A1-2	0.0921	8873.9	96351		711/36
Spike 61A1-3	0.0983	11353.55	115499	4.4	711/39
Spike-61A1-4	0.0951	10496.49	110373		711/39

TABLE 2. Reanalyses of Spikes 46A-3 to 10 and 61A1-1 to 4
Source 711/44-46 ~~for~~ BW 7-1-05

sample	Mass (g) of sample	cpm alpha	mass correct (cpm/samp mass)	% difference between dups
Spike 46A-3	0.0504	4505.45	89394	0.8
Spike-46A-4	0.0511	4532.20	88693	
Spike 46A-5	0.1005	8980.87	89362	2.7
Spike-46A-6	0.0994	8640.13	86923	
Spike 46A-7	0.0994	8728.31	87810	0.7
Spike-46A-8	0.0996	8685.82	87207	
Spike 46A-9	0.1015	9044.66	89110	0.8
Spike-46A-10	0.1015	8974.62	88420	
Spike 61A1-1	0.0904	8844.35	97836	1.2
Spike 61A1-2	0.0921	8905.81	96697	
Spike 61A1-3	0.0983	11338.94	115350	3.0
Spike-61A1-4	0.0951	10638.56	111867	

7-7-05 BW

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TABLE 3. Initial Analyses of New Samples Spike 61A1-5 to 8
Source 711/44-46

sample	Mass (g) of vial + acid	Mass (g) of vial + acid + sample	Mass (g) of sample	cpm alpha	mass correct (cpm/samp mass)	% difference between dups
Spike 61A1-5	7.8236	7.9153	0.0917	8865.41	96678	-0.3
Spike-61A1-6	7.8992	7.9909	0.0917	8895.19	97003	
Spike-61A1-7*	7.9430	8.0415	0.0985	8856.63	89915	-1.5
Spike-61A1-8*	7.8806	7.9768	0.0962	8783.10	91300	

*Acid matirx 0.4 M instead of 0.02M HNO3

TABLE 4. Comparison of Initial Analyses with the Reanalyses for samples Spikes 4A 46A-3 to 10 and 61A1-1 to 4
BW 7-1-05
Source: 711/47

sample	average mass correct (cpm/samp mass) Initial Analyses	avearage mass correct (cpm/samp mass) Reanalyses	Percent Difference
Spike 46A-3 and 4	88855	89043	-0.2
Spike 46A-5and 6	87260	88142	-1.0
Spike 46A-7and 8	87956	87508	0.5
Spike 46A-9 and 10	88302	88765	-0.5
Spike 61A1-1 and 2	97828	97266	0.6
Spike 61A1-3 and 4	112936	113609	-0.6

For whatever reason, Spikes 61A1-3 and 4 are consistently high. Anticipate not using these values with the CAL series 7 to 10 data. Suggest using Spikes 61A1-1,-2,-5,-6 in combination, or a subset for the LSA results reported on 711/38-39. CAL series 7 to 10 will be reanalyzed with Spikes 61A1-5 and 6 as a check.

7-7-05 BW

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Preparation of QA Samples for Div 01 Cation Analyses

Task initially assigned to Bradley Werling and Marla Roberts

Objective:
To prepare a cation QA sample to include with samples sent to Div 01.

Target Concentrations:
10 ppm of calcium, magnesium, potassium, sodium, and iron

Reagents:
Spex Certi-Prep Instrument Calibration Standard 3, cat # CL-CAL-3, lot # 12-71MS
Type 1 water (Barnstead/Thermolyne model D11901 sn 1190010979691)

Equipment
10mL volumetric pipet
1000mL volumetric flask
1L HDPE bottle
disposable pipet
100mL pp beaker

Procedure:
A 1000mL vol. flask was filled with about 500mL of type 1 water. 10 mL (vol pipet) of Spex standard was transferred into the vol flask. The vol flask was briefly swirled. The vol flask was filled to mark with type 1 water. The final portion of water was added with a disposable pipet filled from a 100mL pp beaker. The vol flask was stoppered and thoroughly mixed. The solution was transferred to a labeled 1000mL HDPE bottle. The label contained the ID (cation QA std), date, initials, and scientific notebook reference.

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Sampling of CAL Series 7 and 8 Experimental Solutions for Div 01 Cation Analyses

Task initially assigned to Marla Roberts and Bradley Werling

Objective:

To characterize CAL series 7 and 8 experimental solution water chemistry at the conclusion of the sorption experiments by analyses of certain cations.

Initial Entry: 711/9-10

Samples and Reagents:

Experimental solutions: CAL7-Np1 to 12 and CAL8-Np1 to 12 (711/31-32).
QA solution: cation QA std (711/49)

Equipment

100 mL pp beakers
Filters – Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 µm pore size
30mL syringe, B-D # 309650
30mL pp bottles

Procedure:

The experimental solution was carefully decanted into a 100mL pp beaker. A syringe was used to collect as much sample as possible from this 100mL beaker. Next a filter was attached to the end of the syringe. The sample was dispensed into a labeled 30mL pp bottle. The label contained a Div 01 ID, initials, and date. This process was repeated for each of the experimental solutions.

Two duplicate QA samples were included in this sample set. Aliquots of cation QA std (711/49) were decanted into two labeled 30mL pp bottles.

The prefix for all Div 01 IDs was CAL78 which represented the experimental solution ID. The suffixes 1 to 12 were used for the individual CAL7 exp solutions. The suffixes 13 and 14 were used for the QA samples. The suffixes 15 to 26 were used for the CAL8 exp solutions. The complete legend relating the CNWRA ID with the DIV 01 IDs is on 711/51.

7-7-05 BW

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Legend for CNWRA + Div 01 IDs for CAL Series 7+8 experiment solution cation analyses

CNWRA Sample ID	Div 01 Sample ID
CAL7-Np1	CAL78-1
CAL7-Np2	CAL78-2
CAL7-Np3	CAL78-3
CAL7-Np4	CAL78-4
CAL7-Np5	CAL78-5
CAL7-Np6	CAL78-6
CAL7-Np7	CAL78-7
CAL7-Np8	CAL78-8
CAL7-Np9	CAL78-9
CAL7-Np10	CAL78-10
CAL7-Np11	CAL78-11
CAL7-Np12	CAL78-12
CAL78-13	CAL78-13
CAL78-14	CAL78-14
CAL8-Np1	CAL78-15
CAL8-Np2	CAL78-16
CAL8-Np3	CAL78-17
CAL8-Np4	CAL78-18
CAL8-Np5	CAL78-19
CAL8-Np6	CAL78-20
CAL8-Np7	CAL78-21
CAL8-Np8	CAL78-22
CAL8-Np9	CAL78-23
CAL8-Np10	CAL78-24
CAL8-Np11	CAL78-25
CAL8-Np12	CAL78-26

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Initial Entry – M Series 7 and CAL Series 11 Sorption Experiments – Np-237 on Montmorillonite and Consolidated Alluvium with 19PB Simulated Waters with calcium over a pH range

Personnel initially assigned to task: Bradley Werling and Marla Roberts

Objective: To investigate Np sorption on a montmorillonite and consolidated alluvium from NC-EWDP-19PB using simulated water (with calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB over a range of pHs.

Conditions:
- $\sum$Np = 200 ppb
- equilibrium with lab CO_2(g); $pCO_2 = 10^{-3.42}$
- solution volume = 30 ml
- mass of solid = 0.1 g
- M/V (g/L) = 3.33

Samples
19PB Simulated water with Ca - 696/14 and 32
NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66. (<35 mesh size fraction)
Montmorillonite: SAZ*Na*<2*FD*Scr - 061/94

Reagents
Neptunium Spike # 61A-1 (369/100) - 60 ppm Np-237 at pH of 5.63
pH buffer 4 (Fisher SB98 lot # 041614)
pH buffer 7 (Fisher SB108 lot # 044065)
pH buffer 10 (Fisher SB 116 lot # 044060)
Ultima-Gold AB liquid scintillation cocktail (Packard 6013309 lot # 91-050201)
0.02 M HNO_3 (SN 696/29-30) for LSA matrix
0.4 M HNO3 (696/195) for pH adjustment
0.1 M NaOH (696/30) for pH adjustment
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Equipment:
Orion meter 920A (sn 039518)
Thermo Orion combination electrode 8103BN (ID 3G)
New Brunswick Scientific Gyratory shaker model G33 (sn 290127990)
Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
Mettler AE240 balance (sn 101237)
Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
Stir Plate

Supplies:
Pipettes and tips
Repipettor for transfer of scintillation cocktail
Polycarbonate centrifuge tubes with caps (50 ml capacity)
7 ml scintillation vials
Weighing paper/boats
10 mL microbeakers
Plasticware and glassware as needed
Squirt bottle for water
Stir bars
Kimwipes

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Basic Outline of Procedure
- Equilibrate simulated water sample with laboratory CO_2.
- Measure the pH of simulated water samples.
- Add solids to experimental solutions.
- Add simulated water to experimental solutions.
- Add pH adjustment solutions to experimental solutions.
- Add neptunium spike to experimental solutions.
- Place experimental solutions on activated gyrator
- During the experiment, completely remove the experimental solution caps during the regular business hours. Loosely cap otherwise.
- Analyze aliquots of experimental solution for pH and neptunium concentration (LSA)

Equilibration of Simulated Waters used in M Series 7 and CAL Series 11 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To equilibrate with ambient atmosphere the simulated water used in the experimental solutions.

