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Investigator

Investigator

Marla Roberts

Sandra Watson

F. Paul Bertlett

Bradley Worling

Signature

Marla Roberts

Sandra Watson

F. Paul Bertlett

Bradley Worling

Initials

MR

SW

FPB

BAW

Initial Entry for Scientific Notebook 811

This notebook contains work for Radionuclide Transport (RT) in both the saturated zone and unsaturated zone (ISIs 20.06002.02.242 and 20.06002.01.232 respectively). 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 the sorption experiment is to determine the sorption capability (K_d 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 (GED) and pH analyses (GED) of reference solutions
- Preparation of experimental solutions
- Liquid Scintillation Analyses (GED) 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 800. That notebook concludes with pH experiments on the sonic core composite samples from wells NC-EWDP-19PB and NC-EWDP-22PC. SN 811 will start with a new series of experiments; 19CALW2, 19CAL2, 22CALW2 and 22CAL2.

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Initial Entry – 19 CAL Series 2 and 22 CAL Series 2 with and without Calcite Sorption Experiments – Np-237 on Consolidated Alluvium with 19PB Simulated Waters with Calcium over a pH range

Personnel initially assigned to task: Marla Roberts

Objective: To investigate Np sorption on a NC-EWDP-19PB 0305 Consolidated Alluvium and NC-EWDP-22PC 1005 Consolidated Alluvium (with and without calcite) 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.

Background: This is series 2 of the 19CAL and 22CAL with and without calcite sorption experiment. 19CAL and 22CAL Series 1 with and without calcite, 800/14-15.

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-3 Simulated Groundwater with Ca – 800/20
- NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
- NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
- NC-EWDP-22PC-1005-Sonic Core Composite – 742/8
- NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Reagents

- Neptunium Spike # 61A-2 (369/103) - 60ppm Np-237 at pH of 5.9
- 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 HNO_3 (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 3I)
- New Brunswick Scientific Gyrotory shaker model G33 (sn 290127990)
- Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
- Mettler Toledo XP205DR (sn 1126461033)
- Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
- Stir Plate
- Centrifuge Tube Holder (Aluminum)

Supplies:

- Pipettes and tips
- Repipettor for transfer of scintillation cocktail
- Polycarbonate centrifuge tubes with caps (50 ml capacity)
- Stirs bars
- Kimwipes

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- 7 ml scintillation vials
- Weighing paper/boats
- 10 mL microbeakers
- Plasticware and glassware as needed
- Squirt bottle for water

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 gyrotator
- For the experiment, during regular business hours the tubes are loosely capped and tightly capped at night.
- Analyze aliquots of experimental solution for pH and neptunium concentration (LSA)

Summary of Tables:

Table 1-Initial mass of tube and mass of tube & solid
 Table 2-Initial mass of tube, solid & simulated water
 Table 3-Mass after pH adjustment, Mass after pH equilibration (overnight)
 Table 4-Mass after Np spike added
 Table 5-Mass of LSA vial & acid, mass of vial, acid and sample
 Table 6-Mass of exp. soln tube before LSA sampling, pH of exp. soln.

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Equilibration of Simulated Waters used in 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions

Personnel initially assigned to task: Marla Roberts

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

Initial Entry: 811/2-3

Procedure

Aeration of simulated water conducted since 19 June 2006— see 800/20

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Addition of Solids to 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experiments

Personnel initially assigned to task: Marla Roberts

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

Initial Entry: 811/2-3

Sample info NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
NC-EWDP-22PC-1005-Sonic Core Composite – 742/8
NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Procedure

Four sets of twelve polycarbonate centrifuge tubes were labeled as follows: 19CALW2- 1 to 12, 19CAL2-1 to 12, 22CALW2-1 to 12 and 22CAL2-1 to 12. The mass of each tube (with cap) was measured and recorded on 811/6-7. An individual experimental tube was uncapped and a funnel made from weighing paper was inserted in the top. Prior to aliquot removal the jar containing the solid was gently rolled to ensure an unbiased aliquot. Approximately 0.2 g of solid was transferred to a piece of tared weighing paper on the balance. The solid was transferred into the appropriate experimental tube 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 811/6-7.

The centrifuge tubes labeled with a "CALW2" indicate the solid contains calcite and the aliquot was removed from a sampling containing calcite (NC-EWDP-19PB-0305-Sonic Core Composite and NC-EWDP-22PC-1005-Sonic Core Composite). The centrifuge tubes labeled with a "CAL2" indicate the solid does not contain calcite and was removed from a treated sample (NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite and NC-EWDP-22PC-1005-Sonic Core Composite w/o Calcite). See 742/25 for further information about calcite removal. The "2" was used to represent the is the second series of 19CAL and 22CAL with and without calcite experiments.

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Table 1

Sample ID	Mass (g) Empty Tube	Mass (g) Tube & Solid
19CALW2-1	22.28559	22.48732
19CALW2-2	22.55031	22.75372
19CALW2-3	22.09889	22.30242
19CALW2-4	22.33535	22.53913
19CALW2-5	22.24456	22.44678
19CALW2-6	22.33066	22.53358
19CALW2-7	22.50756	22.71007
19CALW2-8	22.30994	22.51334
19CALW2-9	22.50856	22.71152
19CALW2-10	22.32980	22.53091
19CALW2-11	22.52696	22.73031
19CALW2-12	22.17489	22.37729
19CAL2-1	22.28125	22.48565
19CAL2-2	22.19969	22.40308
19CAL2-3	22.19142	22.39356
19CAL2-4	22.31078	22.51592
19CAL2-5	22.51929	22.72179
19CAL2-6	22.25221	22.45750
19CAL2-7	22.27964	22.48219
19CAL2-8	22.10954	22.31172
19CAL2-9	22.11465	22.31898
19CAL2-10	22.18565	22.39021
19CAL2-11	22.27355	22.47632
19CAL2-12	22.27958	22.48519
22CALW2-1	22.51152	22.71357
22CALW2-2	22.30606	22.50930
22CALW2-3	22.28852	22.49029
22CALW2-4	22.30925	22.51580
22CALW2-5	22.04948	22.25035
22CALW2-6	22.27659	22.48078
22CALW2-7	22.04657	22.25063
22CALW2-8	22.31122	22.51501
22CALW2-9	22.26157	22.46476
22CALW2-10	22.16082	22.36485
22CALW2-11	22.22219	22.42425
22CALW2-12	22.07648	22.28379
22CAL2-1	22.24446	22.44845
22CAL2-2	22.27237	22.47230
22CAL2-3	22.51977	22.72186
22CAL2-4	22.16200	22.36688
22CAL2-5	22.31763	22.52135
22CAL2-6	22.57426	22.77556
22CAL2-7	22.27677	22.47770
22CAL2-8	22.55430	22.75530

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Table 1 cont.

Sample ID	Mass (g) Empty Tube	Mass (g) Tube & Solid
22CAL2-9	22.52781	22.72827
22CAL2-10	22.30684	22.51034
22CAL2-11	22.15004	22.35213
22CAL2-12	22.05029	22.25621

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Addition of Simulated Water Solution to 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experiment

Personnel initially assigned to task: Marla Roberts and Sandra Watson

Objective: To add simulated water to centrifuge tubes as preparation for sorption experiment.