Initial Entry: 711/52-53

Procedure

Aeration of simulated water conducted since 23 Feb 2005 – see 696/14 and 32

To Page No._____

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by

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pH Analyses of Simulated Water for use in M Series 7 and CAL Series 11 Experimental Solutions.

Bradley Werling will be performing this task.

Objective: To determine the pH of the simulated water to be used in sorption experiments.

Initial Entry: 711/52-53

Basic Outline of Procedure

Fresh pH buffers 4, 7 and 10 (60mL bottles with stir bars) were used to calibrate the pH meter (using stir plate). The temperature, calibration setpoints and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

About a 10 mL aliquot of the simulated water was poured out of the carboy into a small glass beaker. This aliquot was transferred into a 10 mL microbeaker with stir bar. The aliquot was placed on a stir plate and then the stir plate was activated. The pH was recorded. pH meter challenged with one of the calibration standards. The pH and solution ID were recorded. The solutions in the microbeakers were not transferred back to into the original experimental solution.

Temp = 20.7 Cal Set pts 4.01, 7.01, 10.06 Slope = 99.2

SAMPLE ID	pH
19PB simulated water with CA	8.67
pH buffer 7 (target 7.01)	7.04

CHALLAGE OF BALANCE
- Masses off campus for calibration check.
- No mass challanges performed on 8 July 05

7-8-05 BAW

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Addition of solids to M Series 7 and CAL Series 11 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To prepare the experimental solutions by adding the solid substrate.

Initial Entry: 711/52-53

Sample info NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66. (<35 mesh size fraction)
Montmorillonite: SAZ*Na*<2*FD*Scr - 061/94

Procedure

Two sets of twelve polycarbonate centrifuge tubes were labeled as follows: M7-Np1 to 12 and CAL11-Np1 to 12. The mass of each tube (with cap) was measured and recorded on 711/55-56. An individual experimental tube was uncapped and a funnel made from weighing paper was inserted in the top. Approximately 0.1 g of solid was transferred to a piece of weighing paper that was tared on the balance. The solid was transferred into the appropriate experimental tube (montmorillonite for M7 and consolidated alluvium for CAL11) and the tube was capped. This "solid addition" step was repeated until all experimental tubes contained the appropriate solid. The mass of each experimental tube was measured again and recorded on 711/55-56.

SAMPLE ID	Mass (g) of empty tube	Mass (g) of tube + solid.
M7-Np1	21.9860	22.0891
M7-Np2	21.9955	22.0962
M7-Np3	22.1127	22.2126
M7-Np4	21.9407	22.0423
M7-Np5	22.1374	22.2402
M7-Np6	22.0240	22.1244
M7-Np7	22.1632	22.2611
M7-Np8	22.1496	22.2524
M7-Np9	22.0584	22.1610
M7-Np10	22.2651	22.3662
M7-Np11	21.9442	22.0544
M7-Np12	22.3147	22.4150

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SAMPLE ID	Mass (g) of empty tube	Mass (g) of tube + solid.
CAL 11-Np1	22.2009	22.3075
CAL 11-Np2	22.0367	22.1389
CAL 11-Np3	22.3472	22.4451
CAL 11-Np4	22.2280	22.3276
CAL 11-Np5	22.2615	22.3601
CAL 11-Np6	21.9457	22.0450
CAL 11-Np7	22.2499	22.3503
CAL 11-Np8	22.1063	22.2079
CAL 11-Np9	22.1085	22.2108
CAL 11-Np10	22.1621	22.2597
CAL 11-Np11	22.2273	22.3273
CAL 11-Np12	22.2321	22.3312

Addition of Simulated Water solution to M Series 7 and CAL Series 11 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To prepare the experimental solutions by adding the simulated water solution.

Initial Entry: 711/52-53

Sample info 19PB Simulated water with Ca - 696/14 and 32

Procedure

About 30mL (10mL pipet) of the simulated water was added to the experimental pc tube. The mass was recorded on 711/57.

7-8-05 BW

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SAMPLE ID	Mass (g) of tube + solid + sim water
M7-Np1	52.2392
M7-Np2	52.4840
M7-Np3	52.4370
M7-Np4	52.1794
M7-Np5	52.4572
M7-Np6	52.3811
M7-Np7	52.4464
M7-Np8	52.4661
M7-Np9	52.3906
M7-Np10	52.6009
M7-Np11	52.3180
M7-Np12	52.6563
CAL11-Np1	52.5230
CAL11-Np2	52.4342
CAL11-Np3	52.6528
CAL11-Np4	52.5763
CAL11-Np5	52.6141
CAL11-Np6	52.2989
CAL11-Np7	52.6086
CAL11-Np8	52.5244
CAL11-Np9	52.5490
CAL11-Np10	52.6138
CAL11-Np11	52.6250
CAL 11-Np12	52.5668

Addition of pH Adjusting solution to M Series 7 and CAL Series 11 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To prepare the experimental solutions by adding the pH adjusting solution.

Initial Entry: 711/52-53

Procedure

Used the same adjustment scheme as Cal Series 9 and 10 (711/21). Used Adjustable eppendorfs 10-100µL and 100-1000µL. Recorded mass of experimental solution after pH adjusting solutions added – 711/58.

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SAMPLE ID	Mass (g) after pH adjustment	Mass (g) after Np spike
M7-Np1	52.4581	52.5555
M7-Np2	52.7000	52.7915
M7-Np3	52.6476	52.7452
M7-Np4	52.3830	52.4756
M7-Np5	52.6641	52.7614
M7-Np6	52.5837	52.6814
M7-Np7	52.6438	52.7404
M7-Np8	52.6420	52.7395
M7-Np9	52.5303	52.6233
M7-Np10	52.6961	52.7941
M7-Np11	52.3301	52.4297
M7-Np12	53.1667	53.2657
CAL11-Np1	52.7502 7-8-05 MR	~~52.84~~ 52.8485
CAL11-Np2	52.6550	52.7534
CAL11-Np3	52.8695	52.9680
CAL11-Np4	52.7877	52.8855
CAL11-Np5	52.8202	52.9182
CAL11-Np6	52.4992	52.5973
CAL11-Np7	52.8038	52.9013
CAL11-Np8	52.6998	52.7984
CAL11-Np9	52.6884	52.7855
CAL11-Np10	52.7149	52.8130
CAL11-Np11	52.6364	52.7341
CAL11-Np12	53.0811	53.1790

7-8-05 BAW

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Witnessed & Understood by me, | Date | Invented by | Date
Recorded by

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Addition of Neptunium Spike to M Series 7 and CAL Series 11 Experimental Solutions

Personnel initially assigned to task: Bradley Werling

Objective: To prepare the experimental solutions by adding the neptunium spike.

Initial Entry: 711/52-53

Reagent: Np Spike # 61A-1 (369/100)

Procedure

Added 100µL (100µL eppendorf pipet) to each experimental solution and recorded the mass of the experimental solution on 711/58

Running of M series 7 and CAL series 11 Sorption Experiment

Initial Entry: 711/52-53

Each experimental soln (711/58) was vortexed for about 30 seconds (after np-spike). The caps were loosened and they were placed on an activated gyrator (~100 RPM).

11 JULY 05 BAW

RUNNING OF M Series 7 and CAL series 11 Sorp Exp

Initial Entry: 711/52-53

Experimental Solutions (711/59) were uncapped and placed on an activated gyratory shaker (~100 RPM) at 0915. At 1645 exp solns were tightly capped and each one vortexed for about 10 seconds. Exp solutions were loosely capped and placed on activated gyratory shaker again (RPM~100).

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Recorded by

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Book No.
TITLE 11 July 05 cont BAW

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Delivery of CAL series 7+8 sample to DivOI for cation analyses

Initial Entry for Sorption Experiment: 711/9-10
Samples: 12 CAL Series 7
12 CAL Series 8
2 QAs
Sample + QA collection: 711/50
Sample ID legend + BW 7-11-05 711/51
QA target conc: 711/49
Chain of Custody on following pages

7-11-05
BAW

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Witnessed & Understood by me, | Date | Invented by | Date
Recorded by

TITLE 11 July 05 cont BAW
Project No.
Book No.