Initial Entry: 811/2-3

Sample Info: 19PB-3 Simulated Groundwater with Calcium 800/20000000^{17 July 2006 MR}Procedure

About 30mL (10mL pipet) of the simulated water was added to the experimental pc tubes (811/6-7). The mass was recorded on 811/9-10.

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Table 2

Sample ID	Mass (g) Tube, Solid & Solution
19CALW2-1	53.14862
19CALW2-2	53.31499
19CALW2-3	52.80606
19CALW2-4	53.36139
19CALW2-5	52.98395
19CALW2-6	53.07805
19CALW2-7	53.34702
19CALW2-8	53.12518
19CALW2-9	53.20691
19CALW2-10	53.14051
19CALW2-11	53.26161
19CALW2-12	52.99346
19CAL2-1	53.08818
19CAL2-2	52.91097
19CAL2-3	52.87004
19CAL2-4	53.02083
19CAL2-5	53.16935
19CAL2-6	52.94294
19CAL2-7	52.84070
19CAL2-8	52.70729
19CAL2-9	52.81755
19CAL2-10	52.87822
19CAL2-11	52.94330
19CAL2-12	52.94723
22CALW2-1	53.16850
22CALW2-2	52.97722
22CALW2-3	52.96674
22CALW2-4	52.98430
22CALW2-5	52.09428
22CALW2-6	52.94030
22CALW2-7	52.52033
22CALW2-8	52.96761
22CALW2-9	52.96338
22CALW2-10	52.89330
22CALW2-11	52.91826
22CALW2-12	52.76671
22CAL2-1	53.09264
22CAL2-2	52.98878
22CAL2-3	53.25174
22CAL2-4	52.86322
22CAL2-5	52.93054
22CAL2-6	52.87524
22CAL2-7	52.84242
22CAL2-8	53.18965

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Table 2 cont.

22CAL2-9	53.14543
22CAL2-10	52.94338
22CAL2-11	52.80118
22CAL2-12	52.71517

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Addition of pH Adjusting solution to 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions

Personnel initially assigned to task: Marla Roberts

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

Initial Entry: 811/2-3

Background: pH experiment (800/79-81) on NC-EWDP-19PB-0305-Sonic Core Composite (696/64-66), NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite (742/25) NC-EWDP-22PC-1005-Sonic Core Composite (742/8) and NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite (742/25) and analyses of the results from the 22CAL1 Series resulted in an adjustment to the ph scheme used in 22CAL Series 2 (without calcite). The adjustment was for all experimental tubes labeled 22CAL2 1-12. To avoid having results cluster between pH 7 and 8, more acid was added to provide a range of pH values. See below for details.

Procedure: Used adjustable eppendorfs 100-1000 μ L and 10/20/25 μ L to add 0.4 M HNO₃ (696/195) for pH adjustment according to the schemes listed below. The tubes were weighed and the masses recorded 811/12-13. The solutions were then votexed for ~30 seconds and placed loosely capped on an activated gyator overnight. The tubes were re-weighed the next day and the mass recorded (*Mass After Equilibration*) 811/12-13.

pH SCHEME FOR:

19CALW2 1-12,
19CAL2 1-12,
22CALW2 1-12

ID	Vol (μ L) of 0.4 M HNO ₃	Vol (μ L) of 0.1 M NaOH
1	210	na
2	205	na
3	200	na
4	195	na
5	190	na
6	185	na
7	180	na
8	158	na
9	140	na
10	105	na
11	10	na
12	na	517

pH SCHEME FOR:

22CAL2 1-12

ID	Vol (μ L) of 0.4 M HNO ₃	Vol (μ L) of 0.1 M NaOH
1	195	na
2	190	na
3	185	na
4	180	na
5	175	na
6	170	na
7	165	na
8	143	na
9	130	na
10	105	na
11	10	na
12	na	517

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Table 3

Sample ID	Mass (g) After pH Adjustment	Mass (g) After Equilibration
19CALW2-1	53.36267	53.33430
19CALW2-2	53.52222	53.49969
19CALW2-3	53.00874	52.98925
19CALW2-4	53.56061	53.51990
19CALW2-5	53.17750	53.13530
19CALW2-6	53.26383	53.23903
19CALW2-7	53.52795	53.48934
19CALW2-8	53.28384	53.13926
19CALW2-9	53.34988	53.32995
19CALW2-10	53.24726	53.23090
19CALW2-11	53.27150	53.25111
19CALW2-12	53.51288	53.49902
19CAL2-1	53.29959	53.28442
19CAL2-2	53.11789	53.10143
19CAL2-3	53.07130	53.05744
19CAL2-4	53.21860	53.20036
19CAL2-5	53.36188	53.34272
19CAL2-6	53.12911	53.10617
19CAL2-7	53.02102	52.98132
19CAL2-8	52.86571	52.84123
19CAL2-9	52.96201	52.94437
19CAL2-10	52.98368	52.96993
19CAL2-11	52.95324	52.82746
19CAL2-12	53.46292	53.44203
22CALW2-1	53.38019	53.33962
22CALW2-2	53.18474	53.16406
22CALW2-3	53.16977	53.13003
22CALW2-4	53.18414	53.16682
22CALW2-5	52.28842	52.25263
22CALW2-6	53.12698	53.09016
22CALW2-7	52.70170	52.67766
22CALW2-8	53.12676	53.10819
22CALW2-9	53.10735	53.05513
22CALW2-10	52.99931	52.95918
22CALW2-11	52.92889	52.89961
22CALW2-12	53.28233	53.23949
22CAL2-1	53.29224	53.26747
22CAL2-2	53.18341	53.16482
22CAL2-3	53.43834	53.41896
22CAL2-4	53.04521	53.03188
22CAL2-5	53.10616	53.08793
22CAL2-6	53.04663	53.00949
22CAL2-7	53.00861	52.90856

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Table 3 cont.

22CAL2-8	53.33671	53.31815
22CAL2-9	53.27644	53.25919
22CAL2-10	53.04893	53.03084
22CAL2-11	52.81180	52.69551
22CAL2-12	53.23134	53.21527

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Addition of Neptunium Spike to 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions

Personnel initially assigned to task: Marla Roberts

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

Initial Entry: 811/2-3

Reagent: Np Spike # 61A-2 (~~369/103~~) ^{369/103} 18 July 06 MR

Procedure

Added 100 μ L (100 μ L eppendorf pipet) to each experimental solution and recorded the mass of the experimental solution on 811/15-16.

In process entry:

In This experiment a new spike was used. In 19CAL + 22CAL Series 1 the spike used was 61A1, however for Series 2 the spike used is 61A2.

For further explanation of reasons for creating new spike, see 800/10.

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Table 4

Sample ID	Mass (g) After Np Added
19CALW2-1	53.43293
19CALW2-2	53.59764
19CALW2-3	53.08707
19CALW2-4	53.61798
19CALW2-5	53.23434
19CALW2-6	53.33710
19CALW2-7	53.58787
19CALW2-8	53.23814
19CALW2-9	53.42812
19CALW2-10	53.32925
19CALW2-11	53.34961
19CALW2-12	53.59706
19CAL2-1	53.38308
19CAL2-2	53.19977
19CAL2-3	53.15608
19CAL2-4	53.29864
19CAL2-5	53.44136
19CAL2-6	53.20489
19CAL2-7	53.08007
19CAL2-8	52.93974
19CAL2-9	53.04291
19CAL2-10	53.06809
19CAL2-11	52.92581
19CAL2-12	53.52041
22CALW2-1	53.43702
22CALW2-2	53.26141
22CALW2-3	53.22776
22CALW2-4	53.26376
22CALW2-5	52.35054
22CALW2-6	53.18771
22CALW2-7	52.77426
22CALW2-8	53.20516
22CALW2-9	53.15282
22CALW2-10	53.05701
22CALW2-11	52.99721
22CALW2-12	53.33762
22CAL2-1	53.36539
22CAL2-2	53.26252
22CAL2-3	53.57637
22CAL2-4	53.12943
22CAL2-5	53.18615
22CAL2-6	53.10756
22CAL2-7	53.06750
22CAL2-8	53.41613

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Table 4 cont.

22CAL2-9	53.35695
22CAL2-10	53.12866
22CAL2-11	52.79413
22CAL2-12	53.31339

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Running of 19CAL + 22CAL Series 2
w/ + w/o calcite sorption experiment

initial entry: 811/2-3

^{18 July 2006 MR}
All exp. solns were vortexed for
~30 seconds. All exp. soln (811/15-16)
were loosely capped and placed on
activated gyrator. (~100rpm)

At 4:30pm all exp. soln. tightly
capped and placed in activated gyrator.

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Running of 19CAL + 22CAL Series 2 w/ +
w/o calcite sorption experiment

initial entry 811/2-3

Time: 9:15am

All exp solns (811/15-16) were

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vortexed for ~10 seconds. All exp.
soln. loosely capped and placed on
activated gyrator.

Time: 4:45pm

All exp. solns (811/15-16) tightly
capped.

20 July 2006 MR

Running of 19CAL + 22CAL Series 2
w/ + w/o calcite sorption experiment

initial entry: 811/2-3

Time: 8:15am

all exp. solns. (811/15-16) were
vortexed for ~10 seconds. All exp.
solns. loosely capped and placed
on activated gyrator.

Time: 4:30pm

All exp. solns (811/15-16) tightly
capped.

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Running of 19CAL + 22CAL Series 2
w/ & w/o calcite sorption experiment

initial entry: 8/1/2-3

Time: 8:30am

All exp. solns (8/1/15-16) vortexed for
~ 30 seconds. All exp. solns. were
loosely capped and placed on
activated gyrator. (~100 rpm).

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Running of 19CAL + 22 CAL Series 2
w/ & w/o calcite sorption experiment

initial entry: 8/1/2-3

time: 10:00am

All exp solns. (8/1/15-16) vortexed for
~ 30 seconds. All exp. solns. were
loosely capped and placed on activated
gyrator. (~100 rpm)

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SW

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P2 2/26/06

Stopped gyratory shaker and ~~replaced~~ tightened
caps of all exp solns for the 19 and 22 cal
series 2 solutions.

I will weigh each solution, followed by
centrifugation and sampling (for measurement
of M_p by LSA), and then measure the pH
of each solution. The general procedure is
found on pp. 20-21 of this notebook. Data are
recorded on the following pages.

No particular problems or obvious contamination
of the tubes (exp. solns) is noted prior to
processing.

1 AUGUST 2006

SW

26 Jul 2006 PB

Liquid Scintillation and pH Analyses of 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions- Np-237 in one type of simulated groundwater (with Ca) over a pH range

Task initially assigned to:

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

Initial Entry: 811/2-3

Sample Info: Four types of experiments were run. Experiments based on consolidated alluvium substrate with and without calcite from NC-EWDP-19PB and NC-EWDP-22PC using 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. Due to lack of space on the LSA caps, the sample IDs were changed. See 811/22 for legend of sample IDs. Experimental solutions were analyzed in duplicate and labeled to identify the sorption solid phase and series, the experimental solution number, and the a/b duplicate designator (example - 19CW2-1a). One simulated water blank solution was analyzed and labeled as, Blank wCa with scientific notebook page reference. The neptunium spike was analyzed in duplicate and labeled Spike 61A2-1 and 61A2-2.

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.250 mL (250µL eppendorf pipet) of 0.02 M HNO₃ added. The mass of all of the LSA vials with acid was recorded (See Table 5).

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to LSA aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (See Table 6). The tubes containing the experimental solutions were placed in the tared aluminum centrifuge holder and mass recorded.

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 5).

Next, 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.

Temp: 20.4 Set Pts.: 4.01 - pH 4
7.02 - pH 7 Slope: 98.0 %
10.06 - pH 10

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Stir bars were placed in each of the exp. tubes. The pH probe was inserted directly into the the tube. A stir plate was used when measuring the pH of the experimental solutions. The pH was recorded (Table 6). The pH meter was challenged with one of the calibration standards after all of the sample pHs were measured. The challenge solution type and measurement was recorded.

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.

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pH challenge: target - 7.00 7.02
measured - 7.00 PB 7/26/06

Balance: Mettler XP205 DR SN 1126461033
calibrated - due 6/05/07

no check mass available (in calibration)

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Table 5

Sample ID	LSA ID	Sample ID	LSA ID	Sample ID
Blank	Blank wCa	22CALW2-1	22CW2 1a	Spike 61A2-1
19CALW2-1	19CW2 1a		22CW2 1b	Spike 61A2-2
	19CW2 1b	22CALW2-2	22CW2 2a	
19CALW2-2	19CW2 2a		22CW2 2b	
	19CW2 2b	22CALW2-3	22CW2 3a	
19CALW2-3	19CW2 3a		22CW2 3b	
	19CW2 3b	22CALW2-4	22CW2 4a	
19CALW2-4	19CW2 4a		22CW2 4b	
	19CW2 4b	22CALW2-5	22CW2 5a	
19CALW2-5	19CW2 5a		22CW2 5b	
	19CW2 5b	22CALW2-6	22CW2 6a	
19CALW2-6	19CW2 6a		22CW2 6b	
	19CW2 6b	22CALW2-7	22CW2 7a	
19CALW2-7	19CW2 7a		22CW2 7b	
	19CW2 7b	22CALW2-8	22CW2 8a	
19CALW2-8	19CW2 8a		22CW2 8b	
	19CW2 8b	22CALW2-9	22CW2 9a	
19CALW2-9	19CW2 9a		22CW2 9b	
	19CW2 9b	22CALW2-10	22CW2 10a	
19CALW2-10	19CW2 10a		22CW2 10b	
	19CW2 10b	22CALW2-11	22CW2 11a	
19CALW2-11	19CW2 11a		22CW2 11b	
	19CW2 11b	22CALW2-12	22CW2 12a	
19CALW2-12	19CW2 12a		22CW2 12b	
	19CW2 12b	22CAL2-1	22C2 1a	
19CAL2-1	19C2 1a		22C2 1b	
	19C2 1b	22CAL2-2	22C2 2a	
19CAL2-2	19C2 2a		22C2 2b	
	19C2 2b	22CAL2-3	22C2 3a	
19CAL2-3	19C2 3a		22C2 3b	
	19C2 3b	22CAL2-4	22C2 4a	
19CAL2-4	19C2 4a		22C2 4b	
	19C2 4b	22CAL2-5	22C2 5a	
19CAL2-5	19C2 5a		22C2 5b	
	19C2 5b	22CAL2-6	22C2 6a	
19CAL2-6	19C2 6a		22C2 6b	
	19C2 6b	22CAL2-7	22C2 7a	
19CAL2-7	19C2 7a		22C2 7b	
	19C2 7b	22CAL2-8	22C2 8a	
19CAL2-8	19C2 8a		22C2 8b	
	19C2 8b	22CAL2-9	22C2 9a	
19CAL2-9	19C2 9a		22C2 9b	
	19C2 9b	22CAL2-10	22C2 10a	
19CAL2-10	19C2 10a		22C2 10b	
	19C2 10b	22CAL2-11	22C2 11a	
19CAL2-11	19C2 11a		22C2 11b	
	19C2 11b	22CAL2-12	22C2 12a	
19CAL2-12	19C2 12a		22C2 12b	
	19C2 12b			