From Page

SAMPLE LIST/CHAIN OF CUSTODY
Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID | Site/Zone ID

Shipper Name/Address: Bradley Werling, DIV20, B19.57
Client

Requested Turnaround: ☐ 2 Weeks ☐ 3 Weeks ☒ Other: 4 weeks
SwRI Contact: Mike Dammann

REMARKS
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) None

Analyses Requested

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Al, Si, Mn, Mg, Ca, K, Na, Sr	by ICP
CAL78-1	7-8-05		W		1	X	
CAL78-2						X	
CAL78-3						X	
CAL78-4						X	
CAL78-5						X	
CAL78-6						X	
CAL78-7						X	
CAL78-8						X	
CAL78-9					1	X	
CAL78-10					1	X	

Nuclear Safety Related - Use appropriate QA procedure
PIC - Brad Werling
ext. 105-05
FAX. 5184

SwRI Project#: 20.06.002.01.242

Relinquished by (Print/Signature): BRAD WERLING / Brad Werling — Date 7-11-05 Time 1417
Received by (Print/Signature) — Date — Time
Relinquished by (Print/Signature) — Date — Time
Received by (Print/Signature) — Date — Time
Relinquished by (Print/Signature) — Date — Time
Received by SwRI lab: (Signature) — Date 7/11/05 Time 1420
Samples Disposed: Date — Time
Samples Disposed by:

Sample Types:
D - Duplicate
ER – Equipment Rinsate
ES – Environmental Sample
FB – Field Blank
FD – Field Duplicate
MS – Matrix Spike
MSD – Matrix Spike Dup
TB – Trip Blank

Matrix Types:
A – Air
B – Biota
D - Dust
E – Emission/Stack
L - Liquid
P – Product
Sd - Solid
S – Soil
SED – Sediment
T - Tissue
W - Water
WP - Wipe

Temp: 22.0°C
Therm #: 067
Comments: BADGE ACTIVE - NP237 at 8.5x10⁻⁷ M or 1.4x10⁻⁴ µCi/mL SN 711150

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Div 01 COC Form 01-01-001, Rev 8/02

Witnessed & Understood by me, | Date | Invented by | Date
Recorded by

Project No.____
Book No.____
TITLE 11 July 05 cont Bradley

Witnessed & Understood by me,
Date
Invented by
Recorded by
Date

Shipper Name/Address: Bradley Werling DIV 20 Blg 57
Client:

SAMPLE LIST/CHAIN OF CUSTODY
Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID: LP
Site/Zone ID:
Analyses Requested: Al, Si, Mn, Mg, Ca, K, Na, Sr by ICP

Requested Turnaround:
☐ 2 Weeks
☐ 3 Weeks
☒ Other: 4 weeks

SwRI Contact: Mike Dammann

REMARKS
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) None

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Al, Si, Mn, Mg, Ca, K, Na, Sr	Remarks
CAL78-11	7-8-05		W		1	X	Nuclear Safety
CAL78-12						X	Related - use
CAL78-13						X	appropriate
CAL78-14						X	QA procedure
CAL78-15						X	
CAL78-16						X	POC - Brad
CAL78-17						X	Werling x 6565
CAL78-18						X	fax x 5184
CAL78-19						X	
CAL78-20						X	

Matrix Types:
A – Air
B – Biota
D - Dust
E – Emission/Stack
L - Liquid
P – Product
Sd - Solid
S – Soil
SED – Sediment
T - Tissue
W - Water
WP - Wipe

Sample Types:
D - Duplicate
ER – Equipment Rinsate
ES – Environmental Sample
FB – Field Blank
FD – Field Duplicate
MS – Matrix Spike
MSD – Matrix Spike Dup
TB – Trip Blank

Temp: 27.0°C
Therm #: 027
Comments: RADIOACTIVE - Np 237 at 8.5×10^{-7} M or 1.4×10^{-4} µCi/mL SN 711/50

Relinquished by (Print/Signature)	Date	Time
Brad Werling / Brad Werling	7-11-05	14:17
Received by (Print/Signature)		
Relinquished by (Print/Signature)		
Received by (Print/Signature)		
Relinquished by (Print/Signature)		

SwRI Project#: 20.06002.01.242
Received by SwRI Lab: (Signature)
Date: 7/11/05 Time: 1420
Samples Disposed: Date ____ Time ____
Samples Disposed by:

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Page No.

From Page

Project No.____
Book No.____
TITLE 11 July 05 cont Bradley

Witnessed & Understood by me,
Date
Invented by
Recorded by
Date

Shipper Name/Address: BRADLEY WERLING DIV 20 BLG 57
Client:

SAMPLE LIST/CHAIN OF CUSTODY
Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID:
Site/Zone ID:
Analyses Requested: Al, Si, Mn, Mg, Ca, K, Na, Sr by ICP

Requested Turnaround:
☐ 2 Weeks
☐ 3 Weeks
☒ Other: 4 weeks

SwRI Contact: Mike Dammann

REMARKS
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) none

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Al, Si, Mn, Mg, Ca, K, Na, Sr by ICP	Remarks
CAL78-21	7-8-05		W		1	X	Nuclear Safety
CAL78-22						X	Related - use
CAL78-23						X	appropriate QA
CAL78-24						X	procedure
CAL78-25						X	
CAL78-26						X	POC - Brad Werling
							phone x 6565
							fax x 5184

Matrix Types:
A – Air
B – Biota
D - Dust
E – Emission/Stack
L - Liquid
P – Product
Sd - Solid
S – Soil
SED – Sediment
T - Tissue
W - Water
WP - Wipe

Sample Types:
D - Duplicate
ER – Equipment Rinsate
ES – Environmental Sample
FB – Field Blank
FD – Field Duplicate
MS – Matrix Spike
MSD – Matrix Spike Dup
TB – Trip Blank

Temp: 27.0°C
Therm #: 027
Comments: RADIOACTIVE - Np 237 at 8.5×10^{-7} M or 1.4×10^{-4} µCi/mL SN 711/50

Relinquished by (Print/Signature)	Date	Time
Brad Werling / Brad Werling	7-11-05	14:17
Received by (Print/Signature)		
Relinquished by (Print/Signature)		
Received by (Print/Signature)		
Relinquished by (Print/Signature)		

SwRI Project#: 20.06002.01.242
Received by SwRI Lab: (Signature)
Date: 7/11/05 Time: 1420
Samples Disposed: Date ____ Time ____
Samples Disposed by:

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Running of M Series 7 and CAL series 11 Sorption Exp

Initial Entry: 711/52-53

Procedure Followed: 11 July 05 711/59

Time uncapped: 11:05 Time loosely capped = 16:30

13 JULY 05 BAW

Running of M series 7 and CAL series 11 Sorption Exp

Initial Entry: 711/52-53

Procedure Followed: 11 July 05 711/59

Time uncapped: 0900 Time loosely capped = na - left uncapped overnight. (unintentionally)

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Liquid Scintillation Renalyses Results for CAL series 7, 8, 9, + 10 Experimental Solutions

Task initially assigned to Bradley Werling

Objective

To determine the amount of neptunium in the experimental solution at the end of the sorption experiments. The LSA results for the neptunium spikes were higher than expected while the samples results were as expected.

Initial Entry 711/9-10

LSA sample preparation 711/26-30

Initial LSA results: 711/36-39

Background information concerning reanalyses: 711/42-48

Reanalyses initiated on 6 July 2005. The same samples and blanks were reanalyzed. Neptunium spikes 61A1-5 and 61A1-6 were used (711/43-44)

Sample Info

Blank (one sample from simulated water with calcium): w Ca 711/26
Neptunium spikes (two samples) – Spike 61A1-5 and Spike 61A1-6
Four experiments derived from 1 solid and 2 types of simulated waters (7+9 duplicate experiments and 8+10 duplicate experiments).
Consolidated Alluvium series 7 (24 samples) using simulated water with calcium
Consolidated Alluvium series 8 (24 samples) using simulated water without calcium.
Consolidated Alluvium series 9 (24 samples) using simulated water with calcium
Consolidated Alluvium series 10 (24 samples) using simulated water without calcium.