Sample ID	Mass (g) of LSA vial & acid	Mass (g) of LSA vial, acid & sample
Blank wCa	7.77486	8.26338
19CW2 1a	7.65462	8.15103
19CW2 1b	7.65254	8.15213
19CW2 2a	7.64232	8.13844
19CW2 2b	7.62748	8.12758
19CW2 3a	7.60594	8.10093
19CW2 3b	7.67776	8.17845
19CW2 4a	7.58947	8.08590
19CW2 4b	7.65664	8.15750
19CW2 5a	7.67118	8.16704
19CW2 5b	7.63149	7/26/06 8.13098 8.13109
19CW2 6a	7.66905	8.16374
19CW2 6b	7.69744	8.19665
19CW2 7a	7.79132	8.28880
19CW2 7b	7.64738	8.14844
19CW2 8a	7.72807	8.22461
19CW2 8b	7.67732	8.17659
19CW2 9a	7.66478	8.15952
19CW2 9b	7.66109	8.15901
19CW2 10a	7.60713	8.09996
19CW2 10b	7.67324	8.17107
19CW2 11a	7.68281	8.17506
19CW2 11b	7.61472	8.11257
19CW2 12a	7.61631	8.10983
19CW2 12b	7.70592	8.20341
19C2 1a	7.60570	8.10050
19C2 1b	7.72619	8.22646
19C2 2a	7.67264	8.16672
19C2 2b	7.59982	8.09964
19C2 3a	7.63121	8.12563
19C2 3b	7.66071	8.16043
19C2 4a	7.61578	8.11222
19C2 4b	7.63769	8.13723
19C2 5a	7.63878	8.13450
19C2 5b	7.67639	8.17653
19C2 6a	7.64554	8.13996
19C2 6b	7.67190	8.17178
19C2 7a	7.65496	8.15012
19C2 7b	7.63638	8.13582
19C2 8a	7.66732	8.16350
19C2 8b	7.62858	8.12866
19C2 9a	7.63518	8.12976
19C2 9b	7.61104	8.10988
19C2 10a	7.66321	8.15993
19C2 10b	7.67340	8.17227
19C2 11a	7.70294	8.19793
19C2 11b	7.67564	8.17413
19C2 12a	7.67745	8.17454
19C2 12b	7.62987	8.12840

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Table 5: cont.

22CW2 1a	7.67601	8.16988
22CW2 1b	7.68192	8.18088
22CW2 2a	7.69117	8.18541
22CW2 2b	7.63435	8.13349
22CW2 3a	7.77844	8.27374
22CW2 3b	7.71803	8.21733
22CW2 4a	7.71097	8.20468
22CW2 4b	7.74759	8.24674
22CW2 5a	7.64519	8.14021
22CW2 5b	7.68319	8.18223
22CW2 6a	7.65278	8.14802
22CW2 6b	7.65847	8.15816
22CW2 7a	7.65199	8.14763
22CW2 7b	7.68919	8.18804
22CW2 8a	7.65820	8.15572
22CW2 8b	7.64431	8.14330
22CW2 9a	7.63638	8.13161
22CW2 9b	7.70544	8.20417
22CW2 10a	7.79888	8.29560
22CW2 10b	7.70472	8.20520
22CW2 11a	7.70205	8.19726
22CW2 11b	7.65314	8.15255
22CW2 12a	7.66436	8.16177
22CW2 12b	7.63511	8.13464
22C2 1a	7.68164	8.17574
22C2 1b	7.61038	8.10902
22C2 2a	7.69565	8.19215
22C2 2b	7.63678	8.13439
22C2 3a	7.66742	8.16373
22C2 3b	7.59333	8.09157
22C2 4a	7.64302	8.13899
22C2 4b	7.60592	8.10478
22C2 5a	7.65056	8.14546
22C2 5b	7.72465	8.22202
22C2 6a	7.70851	8.20375
22C2 6b	7.69170	8.19151
22C2 7a	7.62909	8.12540
22C2 7b	7.69523	8.19416
22C2 8a	7.62907	8.12509
22C2 8b	7.65821	8.15700
22C2 9a	7.65375	8.14933
22C2 9b	7.66250	8.16059
22C2 10a	7.64964	8.14633
22C2 10b	7.60445	8.10248
22C2 11a	7.78765	8.28365
22C2 11b	7.59394	8.08920
22C2 12a	7.66252	8.15652
22C2 12b	7.64809	8.14392
Spike 61A2-1	7.67846	7.92879
Spike 61A2-2	7.62287	7.87470

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PB

Table 6:

19CALW2-1

Before 53.17982

pH 4.39

19CALW2-2

53.36932

4.55

Sample ID	Mass (g) Before LSA	pH of Exp. Soln.
19CALW2-1	53	
19CALW2-2	PB 26 Jul 2006	4.55
19CALW2-3	52.93427	4.65
19CALW2-4	PB 26 Jul 06 53.355 53.35497	4.89
19CALW2-5	53.12153	5.09
19CALW2-6	52.61374	5.33
19CALW2-7	53.44703	5.59
19CALW2-8	53.00860	7.29
19CALW2-9	53.27380	7.69
19CALW2-10	53.12790	8.03
19CALW2-11	53.09177	8.47
19CALW2-12	53.39291	8.61
19CAL2-1	53.25483	4.95
19CAL2-2	53.07502	7.36
19CAL2-3	53.05273	7.63
19CAL2-4	53.16947	7.72
19CAL2-5	53.29042	7.68
19CAL2-6	52.94998	7.75
19CAL2-7	52.95051	7.83
19CAL2-8	52.78971	8.13
19CAL2-9	52.90489	8.31
19CAL2-10	52.94648	8.26
19CAL2-11	52.82150	8.47
19CAL2-12	53.41200	8.65
22CALW2-1	53.22134	3.96
22CALW2-2	53.04318	4.07
22CALW2-3	53.09433	4.22
22CALW2-4	53.10914	4.32
22CALW2-5	52.19822	4.41
22CALW2-6	52.87505	4.75
22CALW2-7	52.48470	4.94
22CALW2-8	53.03131	6.68
22CALW2-9	53.02758	7.41
22CALW2-10	52.92573	7.94
22CALW2-11	52.83239	8.38
22CALW2-12	53.23130	8.71
22CAL2-1	53.14967	7.57
22CAL2-2	52.98600	7.68
22CAL2-3	53.30326	7.70
22CAL2-4	52.91402	7.81
22CAL2-5	53.03582	7.90
22CAL2-6	52.94904	7.85
22CAL2-7		
22CAL2-8		

4.39

(*) (*)

see

p. 26

(*)

see

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Table 6: cont mass (g) pH

22CAL2-9	53.16584	8.12
22CAL2-10	52.96500	8.24
22CAL2-11	52.61410	8.50
22CAL2-12	53.16991	8.72

mass before sampling pH

22 CAL 2-7	52.89146	7.95
22 CAL 2-8	53.25652	7.98

⊛ Note: 22 CAL 2-7 and 2-8 are entered here because tape on the chart on p. 25 precludes any data entry for those solutions.

⊛ ⊛ Intermediate pH probe challenge w/ pH 7 buffer - Reading = 6.99

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Preparation of Cation and Anion QA Solutions for 19CAL Series 1 and 22CAL Series 1 with and without Calcite Sorption Experiment

Series 2
Series 2

Personnel Involved: Sandra Watson

Objective: To prepare QA solutions to include with 19CAL Series 1 and 22CAL Series 1 with and without Calcite cation and anion samples sent to Div 01 for analyses.

Reagents: Spex Certiprep Instrument Calibration Standard 3 (cat # CL-CAL-3, lot # 29-19AS)
Spex Certiprep IC Instrument Check Standard 2 (cat # ICMIX2-100, lot # 28-164AS)
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Supplies: Volumetric pipets
Volumetric flasks
HDPE bottles
Squirt bottle for water
Kimwipes

Procedure

Added 20 mL (volumetric pipet) of cation mix (Spex CL-CAL-3) into a 1000 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 1 L HDPE bottle and labeled Cation Std, date, initials, and SN ref. The cation dilution factor is 50.

Added 10 mL (volumetric pipet) of anion mix (Spex ICMIX2-100) into a 500 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 500 mL HDPE bottle and labeled Anion Std, date, initials, and SN. The anion dilution factor is 50.

Table of Target Conc (ppm) of QA standards for samples

Species	Conc (ppm) in Spex Standard	Dilution Factor	Target Conc (ppm) of QA Standard
K	1000	50	20
Ca	1000	50	20
Na	1000	50	20
Mg	1000	50	20
HPO4	600	50	12
Br	400	50	8
NO3	400	50	8
SO4	400	50	8
Cl	200	50	4
F	100	50	2

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Sampling of 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions for Div 01 Cation and Anion Analyses

Task initially assigned to: Paul Bertetti and Sandra Watson

Objective:

To characterize 19CAL Series 2 and 22CAL Series 2 with and without Calcite Sorption Experimental Solutions water chemistry at the conclusion of the sorption experiments by analyses of cations and anions.

Initial Entry: 811/2-3

Samples and Reagents:

Total Solutions: 50 cation and 50 anion

Experimental solutions: 48 cation and 48 anion from experimental solutions: 19CALW2-1 to 12, 19CAL2-1 to 12 and 22CALW2-1 to 12, 22CAL2-1 to 12 (811/15-16).

QA solutions: two cation QA and two anion QA stds (811/27)

Nitric acid: Labeled 1:1 nitric acid 742/74

Equipment:

100 mL pp beakers

Filters – Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 μ m pore size

10mL syringe, BD#309604-04

30mL pp bottles

60mL pp bottles

Eppendorff pipette and tips

Note:

The cation and anion analyses samples were collected after the pH analyses was conducted (811/25-26)

Background:

Experimental solutions were to be characterized by cation and anion analyses. Due to limited volume of each experimental solution (about 30 mL), anion samples were generated by adding Type 1 water to small aliquots removed from the cation samples. The anion samples Division 1 analyzes are diluted and do not directly represent the concentrations in the experimental solutions. Dilution factors will be determined by mass not volume.

Procedure:

A 10cc syringe was used to collect as much sample as possible from the centrifuge tube. Next a filter was attached to the end of the syringe. The sample was dispensed into a labeled 60mL pp bottle for cation analysis. The label contained a Div 01 ID, initials, and date. The filter was set aside in a contained

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location and the syringe was again inserted into the centrifuge tube and the experimental solution was withdrawn. The filter was again placed on the syringe and the sample was dispensed into the labeled pp bottle. The filter was then properly discarded in the solid radioactive waste bin. This process was repeated for each of the experimental solutions.

The first step in generating the anion samples was recording the mass of appropriately labeled 30 mL pp bottles (811/32-33). Using an eppendorff pipet, about a 5mL aliquot of each cation sample was transferred to the appropriate anion bottle. The new mass of the each anion bottle was measured and recorded. Approximately 15mL of Type 1 water was then transferred to each anion bottle. The new mass of the each anion bottle was measured and recorded. The dilution factor was about four and determined by mass for each sample.

Each cation sample was preserved by adding 70 μ L (eppendorff pipette) of 1:1 nitric acid.

A total of four QA samples were generated: two duplicate cation and two duplicate anion. Aliquots of cation and anion QA stds (811/27) were decanted into two labeled 60mL pp bottles and two labeled 30mL pp bottles respectively.

The following describes the Division 1 sample ID scheme. The first number (19 or 22) represents the well which the solid material was collected from. The "C" indicates that the solid material was a composite sample from several borehole depths. For samples also containing a "W", the solid was not previously treated to remove calcite. The "2" indicates the experimental series number for these samples. Following a space, the final number (1-12) in the sample ID represents the individual experimental solution. The suffix (C or A) represents whether the sample is intended for cation or anion analysis.

In addition, sample IDs 19C2-13C, 22C2-13C, 19C2-13A and 22C2-13A were used for the cation and anion QA samples respectively. The following is complete legend relating the GED IDs with the DIV 01 IDs.

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GED Sample ID	Div 01 Sample ID- Cations	Div 01 Sample ID- Anions
19CALW2-1	19CW2 1C	19CW2 1A
19CALW2-2	19CW2 2C	19CW2 2A
19CALW2-3	19CW2 3C	19CW2 3A
19CALW2-4	19CW2 4C	19CW2 4A
19CALW2-5	19CW2 5C	19CW2 5A
19CALW2-6	19CW2 6C	19CW2 6A
19CALW2-7	19CW2 7C	19CW2 7A
19CALW2-8	19CW2 8C	19CW2 8A
19CALW2-9	19CW2 9C	19CW2 9A
19CALW2-10	19CW2 10C	19CW2 10A
19CALW2-11	19CW2 11C	19CW2 11A
19CALW2-12	19CW2 12C	19CW2 12A
19CAL2-1	19C2 1C	19C2 1A
19CAL2-2	19C2 2C	19C2 2A
19CAL2-3	19C2 3C	19C2 3A
19CAL2-4	19C2 4C	19C2 4A
19CAL2-5	19C2 5C	19C2 5A
19CAL2-6	19C2 6C	19C2 6A
19CAL2-7	19C2 7C	19C2 7A
19CAL2-8	19C2 8C	19C2 8A
19CAL2-9	19C2 9C	19C2 9A
19CAL2-10	19C2 10C	19C2 10A
19CAL2-11	19C2 11C	19C2 11A
19CAL2-12	19C2 12C	19C2 12A
22CALW2-1	22CW2 1C	22CW2 1A
22CALW2-2	22CW2 2C	22CW2 2A
22CALW2-3	22CW2 3C	22CW2 3A
22CALW2-4	22CW2 4C	22CW2 4A
22CALW2-5	22CW2 5C	22CW2 5A
22CALW2-6	22CW2 6C	22CW2 6A
22CALW2-7	22CW2 7C	22CW2 7A
22CALW2-8	22CW2 8C	22CW2 8A
22CALW2-9	22CW2 9C	22CW2 9A
22CALW2-10	22CW2 10C	22CW2 10A
22CALW2-11	22CW2 11C	22CW2 11A
22CALW2-12	22CW2 12C	22CW2 12A
22CAL2-1	22C2 1C	22C2 1A
22CAL2-2	22C2 2C	22C2 2A
22CAL2-3	22C2 3C	22C2 3A
22CAL2-4	22C2 4C	22C2 4A
22CAL2-5	22C2 5C	22C2 5A
22CAL2-6	22C2 6C	22C2 6A
22CAL2-7	22C2 7C	22C2 7A
22CAL2-8	22C2 8C	22C2 8A
22CAL2-9	22C2 9C	22C2 9A
22CAL2-10	22C2 10C	22C2 10A
22CAL2-11	22C2 11C	22C2 11A