Results

Printout of LSA analyses following:

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		Recorded by	

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Book No._____ TITLE 13 JULY 05 CONT BAW

7/12/05 12:26:17 AM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 1

Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

Assay Definition-

Assay Description:

Assay Type: Alpha/Beta
Report Name: Np_Pa_Exp
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\20050706_1848.results
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.038
Assay File Name: C:\Packard\TriCarb\Assays\Pa_Np_Exp_AB.lsa

Count Conditions-

Nuclide: NP/PA
Quench Indicator: SIS
External Std Terminator (sec): n/a
Pre-Count Delay (min): 0.00
Alpha/Beta Standards:
Low Energy: NP/PA
Count Time (min): 240.00
Count Mode: Normal
Assay Count Cycles: 1 Repeat Sample Count: 1
#Vials/Sample: 1 Calculate % Reference: Off

Background Subtract: On - 1st Vial
Low CPM Threshold: Off
2 Sigma % Terminator: On - Any Region

In Use Discriminator: 143

Regions	LL	UL	Bkg Subtract	2Sigma % Terminator
Beta A	0.0	400.0	1st Vial	0.00
Beta B	100.0	400.0	1st Vial	0.00
Alpha	100.0	400.0	1st Vial	2.00

Count Corrections-

Static Controller: On Luminescence Correction: Off
Colored Samples: n/a Heterogeneity Monitor: n/a
Coincidence Time (nsec): 18 Delay Before Burst (nsec): 75

Half Life-

Half Life Correction: Off

Regions	Half Life	Units	Reference Date	Reference Time
Beta A				
Beta B				
Alpha				

IPA Block Data
Software Version IC: 2.11
Software Version EC: 1.31
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 19.32 Date Processed: 7/6/05 6:48:49 PM
14C Chi Square: 36.27 Date Processed: 7/6/05 6:48:49 PM
3H E^2/B (1-18.6 keV): 284.58 Date Processed: 7/6/05 6:48:49 PM
14C E^2/B (4-156 keV): 544.40 Date Processed: 7/6/05 6:48:49 PM
3H Efficiency (0-18.6 keV): 65.06 Date Processed: 7/6/05 6:48:49 PM
14C Efficiency (0-156 keV): 96.73 Date Processed: 7/6/05 6:48:49 PM
IPA Background Date Processed: 7/6/05 6:48:49 PM

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

TITLE 13 July 05 CONT BAW Project No._____
Book No._____

Fro 7/12/05 12:26:19 AM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 2

Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

3H Background CPM (0-18.6 keV): 15.00 Date Processed: 7/6/05 6:48:49 PM
14C Background CPM (0-156 keV): 21.77 Date Processed: 7/6/05 6:48:49 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMa	alpha2S%	SIS	
MESSAGES									
1	240.00	19.87	2.90	3.35	7.05	0.38	20.97	168.1	WCA 7/1/26
B									
Missing vial 2.									CAL series 7
3	71.60	39.05	4.87	16.88	6.45	139.29	2.01	293.5	>Np1<a/b
4	71.79	37.94	4.97	16.04	6.65	138.92	2.01	292.2	
5	71.02	42.04	4.65	17.18	6.41	140.51	2.01	283.0	>Np2<a/b
6	70.82	43.01	4.58	17.78	6.29	140.84	2.01	286.0	
7	68.00	41.95	4.75	16.93	6.60	146.68	2.01	281.2	>Np3<a/b
8	65.15	42.35	4.81	16.62	6.81	153.16	2.01	276.2	
9	68.56	42.89	4.66	16.86	6.59	145.49	2.01	275.8	>Np4<a/b
10	67.21	42.31	4.75	17.37	6.54	148.41	2.01	286.6	
11	68.73	42.61	4.67	18.02	6.33	145.15	2.01	286.4	>Np5<a/b
12	67.93	42.69	4.69	18.20	6.33	146.83	2.01	290.1	
13	67.95	41.47	4.79	17.82	6.40	146.85	2.01	291.6	>Np6<a/b
14	68.56	42.51	4.69	17.65	6.41	145.49	2.01	286.7	
15	68.75	41.76	4.74	16.43	6.69	145.08	2.01	278.9	>Np7<a/b
16	68.34	40.71	4.84	16.58	6.67	145.96	2.01	282.7	
17	69.37	40.89	4.79	16.64	6.61	143.79	2.01	283.2	>Np8<a/b
18	70.31	42.25	4.65	17.71	6.32	141.85	2.01	290.2	
19	69.96	42.69	4.63	18.04	6.27	142.56	2.01	290.6	>Np9<a/b
20	69.99	41.51	4.72	16.95	6.51	142.56	2.01	286.7	
21	71.03	40.70	4.75	16.47	6.57	140.45	2.01	286.8	>Np10<a/b
22	71.24	42.93	4.57	17.94	6.24	140.01	2.01	281.3	
23	68.49	44.39	4.55	18.66	6.21	145.63	2.01	289.5	>Np11<a/b
24	66.81	42.72	4.73	17.86	6.45	149.33	2.01	288.4	
25	161.73	18.79	6.04	7.70	7.45	61.45	2.02	287.2	>Np12<a/b
26	164.51	19.73	5.77	8.10	7.14	60.41	2.02	281.6	
Missing vial 27.									CAL series 8
28	68.49	48.99	4.26	19.79	6.00	145.63	2.01	283.5	>Np1<a/b
29	68.55	50.03	4.20	20.51	5.87	145.51	2.01	285.6	
30	72.73	44.72	4.41	18.52	6.06	137.14	2.01	284.7	>Np2<a/b
31	71.49	45.97	4.36	19.32	5.96	139.50	2.01	287.9	
32	70.18	45.45	4.43	18.29	6.21	142.11	2.01	278.9	>Np3<a/b
33	70.57	45.40	4.42	18.82	6.09	141.34	2.01	290.7	
34	73.59	43.71	4.45	17.16	6.30	135.51	2.01	273.9	>Np4<a/b
35	73.57	42.42	4.55	17.56	6.22	135.60	2.01	287.6	
36	71.81	45.47	4.38	17.94	6.21	138.88	2.01	276.0	>Np5<a/b
37	72.50	44.96	4.40	18.67	6.04	137.55	2.01	286.3	
38	71.25	46.82	4.31	18.75	6.07	139.97	2.01	280.1	>Np6<a/b
39	72.15	46.95	4.28	19.00	5.99	138.22	2.01	285.1	
40	71.29	45.85	4.37	18.35	6.15	139.89	2.01	279.6	>Np7<a/b
41	71.44	47.74	4.25	19.63	5.90	139.61	2.01	285.8	
42	73.07	43.86	4.46	18.45	6.06	136.49	2.01	290.3	>Np8<a/b
43	72.46	44.66	4.42	18.51	6.07	137.64	2.01	284.2	
44	71.86	47.54	4.25	19.25	5.96	138.78	2.01	278.9	>Np9<a/b
45	71.35	46.18	4.35	17.74	6.27	139.78	2.01	272.0	
46	69.72	46.61	4.37	18.93	6.10	143.11	2.01	275.8	>Np10<a/b
47	69.46	46.37	4.39	18.46	6.21	143.59	2.01	281.7	
48	67.79	47.65	4.36	20.01	5.99	147.21	2.01	286.8	>Np11<a/b
49	68.30	48.42	4.30	20.98	5.80	146.08	2.01	292.0	
50	76.97	46.48	4.18	18.99	5.81	129.54	2.01	291.4	>Np12<a/b
51	73.67	46.04	4.29	19.18	5.90	135.39	2.01	286.0	
Missing vial 52.									