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22CAL2-12	22C2 12C	22C2 12A
Cation QA Std - NpCS SN#811/27 (7/26/2006)	19C2 13C	na
Cation QA Std - NpCS SN#811/27 (7/26/2006)	22C2 13C	na
Anion QA Std - NpCS SN#811/27 (7/26/2006)	na	19C2 13A
Anion QA Std - NpCS SN#811/27 (7/26/2006)	na	22C2 13A

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Div 01 Sample ID- Anions	Mass (g) Empty Bottle	Mass (g) bottle & exp. soln.	Mass (g) bottle, exp. soln. & Type 1 water
19CW2-1A	8.27521	13.21338	28.41197
19CW2-2A	8.20146	13.11976	28.29165
19CW2-3A	8.27319	13.19928	28.36801
19CW2-4A	8.18078	13.11837	28.31988
19CW2-5A	8.20306	13.13229	28.31072
19CW2-6A	8.18609	13.11557	28.28618
19CW2-7A	8.20062	13.14955	28.32068
19CW2-8A	8.23770	13.15883	28.27990
19CW2-9A	8.24212	13.15259	28.28628
19CW2-10A	8.20752	13.13609	28.25101
19CW2-11A	8.22583	13.16446	28.26100
19CW2-12A	8.22046	13.14661	28.21846
19C2-1A	8.20067	13.16190	28.13814
19C2-2A	8.18833	13.13160	28.13565
19C2-3A	8.19099	13.12796	28.15169
19C2-4A	8.20184	13.14990	28.25205
19C2-5A	8.19480	13.14183	28.07249
19C2-6A	8.26567	13.19471	28.29465
19C2-7A	8.19303	13.11646	28.15489
19C2-8A	8.20243	13.15166	28.19349
19C2-9A	8.20064	13.13872	27.96363
19C2-10A	8.19691	13.12673	28.12216
19C2-11A	8.19627	13.11720	28.16223
19C2-12A	8.20359	13.13613	28.12670
22CW2-1A	8.41011	13.36435	28.29178
22CW2-2A	8.36939	13.32515	28.51536
22CW2-3A	8.36548	13.31487	28.46322
22CW2-4A	8.37523	13.32135	28.43581
22CW2-5A	8.37769	13.30579	28.44490
22CW2-6A	8.35695	13.29268	28.38762
22CW2-7A	8.34639	13.29460	28.10806
22CW2-8A	8.31961	13.25978	28.39252
22CW2-9A	8.42523	13.35850	28.36320
22CW2-10A	8.34117	13.27888	28.37638
22CW2-11A	8.41992	13.34429	28.38952
22CW2-12A	8.34914	13.30132	27.80250

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Div 01 Sample ID- Anions	Mass (g) Empty Bottle	Mass (g) bottle & exp. soln.	Mass (g) bottle, exp. soln. & Type 1 water
22C2-1A	8.37115	13.49571	28.44575
22C2-2A	8.40863	13.44786	28.54546
22C2-3A	8.34258	13.35142	28.29622
22C2-4A	8.35736	13.34462	28.46371
22C2-5A	8.39023	13.36747	28.47688
22C2-6A	8.44853	13.42100	28.57127
22C2-7A	8.41662	13.39075	28.44790
22C2-8A	8.47955	13.44709	28.53590
22C2-9A	8.37084	13.32870	28.34735
22C2-10A	8.40813	13.36830	28.40558
22C2-11A	8.42525	13.39486	28.48682
22C2-12A	8.35361	13.30986	28.44329

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Delivery of 19CAL2 series w/ and w/o Calcite and 22CAL2 series w/ and w/o calcite samples to Div. 01 for Cation and Anion Analyses

Initial Entry for Sorption Exp : 811 / 2

Total Samples : 100

-48 Cation

(12) 19CW2, (12) 19C2, (12) 22CW2, (12) 22C2

-48 Anion

(12) 19CW2, (12) 19C2, (12) 22CW2, (12) 22C2

-4 QA

(2) cation, (2) anion

Sample and QA collection : 811 / 28-29

Sample legend : 811 / 30-31

QA target concentrations : 811 / 27

Chain of Custody on following pages :

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Shipper Name/Address		Client		Sample Collection Date (m/d/yy)		Sample Collection Time		Matrix Type		Sample Type		# of Containers			
Bradley Wierling Division 20 B-57		Div. 20		8/1/06				W		W		1			
Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <i>4 weeks</i>		SwRI Contact Mike Dammann		SwRI Project#: 20.0602.01.242		Received by SwRI Lab: (Signature) <i>[Signature]</i>		Date: 8/1/06		Time: 0920		Samples Disposed: Date: Time:			
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		Relinquished by (Print/Signature)		Received by (Print/Signature)		Date			
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 Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Therm #: 027		Comments: RADIOACTIVE Np 237 @ 8.5 x 10 ⁻⁷ M or 1.4 x 10 ⁻⁴ µCi/mL SW 8/1/06		Relinquished by (Print/Signature)		Received by (Print/Signature)		Date		Time	
Remarks: Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)		PQ.P. IS APPLIES		PQC - B. WERLING 6565											

1 AUGUST 2006 SW

1 AUGUST 2006

SW

SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166							Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 wks</u>	
Client Bradley Werling Division 20 B-57						SwRI Contact Mike Dammann		
Client Purchase Order/Other ID Div. 20				Site/Zone ID				
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analyses Requested		
19CW2-11C	07/25/06		M		1	ANALYSES OF MAJOR AND MINOR ELEMENTS BY ICP		
19CW2-12C					1			
19C2-1C					1			
19C2-2C					1			
19C2-3C					1			
19C2-4C					1			
19C2-5C					1			
19C2-6C					1			
19C2-7C					1			
19C2-8C					1			
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: <u>22.0°C</u> Therm #: <u>027</u>								
Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank								
Comments: RADIOACTIVE Np-237 @ 8.5 x 10 ⁻⁷ M or 1.1 x 10 ⁻⁷ µCi/mL SW 8/1/34						SwRI Project#: <u>20-06002-01-242</u> Received by SwRI Lab: (Signature) <u>[Signature]</u> Date: <u>8/1/06</u> Time: <u>0920</u> Samples Disposed: _____ Date: _____ Time: _____ Samples Disposed by: _____		