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								(CAL series 9)
53	66.95	48.78	4.32	19.77	6.06	148.99	2.01	284.9 >Np1<a/b
54	67.38	49.56	4.26	20.32	5.95	148.08	2.01	282.8
55	67.49	48.98	4.29	19.27	6.13	147.84	2.01	278.3 >Np2<a/b
56	68.31	49.53	4.23	21.09	5.78	146.01	2.01	290.5
57	70.89	50.94	4.08	20.57	5.76	140.73	2.01	284.5 >Np3<a/b
58	71.68	48.73	4.19	20.00	5.83	139.13	2.01	285.9
59	75.61	47.36	4.16	19.43	5.78	131.88	2.01	286.8 >Np4<a/b
60	74.05	47.71	4.18	19.23	5.87	134.69	2.01	278.3
61	72.51	47.39	4.24	19.42	5.90	137.55	2.01	284.6 >Np5<a/b
62	72.02	46.50	4.31	18.60	6.07	138.50	2.01	276.8
63	71.87	47.04	4.28	19.58	5.90	138.76	2.01	284.4 >Np6<a/b
64	72.31	47.38	4.25	18.80	6.02	137.93	2.01	279.7
65	70.77	48.70	4.21	18.69	6.11	140.94	2.01	273.8 >Np7<a/b
66	71.02	46.46	4.34	19.06	6.02	140.44	2.01	281.8
67	75.92	44.64	4.33	18.63	5.91	131.35	2.01	290.9 >Np8<a/b
68	75.60	44.16	4.37	17.98	6.05	131.92	2.01	284.2
69	77.32	43.76	4.35	17.49	6.09	128.97	2.01	281.4 >Np9<a/b
70	78.38	43.61	4.33	17.62	6.02	127.20	2.01	281.8
71	71.29	49.07	4.18	19.43	5.95	139.92	2.01	281.4 >Np10<a/b
72	70.27	48.10	4.26	19.79	5.92	141.94	2.01	289.5
73	73.05	48.00	4.19	19.84	5.80	136.51	2.01	283.4 >Np11<a/b
74	73.33	47.96	4.19	20.35	5.71	135.99	2.01	289.0
75	156.75	22.66	5.25	9.76	6.40	63.42	2.02	290.1 >Np12<a/b
76	157.22	23.08	5.17	9.23	6.64	63.23	2.02	279.4
Missing vial 77.								
								(CAL series 10)
78	71.24	48.05	4.24	19.67	5.90	140.01	2.01	284.5 >Np1<a/b
79	71.83	49.01	4.16	20.20	5.79	138.88	2.01	286.1
80	70.76	52.60	4.00	21.07	5.69	140.96	2.01	284.2 >Np2<a/b
81	71.57	52.62	3.98	21.28	5.62	139.34	2.01	281.0
82	71.64	49.91	4.12	21.12	5.65	139.21	2.01	291.5 >Np3<a/b
83	71.57	49.49	4.14	19.84	5.86	139.34	2.01	285.5
84	71.74	50.49	4.08	19.92	5.84	139.03	2.01	278.4 >Np4<a/b
85	71.50	52.90	3.97	21.72	5.56	139.49	2.01	282.8
86	70.80	52.53	4.00	20.81	5.73	140.88	2.01	278.5 >Np5<a/b
87	71.27	49.47	4.15	20.68	5.73	139.96	2.01	290.2
88	79.03	47.95	4.05	19.61	5.63	126.18	2.01	282.5 >Np6<a/b
89	79.69	46.22	4.13	18.69	5.77	125.13	2.01	283.8
90	72.12	51.47	4.02	20.27	5.77	138.29	2.01	277.7 >Np7<a/b
91	70.96	52.30	4.01	21.29	5.65	140.56	2.01	281.6
92	72.53	50.79	4.05	21.42	5.57	137.51	2.01	288.7 >Np8<a/b
93	72.29	53.28	3.93	21.89	5.51	137.97	2.01	289.3
94	73.36	52.88	3.92	21.90	5.47	135.93	2.01	288.7 >Np9<a/b
95	72.29	54.77	3.86	21.75	5.53	137.95	2.01	275.2
96	72.81	53.14	3.92	21.20	5.59	136.96	2.01	286.8 >Np10<a/b
97	71.87	51.72	4.02	21.15	5.63	138.79	2.01	286.8
98	70.66	54.26	3.92	21.92	5.56	141.16	2.01	284.7 >Np11<a/b
99	69.87	54.15	3.95	22.71	5.48	142.76	2.01	289.0
100	73.49	52.57	3.93	20.91	5.61	135.69	2.01	280.6 >Np12<a/b
101	74.78	53.09	3.88	22.59	5.32	133.35	2.01	291.4
Missing vial 102.								
103	1.10	6962.86	2.29	2773.01	3.62	9116.89	2.00	284.4 Spike 61A1-5
104	1.13	7081.90	2.24	2671.87	3.64	8944.75	1.99	274.6 Spike 61A1-6

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Running of N series 7 and CAL series 11 Sorption Exp

Initial Entry: 711/52-3

Procedure followed: 11 July 05 711/59

Time uncapped: na- left uncapped overnight (711/64)

Time loosely capped: 16:50

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Liquid Scintillation and pH Analyses of M series 7 (montmorillonite) and CAL series 11 (consolidated alluvium) – Np-237 in one type of simulated groundwater (with Ca) over a pH range

Task initially assigned to Bradley Werling and Marla Roberts.

Objective: To determine the neptunium concentration and pH of experimental solutions and use in calculations for determining the sorption coefficient (Kd) under specified conditions

Initial Entry 711/52-53

Sample Info Two types of experiments run. Experiments based on montmorillonite and consolidated alluvium substrate from NC-EWDP-19PB and one simulated water (with calcium) based on 19PB shallow string groundwater.

Procedure

LSA vials (size 7 mL) were labeled for experimental solutions, blank solution, and neptunium spikes solutions. Experimental solutions were analyzed in duplicate and labeled to identify the sorption solid phase and series, the radionuclide, the experimental solution number, and the a/b duplicate designator (example – CAL11-Np1a and CAL11-Np1b). One simulated water blank solution was analyzed and labeled as w CA with scientific notebook page reference. The neptunium spike was analyzed in duplicate and labeled Spike 61A1-9 and 61A1-10.

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.5 mL (500µL eppendorf pipet) of 0.02 M HNO_3 added. The mass of all of the LSA vials with acid was recorded (See table 1 column 1).

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (See table 2, column 1). The tubes containing the experimental solutions were placed in a tared 30 mL polypropylene beaker when being weighed.

For experimental solutions, 0.5 mL (500µL eppendorf pipet) of the experimental solution was transferred into the appropriate LSA vial. For the simulated water blank, 0.5 mL (500µL eppendorf pipet) of simulated water was added to the LSA vial. For the neptunium spike solutions, 0.1 mL (100µL eppendorf pipet) of the spike was added into each LSA vial. The mass of all of the LSA vials was recorded after the addition of the appropriate sample solution (See table 1 column 2).

The pH of the experimental solutions was taken. Fresh pH buffers of 4, 7 and 10 (60mL bottles with stir bars) were used to calibrate the pH meter (using stir plate). The temperature, calibration setpoints and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

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A 10 mL aliquot (pipet with disposable 10 mL tip) of experimental solution was transferred into a 10 mL microbeaker with stir bar. A stir plate was used when measuring the pH of the experimental solutions. The pH was recorded (See table 2, column 2). pH meter challenged with one of the calibration standards after all of the sample pHs were measured. The challenge solution type and measurement was recorded. The solutions in the microbeakers were transferred back to into the original experimental solution.

All LSA vials had 5 mL (Repipet bottle top dispenser) of LSA cocktail added. The LSA vials were tightly capped and rigorously shaken by hand.

CHALLANGE OF BALANCE (TARGET = 20.0001g)
At start of day = 20.0001g At end of day = 20.0002g

TABLE 1 - LSA info

ID	Mass (g) of vial + acid	Mass (g) of vial + acid + sample
w CA 711/70	7.8533	8.3476
M7-Np1a	7.9261	8.4127
M7-Np1b	7.9504	8.4397
M7-Np2a	7.9064	8.3993
M7-Np2b	7.8987	8.3857
M7-Np3a	7.8962	8.3895
M7-Np3b	7.9504	8.4478
M7-Np4a	7.8428	8.3371
M7-Np4b	7.9242	8.4213
M7-Np5a	7.8602	8.3434
M7-Np5b	7.8138	8.3097
M7-Np6a	7.9119	8.4130
M7-Np6b	7.8496	8.3456
M7-Np7a	7.8704	8.3569
M7-Np7b	7.8539	8.3520
M7-Np8a	7.8360	8.3313
M7-Np8b	7.8691	8.3652
M7-Np9a	7.8775	8.3593* cocktail added
M7-Np9b	7.8450	8.3296 immediately
M7-Np10a	7.9115	8.4046

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From Page No. TABLE 1 cont - LSA in G

ID	Mass(g) of vial + acid	Mass(g) of vial + acid + sample
M7-Np10b	7.8596 ~~7.9116~~ BWS 7-15-05	8.3553
M7-Np11a	7.9022	8.3953
M7-Np11b	7.8638	8.3563
M7-Np12a	7.9133	8.4046
M7-Np12b	7.8480	8.3384
CAL11-Np1a	7.8857	8.3784
CAL11-Np1b	7.9580	8.4477
CAL11-Np2a	7.8989	8.3827
CAL11-Np2b	7.9056	8.4029
CAL11-Np3a	7.9261	8.4141
CAL11-Np3b	7.9686	8.4618
CAL11-Np4a	7.8678	8.3579
CAL11-Np4b	7.9021	8.3949
CAL11-Np5a	7.8651	8.3566
CAL11-Np5b	7.8671	8.3604
CAL11-Np6a	7.8116	8.3072
CAL11-Np6b	7.8487	8.3444
CAL11-Np7a	7.9095 ~~7.8780~~ BWS 7-15-05	8.4047
CAL11-Np7b	7.8479	8.3418
CAL11-Np8a	7.8962	8.3902
CAL11-Np8b	7.8582	8.3532
CAL11-Np9a	7.9437	8.4366
CAL11-Np9b	7.9436	8.4396
CAL11-Np10a	7.9557	8.4493
CAL11-Np10b	7.9348	8.4290
CAL11-Np11a	7.8894	8.3814
CAL11-Np11b	7.8662	8.3532
CAL11-Np12a	7.8772	8.3702
CAL11-Np12b	7.8984	8.3918
Spike 61A1-9	7.8587	7.9519
Spike 61A1-10	7.8389	7.9374

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From Page No. pH analyses recorded in Table 2