1 AUGUST 2006

SW

SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166							Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 wks</u>	
Client Bradley Werling Division 20 B-57						SwRI Contact Mike Dammann		
Client Purchase Order/Other ID Div. 20				Site/Zone ID				
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analyses Requested		
19C2-9C	07/25/06		M		1	ANALYSES OF MAJOR AND MINOR ELEMENTS BY ICP		
19C2-10C					1			
19C2-11C					1			
19C2-12C					1			
19C2-13C					1			
22CW2-1C					1			
22CW2-2C					1			
22CW2-3C					1			
22CW2-4C					1			
22CW2-5C					1			
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: <u>22.0°C</u> Therm #: <u>027</u>								
Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank								
Comments: RADIOACTIVE Np-237 @ 8.5 x 10 ⁻⁷ M or 1.1 x 10 ⁻⁷ µCi/mL SW 8/1/34						SwRI Project#: <u>20-06002-01-242</u> Received by SwRI Lab: (Signature) <u>[Signature]</u> Date: <u>8/1/06</u> Time: <u>0920</u> Samples Disposed: _____ Date: _____ Time: _____ Samples Disposed by: _____		

SW
1 AUGUST 2006

<p>SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166</p>		<p>Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 Wks</u></p>	
<p>Bradley Werling Division 20 B-57</p>		<p>SwRI Contact Mike Dammann</p>	
<p>Client Purchase Order/Other ID Div. 20</p>		<p>Site/Zone ID</p>	
<p>Analyses Requested</p>			
Sample ID	Sample Collection Date (m/d/yy)	Sample Collection Time	Matrix Type
22CW2-6C	07/28/06		M
22CW2-7C			
22CW2-8C			
22CW2-9C			
22CW2-10C			
22CW2-11C			
22CW2-12C			
22C2-1C			
22C2-2C			
22C2-3C			
<p>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</p>			
<p>Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank</p>			
<p>Therm #: 027 Comments: RADIOACTIVE N/A 237 @ 8.5 x 10⁻⁷ M or 1.1 x 10⁻⁴ uCi/mL SW 8/11/34</p>			
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
<p>REMARKS Preservation a = HCl to pH <2 b = HNO₃ to pH <2 c = H₂SO₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)</p>			
<p>SwRI Project: 20.06002.01.212</p>			
<p>Received by SwRI Lab (Signature) [Signature]</p>			
Date	Time	Samples Disposed Date	Time
8/11/06	0920		
<p>Samples Disposed by:</p>			

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

Page ___ of ___

<p>SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166</p>		<p>Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 Wks</u></p>	
<p>Bradley Werling Division 20 B-57</p>		<p>SwRI Contact Mike Dammann</p>	
<p>Client Purchase Order/Other ID Div. 20</p>		<p>Site/Zone ID</p>	
<p>Analyses Requested</p>			
Sample ID	Sample Collection Date (m/d/yy)	Sample Collection Time	Matrix Type
22C2-4C	07/28/06		M
22C2-5C			
22C2-6C			
22C2-7C			
22C2-8C			
22C2-9C			
22C2-10C			
22C2-11C			
22C2-12C			
22C2-13C			
<p>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</p>			
<p>Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank</p>			
<p>Therm #: 027 Comments: RADIOACTIVE N/A 237 @ 8.5 x 10⁻⁷ M or 1.1 x 10⁻⁴ uCi/mL SW 8/11/34</p>			
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
Relinquished by (Print/Signature)	Date	Time	Time
Received by (Print/Signature)	Date	Time	Time
<p>REMARKS Preservation a = HCl to pH <2 b = HNO₃ to pH <2 c = H₂SO₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)</p>			
<p>SwRI Project: 20.06002.01.212</p>			
<p>Received by SwRI Lab (Signature) [Signature]</p>			
Date	Time	Samples Disposed Date	Time
8/11/06	0920		
<p>Samples Disposed by:</p>			

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

Page ___ of ___

SW
1 AUGUST 2006

SW
1 AUGUST 2006

Shipper Name/Address Bradley Werling B-57 Div. 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 wks</u>	
Client Div. 20		Client Purchase Order/Other ID		Site/Zone ID		SwRI Contact Mike Dammann	
Sample ID 19CW2-1A 19CW2-2A 19CW2-3A 19CW2-4A 19CW2-5A 19CW2-6A 19CW2-7A 19CW2-8A 19CW2-9A 19CW2-10A		Sample Collection Date (m/d/yy) 7/28/06		Sample Collection Time W		Matrix Type W	
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 #: 027		Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		# of Containers 1		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature)	
Comments: RADIOACTIVE No 237 @ 8.5 x 10 ⁻⁷ M or 1.4 x 10 ⁻⁴ µCi/mL SU 811/34		Relinquished by (Print/Signature) Received by (Print/Signature)		Date 8/1/06		Time 0920	
Div 01 COC Form 01-01-001, Rev 8/02		Page 1 of 1		SwRI Project: 20.06002.01.242		Received by SwRI Lab: (Signature) Date 8/1/06	
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 #: 027		Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		# of Containers 1		Relinquished by (Print/Signature) Received by (Print/Signature)	
Comments: RADIOACTIVE No 237 @ 8.5 x 10 ⁻⁷ M or 1.4 x 10 ⁻⁴ µCi/mL SW 811/34		Relinquished by (Print/Signature) Received by (Print/Signature)		Date 8/1/06		Time 0920	
Div 01 COC Form 01-01-001, Rev 8/02		Page 1 of 1		SwRI Project: 20.06002.01.242		Received by SwRI Lab: (Signature) Date 8/1/06	

SW
1 AUGUST 2006

Shipper Name/Address Bradley Werling B-57 Division 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 wks</u>	
Client Division 20		Client Purchase Order/Other ID		Site/Zone ID		SwRI Contact Mike Dammann	
Sample ID 19CW2-11A 19CW2-12A 19C2-1A 19C2-2A 19C2-3A 19C2-4A 19C2-5A 19C2-6A 19C2-7A 19C2-8A		Sample Collection Date (m/d/yy) 7/28/06		Sample Collection Time W		Matrix Type W	
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 #: 027		Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		# of Containers 1		Relinquished by (Print/Signature) Received by (Print/Signature)	
Comments: RADIOACTIVE No 237 @ 8.5 x 10 ⁻⁷ M or 1.4 x 10 ⁻⁴ µCi/mL SW 811/34		Relinquished by (Print/Signature) Received by (Print/Signature)		Date 8/1/06		Time 0920	
Div 01 COC Form 01-01-001, Rev 8/02		Page 1 of 1		SwRI Project: 20.06002.01.242		Received by SwRI Lab: (Signature) Date 8/1/06	

SW
1 AUGUST 2006

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 WKS

Client: Bradley Werling
B-57
Div. 20

Client Purchase Order/Other ID: _____
 Site/Zone ID: _____

SwRI Contact: Mike Damman

REMARKS
 Preservation
 a = HCl to pH <2
 b = HNO₃ to pH <2
 c = H₂SO₄ to pH <2
 d = NaOH to pH >12
 e = Cool (4°C±2°C)
 f = Other (specify)
None
PQP-15 APPLIES
POC-B. WERLING
6565

Sample ID	Sample Collection Date (m/d/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Relinquished by (Print/Signature)	Date	Time	SwRI Project:
19C2-9A	7/28/06		M		1	Relinquished by (Print/Signature) <u>Sandra Watson / Sandra Watson</u>			20.06002.01.2-12
19C2-10A						Received by (Print/Signature)			(Signature) <u>[Signature]</u>
19C2-11A						Relinquished by (Print/Signature)			Date <u>8/1/06</u>
19C2-12A						Received by (Print/Signature)			Time <u>0920</u>
19C2-13A						Relinquished by (Print/Signature)			Samples Disposed: Date
22CW2-1A						Received by (Print/Signature)			Time
22CW2-2A						Relinquished by (Print/Signature)			Samples Disposed by:
22CW2-3A						Received by (Print/Signature)			
22CW2-4A						Relinquished by (Print/Signature)			
22CW2-5A						Received by (Print/Signature)			