Temp = 21.0°C Setpts = 4.01, 7.01, 10.05 Slope = 99.2

Challange after analyses: Target = 7.01 Measured = 6.99

TABLE 2 - Experimental Soln in G

ID	Mass(g) of Exp Soln tube Before LSA Sampling	pH of exp soln.
M7-Np1	51.8054	7.12
M7-Np2	52.1346	7.27
M7-Np3	51.8340	7.34
M7-Np4	51.8705	7.50
M7-Np5	51.9113	7.59
M7-Np6	51.9280	7.63
M7-Np7	51.7082	7.71
M7-Np8	51.8918	7.92
M7-Np9	52.1706	8.13
M7-Np10	52.2945	8.40
M7-Np11	51.8319	8.65
M7-Np12	52.5808	8.91
CAL11-Np1	52.3832	7.10
CAL11-Np2	52.3289	7.31
CAL11-Np3	52.3877	7.41
CAL11-Np4	52.3283	7.52
CAL11-Np5	52.2985	7.64
CAL11-Np6	52.0152	7.72
CAL11-Np7	52.3256	7.79
CAL11-Np8	52.1499	7.97
CAL11-Np9	52.3203	8.21
CAL11-Np10	52.3959	8.37
CAL11-Np11	52.1809	8.64
CAL11-Np12	52.5696	8.82

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Sampling of M Series 7 and CAL Series 11 Experimental Solutions for Div 01 Cation Analyses

Task initially assigned to Marla Roberts and Bradley Werling

Objective:

To characterize M Series 7 and CAL series 11 experimental solution water chemistry at the conclusion of the sorption experiments by analyses of certain cations.

Initial Entry: 711/52-53

Samples and Reagents:

Experimental solutions: M7-Np1 to 12 and CAL11-Np1 to 12 (711/73).
QA solution: cation QA std (711/49)

Equipment:

100 mL pp beakers
Filters – Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 µm pore size
30mL syringe, B-D # 309650
30mL pp bottles

Note:

The cation analyses samples were collected after the pH analyses was conducted (771/70-73)

Procedure:

The experimental solution was carefully decanted into a 100mL pp beaker. A syringe was used to collect as much sample as possible from this 100mL beaker. Next a filter was attached to the end of the syringe. The sample was dispensed into a labeled 30mL pp bottle. The label contained a Div 01 ID, initials, and date. This process was repeated for each of the experimental solutions.

Two duplicate QA samples were included in this sample set. Aliquots of cation QA std (711/49) were decanted into two labeled 30mL pp bottles.

The prefix for all Div 01 IDs was MC711 which represented the experimental solution ID. M for montmorillonite and C for consolidated alluvium. "7" for M series 7 and "11" for CAL series 11. The suffixes 1 to 12 were used for the individual M7 exp solutions. The suffixes 13 and 14 were used for the QA samples. The suffixes 15 to 26 were used for the CAL11 exp solutions. The complete legend relating the CNWRA ID with the DIV 01 IDs is on 711/75

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Legend for CNWRA and Div01 IDs for M series 7 and CAL series 11 experimental solution cation analyses.

CNWRA Sample ID	Div 01 Sample ID
M7-Np1	MC711-1
M7-Np2	MC711-2
M7-Np3	MC711-3
M7-Np4	MC711-4
M7-Np5	MC711-5
M7-Np6	MC711-6
M7-Np7	MC711-7
M7-Np8	MC711-8
M7-Np9	MC711-9
M7-Np10	MC711-10
M7-Np11	MC711-11
M7-Np12	MC711-12
MC711-13	MC711-13
MC711-14	MC711-14
CAL11-Np1	MC711-15
CAL11-Np2	MC711-16
CAL11-Np3	MC711-17
CAL11-Np4	MC711-18
CAL11-Np5	MC711-19
CAL11-Np6	MC711-20
CAL11-Np7	MC711-21
CAL11-Np8	MC711-22
CAL11-Np9	MC711-23
CAL11-Np10	MC711-24
CAL11-Np11	MC711-25
CAL11-Np12	MC711-26

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BWS

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Liquid Scintillation Analyses Results for M Series 7 and CAL Series 11 Experimental Solutions

Task initially assigned to Bradley Werling

Objective

To determine the amount of neptunium in the experimental solution at the end of the sorption experiments.

Initial Entry 711/52-53

LSA sample preparation 711/70-72

Sample Info

Blank (one sample from simulated water with calcium): w Ca 711/70
Neptunium spikes (two samples) – Spike 61A1-9 and Spike 61A1-10 (711/70)
Two experiments derived from 2 solids and 1 type of simulated water
Montmorillonite series 7 (24 samples) using simulated water with calcium
Consolidated Alluvium series 11 (24 samples) using simulated water with calcium

Results

Printout of LSA analyses following:

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Assay Definition-

Assay Description:


Assay Type: Alpha/Beta
Report Name: Np_Pa_Exp
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\20050715_1932.results
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.039
Assay File Name: C:\Packard\TriCarb\Assays\Pa_Np_Exp_AB.lsa

Count Conditions-

Nuclide: NP/PA
   Quench Indicator: SIS
   External Std Terminator (sec): n/a
   Pre-Count Delay (min): 0.00
Alpha/Beta Standards:
   Low Energy: NP/PA
Count Time (min): 240.00
Count Mode: Normal
Assay Count Cycles: 1          Repeat Sample Count: 1
#Vials/Sample: 1               Calculate % Reference: Off

Background Subtract: On - 1st Vial
Low CPM Threshold: Off
2 Sigma % Terminator: On - Any Region

In Use Discriminator: 143

Regions        LL       UL  Bkg Subtract  2Sigma % Terminator
Beta A        0.0    400.0      1st Vial                 0.00
Beta B      100.0    400.0      1st Vial                 0.00
Alpha       100.0    400.0      1st Vial                 2.00

Count Corrections-

Static Controller: On          Luminescence Correction: Off
Colored Samples: n/a           Heterogeneity Monitor: n/a
Coincidence Time (nsec): 18    Delay Before Burst (nsec): 75

Half Life-

Half Life Correction: Off
Regions    Half Life          Units      Reference Date      Reference Time
Beta A
Beta B
Alpha

IPA Block Data
Software Version IC: 2.11
Software Version EC: 1.31
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 34.27    Date Processed: 7/15/05 7:32:09 PM
14C Chi Square: 24.87   Date Processed: 7/15/05 7:32:09 PM
3H E^2/B (1-18.6 keV): 273.84  Date Processed: 7/15/05 7:32:09 PM
14C E^2/B (4-156 keV): 517.79   Date Processed: 7/15/05 7:32:09 PM
3H Efficiency (0-18.6 keV): 65.22  Date Processed: 7/15/05 7:32:09 PM
14C Efficiency (0-156 keV): 97.02  Date Processed: 7/15/05 7:32:09 PM
IPA Background Date Processed: 7/15/05 7:32:09 PM
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3H Background CPM (0-18.6 keV): 15.45 Date Processed: 7/15/05 7:32:09 PM
14C Background CPM (0-156 keV): 22.90 Date Processed: 7/15/05 7:32:09 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMa	alpha2S%	SIS	
MESSAGES									
1	240.00	19.49	2.92	3.44	6.96	0.42	20.00	171.8	w CA 711/70
B									M Series 7
Missing vial 2.									
3	85.94	29.05	5.53	11.85	7.40	115.94	2.01	281.3	>Np1<a b
4	85.09	28.89	5.58	12.13	7.32	117.11	2.01	294.0	
5	94.57	27.82	5.48	11.16	7.36	105.33	2.01	279.6	>Np2<a b
6	95.25	28.12	5.42	11.48	7.21	104.57	2.01	279.5	
7	87.46	30.25	5.33	12.49	7.10	113.92	2.01	284.5	>Np3<a b
8	86.96	30.93	5.26	12.34	7.17	114.59	2.01	278.8	
9	94.29	28.75	5.36	11.37	7.28	105.64	2.01	279.8	>Np4<a b
10	92.45	29.61	5.29	12.03	7.09	107.77	2.01	281.3	
11	90.27	28.75	5.46	11.30	7.46	110.36	2.01	275.0	>Np5<a b
12	89.30	31.21	5.16	13.06	6.83	111.58	2.01	285.0	
13	89.09	33.58	4.90	13.78	6.61	111.84	2.01	276.7	>Np6<a b
14	89.49	30.38	5.26	12.67	6.96	111.35	2.01	287.7	
15	89.19	29.22	5.42	12.04	7.20	111.75	2.01	281.6	>Np7<a b
16	88.14	31.93	5.11	13.34	6.78	113.05	2.01	285.6	
17	89.46	31.53	5.12	12.67	6.96	111.37	2.01	286.8	>Np8<a b
18	87.26	30.02	5.37	12.16	7.23	114.18	2.01	276.6	
19	93.08	28.26	5.46	11.45	7.29	107.04	2.01	282.4	>Np9<a b
20	92.55	27.82	5.53	11.81	7.17	107.64	2.01	290.0	
21	82.68	30.53	5.43	13.10	7.07	120.54	2.01	292.9	>Np10<a b
22	80.57	32.39	5.26	12.92	7.22	123.70	2.01	283.0	
23	75.39	35.30	5.09	14.57	6.91	132.25	2.01	288.7	>Np11<a b
24	76.56	36.20	4.97	15.25	6.67	130.20	2.01	289.0	
25	72.27	43.19	4.51	17.87	6.22	137.97	2.01	286.3	>Np12<a b
26	73.16	43.68	4.45	18.28	6.10	136.32	2.01	285.8	
Missing vial 27.									CAL Series 11
28	68.79	13.86	10.86	5.45	13.90	144.95	2.01	282.4	>Np1<a b
29	70.07	13.96	10.71	5.14	14.40	142.37	2.01	259.2	
30	70.34	13.72	10.84	4.70	15.34	141.75	2.01	251.1	>Np2<a b
31	68.81	14.91	10.23	5.83	13.25	144.91	2.01	267.9	
32	70.36	13.78	10.80	5.21	14.22	141.77	2.01	271.3	>Np3<a b
33	70.13	14.37	10.45	5.70	13.35	142.18	2.01	285.9	
34	70.66	14.23	10.50	5.83	13.09	141.12	2.01	290.4	>Np4<a b
35	69.39	14.74	10.29	6.31	12.47	143.71	2.01	300.9	
36	70.05	15.43	9.87	6.25	12.50	142.35	2.01	283.5	>Np5<a b
37	70.32	14.75	10.22	6.24	12.49	141.79	2.01	291.1	
38	69.05	16.61	9.36	6.26	12.57	144.43	2.01	274.8	>Np6<a b
39	69.31	16.22	9.52	6.74	11.91	143.88	2.01	283.3	
40	69.17	15.75	9.76	6.20	12.65	144.17	2.01	278.4	>Np7<a b
41	68.83	14.78	10.30	6.19	12.69	144.88	2.01	285.3	
42	69.30	15.18	10.05	6.18	12.66	143.88	2.01	278.9	>Np8<a b
43	68.20	15.98	9.71	6.73	12.01	146.25	2.01	287.8	
44	69.16	14.31	10.55	6.26	12.56	144.19	2.01	300.1	>Np9<a b
45	70.28	18.33	8.59	7.26	11.25	141.87	2.01	279.7	
46	69.01	16.17	9.56	6.64	12.06	144.53	2.01	286.6	>Np10<a b
47	69.17	17.43	9.00	6.52	12.21	144.18	2.01	258.9	
48	67.00	15.81	9.87	6.45	12.47	148.84	2.01	285.9	>Np11<a b
49	69.00	16.45	9.43	7.07	11.55	144.51	2.01	301.0	
50	142.89	13.37	8.34	5.15	10.59	69.57	2.02	267.5	>Np12<a b
51	142.54	12.95	8.58	4.80	11.19	69.75	2.02	261.6	
Missing vial 52.									

Witnessed & Understood by me, | Date | Invented by | Date | Recorded by

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7/18/05 5:16:41 PM QuantaSmart (TM) - 1.31 - Serial# 405314 Page # 3

Protocol# 15 - Pa_Np_Exp_AB.lsa User: Bertetti

53	1.12	6903.72	2.28	2266.20	3.97	8983.51	1.99	245.5	Spike 61A1-9
54	1.06	7227.68	2.29	2255.99	4.09	9512.79	1.99	238.9	Spike 61A1-10

22 JUL 05 BAW

Delivery of M series 7 and CAL series 11 samples to DIV01 for cation analyses

Initial Entry for Sorption Experiment: 711/52-53

Samples: 12 M Series 7
12 CAL Series 11
2 QAs

Sample + QA collection: 711/74-75
Sample ID legend: 711/75
QA target conc 711/49
Chain of Custody on following pages.

22 Jul 05 BAW

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Witnessed & Understood by me, | Date | Invented by | Date | Recorded by

Witnessed & Understood by me, Date

Recorded by Invented by Date

From Page No.

Project No.
Book No.
TITLE 22 JUL 05 cont BAW

Shipper Name/Address: BRADLEY WERLING DIV 20 BLD 57

Client:

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID:

Site/Zone ID:

Requested Turnaround:
☐ 2 Weeks
☐ 3 Weeks
☒ Other: 4 weeks

SwRI Contact: Mike Dammann

Analyses Requested: Al, Si, Mn, Mg, La, K, Na, Sr by ICP

REMARKS
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) none

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Al, Si, Mn, Mg, La, K, Na, Sr by ICP	Remarks
MC711-1	7-15-05		W		1	X	Nuclear Safety
MC711-2						X	Related - use
MC711-3						X	appropriate QA
MC711-4						X	procedures
MC711-5						X	
MC711-6						X	POC - Brad Werling
MC711-7						X	ext 6565
MC711-8						X	fax 5184
MC711-9						X	
MC711-10						X	

Matrix Types:
A – Air
B – Biota
D - Dust
E – Emission/Stack
L - Liquid
P – Product
Sd - Solid
S – Soil
SED – Sediment
T - Tissue
W - Water
WP - Wipe

Sample Types:
D - Duplicate
ER – Equipment Rinsate
ES – Environmental Sample
FB – Field Blank
FD – Field Duplicate
MS – Matrix Spike
MSD – Matrix Spike Dup
TB – Trip Blank

Temp: 22.0°C Therm #: 027

Comments: RADIOACTIVE - Np.237 at 8.5×10⁻⁷M or 1.4×10⁻⁴ µCi/mL
SN 711/74

Relinquished by (Print/Signature)	Date	Time
Maria Roberts Maria Roberts	7-22-05	11:25

Received by (Print/Signature)	Date	Time

Relinquished by (Print/Signature)	Date	Time

Received by (Print/Signature)	Date	Time

Relinquished by (Print/Signature)	Date	Time

SwRI Project#: 20.06002.01.242

Received by SwRI Lab: (Signature)

Date 7/22/05 Time 1125

Samples Disposed: Date Time

Samples Disposed by:

Div 01 COC Form 01-01-001, Rev 8/02

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No.

Witnessed & Understood by me, Date

Recorded by Invented by Date

To Page No.

From Page

TITLE 22 JUL 05 cont BAW

Project No.
Book No.

Shipper Name/Address: BRADLEY WERLING DIV 20 BLD 57

Client:

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID:

Site/Zone ID:

Requested Turnaround:
☐ 2 Weeks
☐ 3 Weeks
☒ Other: 4 weeks

SwRI Contact: Mike Dammann

Analyses Requested: Al, Si, Mn, Mg, La, K, Na, Sr by ICP

REMARKS
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) none

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Al, Si, Mn, Mg, La, K, Na, Sr by ICP	Remarks
MC711-11	7-15-05		W		1	X	Nuclear Safety
MC711-12						X	Related - use
MC711-13						X	appropriate QA
MC711-14						X	procedures
MC711-15						X	
MC711-16						X	POC - Brad Werling
MC711-17						X	ext 6565
MC711-18						X	fax 5184
MC711-19						X	
MC711-20						X	

Matrix Types:
A – Air
B – Biota
D - Dust
E – Emission/Stack
L - Liquid
P – Product
Sd - Solid
S – Soil
SED – Sediment
T - Tissue
W - Water
WP - Wipe

Sample Types:
D - Duplicate
ER – Equipment Rinsate
ES – Environmental Sample
FB – Field Blank
FD – Field Duplicate
MS – Matrix Spike
MSD – Matrix Spike Dup
TB – Trip Blank

Temp: 22.0°C Therm #: 027

Comments: RADIOACTIVE - Np.237 at 8.5×10⁻⁷M or 1.4×10⁻⁴ µCi/mL
SN 711/74

Relinquished by (Print/Signature)	Date	Time
Maria Roberts Maria Roberts	7-22-05	11:22

Received by (Print/Signature)	Date	Time

Relinquished by (Print/Signature)	Date	Time

Received by (Print/Signature)	Date	Time

Relinquished by (Print/Signature)	Date	Time

SwRI Project#: 20.06002.01.242

Received by SwRI Lab: (Signature)

Date 7/22/05 Time 1125

Samples Disposed: Date Time

Samples Disposed by:

Div 01 COC Form 01-01-001, Rev 8/02

Page 2 of 3

Project No.
Book No.
TITLE 22 JULY 05 CONT BAW

SAMPLE LIST/CHAIN OF CUSTODY
Southwest Research Institute®
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Client Purchase Order/Other ID
Site/Zone ID
Shipper Name/Address: BRADLEY WERLING, DIV 20, BLD 57
Client

Requested Turnaround: ☐ 2 Weeks ☐ 3 Weeks ☒ Other: 4 weeks
SwRI Contact: Mike Dammann

Preservation: a = HCl to pH <2; b = HNO_3 to pH <2; c = H_2SO_4 to pH <2; d = NaOH to pH >12; e = Cool (4°C±2°C); f = Other (specify) None

REMARKS: Nuclear Safety Related - use appropriate QA procedures; RL Brad Werling ext 6565 fax 5184

Analyses Requested: Al, Si, Mn, Mg, Ca, K, Na, Sr by ICP

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
MC711-21	7-15-05		W		1
MC711-22					
MC711-23					
MC711-24					
MC711-25					
MC711-26					

Matrix Types: A – Air; B – Biota; D - Dust; E – Emission/Stack; L - Liquid; P – Product; Sd - Solid; S – Soil; SED – Sediment; T - Tissue; W - Water; WP - Wipe

Sample Types: D - Duplicate; ER – Equipment Rinsate; ES – Environmental Sample; FB – Field Blank; FD – Field Duplicate; MS – Matrix Spike; MSD – Matrix Spike Dup; TB – Trip Blank

Temp: 7 7 0°C
Therm #: 97
Comments: RADIOACTIVE - ND-2377 85+45 M or 1.4×10⁻⁴ µCi/ml SN 711/4

Relinquished by (Print/Signature)	Date	Time
Martin Rubio	7-22-05	11:22
Received by (Print/Signature)	Date	Time
Roberto Tafoya Rubio		
Relinquished by (Print/Signature)	Date	Time
Received by (Print/Signature)	Date	Time
Relinquished by (Print/Signature)	Date	Time

SwRI Project#: 20.06.007.01.242
Received by SwRI Lab: (Signature) Date 7/22/05 Time 1125
Samples Disposed: Date Time
Samples Disposed by:

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Div 01 Cation Results for CAL series 7+8 Experimental Solutions at conclusion of experiment

Initial Entry for Sorption Experiment: 711/9-10
Samples: 12 CAL series 7
12 CAL series 8
2 QAs
Sample + QA collection 711/50
Sample ID legend 711/51
QA target conc 711/49
Delivery + Chain of Custody to Div 01: 711/60-63.

QA Results for Div 01 Cation Analyses of CAL series 7 and 8 Experimental Solutions

For Sample CAL78-13

Analyte	Target Conc (mg/L)	Measured Conc (mg/L)	Percent Difference
Calcium	10	9.99	-0.10
Magnesium	10	9.50	-5.00
Potassium	10	8.46	-15.40
Sodium	10	11.20	12.00
Iron	10	not analyzed	na

For Sample CAL78-14

Analyte	Target Conc (mg/L)	Measured Conc (mg/L)	Percent Difference
Calcium	10	10.1	1.00
Magnesium	10	9.6	-3.90
Potassium	10	10.2	2.00
Sodium	10	9.0	-10.50
Iron	10	not analyzed	na

Source 733/3

8-30-05 BAW

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Recorded by

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-1

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264792
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	10.3	0.5
Magnesium	1.27	0.5
Manganese	0.0877	0.05
Potassium	4.81	2.5
Silicon	15.6	0.5
Sodium	77.4	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-2

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264803
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	9.18	0.5
Magnesium	0.976	0.5
Manganese	<0.0500	0.0500
Potassium	5.40	2.5
Silicon	14.6	0.5
Sodium	77.0	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-3

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264811
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	9.00	0.5
Magnesium	1.02	0.5
Manganese	<0.0500	0.0500
Potassium	4.65	2.5
Silicon	14.2	0.5
Sodium	77.8	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-4

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264812
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	7.91	0.5
Magnesium	0.910	0.5
Manganese	<0.0500	0.0500
Potassium	4.89	2.5
Silicon	13.2	0.5
Sodium	71.2	2.5
Strontium	<0.0500	0.0500

Witnessed 8

Recorded by

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-5

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264813
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	8.66	0.5
Magnesium	0.969	0.5
Manganese	<0.0500	0.0500
Potassium	4.57	2.5
Silicon	14.2	0.5
Sodium	76.4	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-6

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264814
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	8.36	0.5
Magnesium	0.951	0.5
Manganese	<0.0500	0.0500
Potassium	4.84	2.5
Silicon	13.9	0.5
Sodium	77.0	2.5
Strontium	<0.0500	0.0500

Recorded by

From Page No

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-7

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264815
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	8.38	0.5
Magnesium	0.979	0.5
Manganese	<0.0500	0.0500
Potassium	4.51	2.5
Silicon	13.9	0.5
Sodium	76.1	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-8

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264816
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	8.21	0.5
Magnesium	0.881	0.5
Manganese	<0.0500	0.0500
Potassium	4.20	2.5
Silicon	13.9	0.5
Sodium	77.0	2.5
Strontium	<0.0500	0.0500

Witnessed 8

Recorded by

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-9

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264817
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	7.88	0.5
Magnesium	0.952	0.5
Manganese	<0.0500	0.0500
Potassium	4.10	2.5
Silicon	13.7	0.5
Sodium	75.4	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-10

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264793
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	7.77	0.5
Magnesium	0.856	0.5
Manganese	<0.0500	0.0500
Potassium	3.65	2.5
Silicon	13.6	0.5
Sodium	76.5	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-11

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264794
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	7.06	0.5
Magnesium	0.793	0.5
Manganese	<0.0500	0.0500
Potassium	4.03	2.5
Silicon	13.9	0.5
Sodium	75.4	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-12

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264795
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	2.85	0.5
Magnesium	<0.500	0.5
Manganese	<0.0500	0.0500
Potassium	2.68	2.5
Silicon	14.7	0.5
Sodium	108	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-15

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264798
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.62	0.5
Magnesium	0.758	0.5
Manganese	<0.0500	0.0500
Potassium	4.01	2.5
Silicon	14.7	0.5
Sodium	93.9	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-16

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264799
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.50	0.5
Magnesium	0.815	0.5
Manganese	<0.0500	0.0500
Potassium	4.49	2.5
Silicon	14.7	0.5
Sodium	95.6	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-17

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264800
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.02	0.5
Magnesium	0.759	0.5
Manganese	<0.0500	0.0500
Potassium	3.35	2.5
Silicon	14.7	0.5
Sodium	95.3	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-18

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264801
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.25	0.5
Magnesium	0.695	0.5
Manganese	<0.0500	0.0500
Potassium	4.50	2.5
Silicon	14.4	0.5
Sodium	94.7	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-19

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264802
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.92	0.5
Magnesium	0.613	0.5
Manganese	<0.0500	0.0500
Potassium	4.00	2.5
Silicon	14.5	0.5
Sodium	92.9	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-20

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264804
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.12	0.5
Magnesium	0.649	0.5
Manganese	<0.0500	0.0500
Potassium	5.50	2.5
Silicon	14.5	0.5
Sodium	93.5	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-21

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264805
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	4.02	0.5
Magnesium	0.681	0.5
Manganese	<0.0500	0.0500
Potassium	3.04	2.5
Silicon	14.6	0.5
Sodium	93.6	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAL78-22

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264806
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.98	0.5
Magnesium	0.658	0.5
Manganese	<0.0500	0.0500
Potassium	4.92	2.5
Silicon	14.4	0.5
Sodium	93.3	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAL78-23

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264807
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.86	0.5
Magnesium	0.687	0.5
Manganese	<0.0500	0.0500
Potassium	3.80	2.5
Silicon	14.7	0.5
Sodium	93.3	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAL78-24

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264808
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.63	0.5
Magnesium	0.618	0.5
Manganese	<0.0500	0.0500
Potassium	4.31	2.5
Silicon	14.7	0.5
Sodium	94.5	2.5
Strontium	<0.0500	0.0500

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SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAL78-25

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264809
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.86	0.5
Magnesium	0.669	0.5
Manganese	<0.0500	0.0500
Potassium	4.04	2.5
Silicon	14.6	0.5
Sodium	94.7	2.5
Strontium	<0.0500	0.0500

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAL78-26

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Water
Lab System ID: 264810
Method: ICP SW846 6010B

Client: Division 20
Date Received: 07/11/05
Project No.: 06002.01.242
SRR: 27780
Task Order: 050711-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.500	0.500
Calcium	3.13	0.5
Magnesium	0.591	0.5
Manganese	<0.0500	0.0500
Potassium	3.81	2.5
Silicon	15.2	0.5
Sodium	128	2.5
Strontium	<0.0500	0.0500

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END OF SCIENTIFIC NOTEBOOK 711
ENTRIES CONTINUED IN SN 733

I have reviewed this scientific notebook and find it in agreement with QAP-001. There is sufficient information regarding methods used for conducting tests, acquiring and analyzing data so that another qualified individual could repeat the activity.

R. C Peary
10/5/2005

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Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	