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 Rinse
 ES - Environmental Sample
 FB - Field Blank
 FD - Field Duplicate
 MS - Matrix Spike
 MSD - Matrix Spike Dup
 TB - Trip Blank

Temp: 21.0°C Therm #: 027
 Comments: RADIOACTIVE
Np 237 @ 8.5 x 10^-7 M or 1.4 x 10^-4 µCi/mL
SW 8/1/06

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

SW
1 AUGUST 2006

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 WKS

Client: Bradley Werling
B-57
Div. 20

Client Purchase Order/Other ID: _____
 Site/Zone ID: _____

SwRI Contact: Mike Damman

REMARKS
 Preservation
 a = HCl to pH <2
 b = HNO₃ to pH <2
 c = H₂SO₄ to pH <2
 d = NaOH to pH >12
 e = Cool (4°C±2°C)
 f = Other (specify)
None
PQP-15 APPLIES
POC-B. WERLING
6565

Sample ID	Sample Collection Date (m/d/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Relinquished by (Print/Signature)	Date	Time	SwRI Project:
22CW2-6A	7/28/06		M		1	Relinquished by (Print/Signature) <u>Sandra Watson / Sandra Watson</u>			20.06002.01.2-12
22CW2-7A						Received by (Print/Signature)			(Signature) <u>[Signature]</u>
22CW2-8A						Relinquished by (Print/Signature)			Date <u>8/1/06</u>
22CW2-9A						Received by (Print/Signature)			Time <u>0920</u>
22CW2-10A						Relinquished by (Print/Signature)			Samples Disposed: Date
22CW2-11A						Received by (Print/Signature)			Time
22CW2-12A						Relinquished by (Print/Signature)			Samples Disposed by:
22C2-1A						Received by (Print/Signature)			
22C2-2A						Relinquished by (Print/Signature)			
22C2-3A						Received by (Print/Signature)			

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 Rinse
 ES - Environmental Sample
 FB - Field Blank
 FD - Field Duplicate
 MS - Matrix Spike
 MSD - Matrix Spike Dup
 TB - Trip Blank

Temp: 21.0°C Therm #: 027
 Comments: RADIOACTIVE
Np 237 @ 8.5 x 10^-7 M or 1.4 x 10^-4 µCi/mL
SW 8/1/06

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

9 August 2006 MR

SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 Weeks</u>			
Client Name/Address Bradley Weeling B-57 Div. 20		SWRI Contact Mike Dammann			
Client Purchase Order/Other ID _____		Site/Zone ID _____			
Analyses Requested		REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) NONE POP-15 APPLIES POP-B WERLUNG 6545			
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
22C2-41A	7/21/06		W		1
22C2-5A					
22C2-6A					
22C2-7A					
22C2-8A					
22C2-9A					
22C2-10A					
22C2-11A					
22C2-12A					
22C2-13A					
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: <u>72.0°C</u> Therm #: <u>027</u>		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		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature)	
Comments: <u>RADIOACTIVE</u> <u>AP 237 @ 8.5 x 10⁻⁷ M or 1.4 x 10⁻⁷ M.C./μL</u> <u>SW 8/11/06</u>		Date 8/11/06 Time 0920		Samples Disposed: Date _____ Time _____	
SWRI Project: <u>2006002.01.242</u>		Received by SWRI Lab: (Signature) 		Samples Disposed by: Date _____ Time _____	

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

Liquid Scintillation Analyses Results for 19CAL Series 2 and 22CAL Series 2 with and without Calcite Experimental Solutions

Personnel Involved: Paul Bertetti, Sandra Watson

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

Initial Entry: 811/2-3

LSA sample preparation: 811/20-24

Sample Info

Blank (1 sample from 19PB-3 simulated water): w Ca 811/20
 Neptunium spikes (2 samples) – Spike 61A2-1 and Spike 61A2-2 (811/20)

Four experiments derived from 4 solids and 1 type of simulated water

- 19CAL Series 2 with Calcite (19CW2 - 24 samples) using 19PB-3 simulated water
- 19CAL Series 2 without Calcite (19C2 - 24 samples) using 19PB-3 simulated water
- 22CAL Series 2 with Calcite (22CW2 - 24 samples) using 19PB-3 simulated water
- 22CAL Series 2 without Calcite (22C2 - 24 samples) using 19PB-3 simulated water

Total number of LSA samples: 99

Results

Printout of LSA analyses following:

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\20060728_1742.results

Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.048

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): 200.00

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

Colored Samples: n/a

Coincidence Time (nsec): 18

Luminescence Correction: Off

Heterogeneity Monitor: n/a

Delay Before Burst (nsec): 75

Half Life-

Half Life Correction: Off

Regions Half Life

Regions	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: 18.98 Date Processed: 7/28/06 5:42:41 PM

14C Chi Square: 20.37 Date Processed: 7/28/06 5:42:41 PM

3H E^2/B (1-18.6 keV): 271.75 Date Processed: 7/28/06 5:42:41 PM

14C E^2/B (4-156 keV): 522.75 Date Processed: 7/28/06 5:42:41 PM

3H Efficiency (0-18.6 keV): 65.06 Date Processed: 7/28/06 5:42:41 PM

14C Efficiency (0-156 keV): 96.70 Date Processed: 7/28/06 5:42:41 PM

IPA Background Date Processed: 7/28/06 5:42:41 PM

3H Background CPM (0-18.6 keV): 15.57 Date Processed: 7/28/06 5:42:41 PM

14C Background CPM (0-156 keV): 22.53 Date Processed: 7/28/06 5:42:41 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	200.00	19.36	3.21	2.94	8.24	0.45	21.20	159.0
---	--------	-------	------	------	------	------	-------	-------

B

Missing vial 2.

3	79.99	11.27	12.29	4.22	15.31	124.58	2.01	263.6
4	79.95	13.15	10.79	4.52	14.54	124.65	2.01	243.7
5	78.71	10.93	12.69	4.11	15.73	126.60	2.01	277.0
6	79.52	10.96	12.61	4.21	15.37	125.32	2.01	268.8
7	80.25	10.59	12.95	4.05	15.78	124.17	2.01	276.4
8	79.65	10.17	13.44	3.99	16.01	125.13	2.01	268.5
9	80.08	9.95	13.67	3.37	18.14	124.44	2.01	254.2
10	80.90	9.71	13.92	3.94	16.04	123.18	2.01	283.4
11	81.43	9.66	13.93	3.48	17.59	122.36	2.01	268.8
12	80.03	9.27	14.54	3.69	16.94	124.51	2.01	278.4
13	79.56	11.65	11.97	4.17	15.48	125.26	2.01	268.0
14	80.03	10.35	13.22	3.78	16.64	124.52	2.01	270.1
15	65.37	14.59	10.76	5.74	13.38	152.56	2.01	268.6
16	65.38	12.99	11.84	5.48	13.82	152.51	2.01	299.6
17	70.16	10.63	13.62	4.04	16.74	142.10	2.01	286.8
18	70.63	11.93	12.31	4.40	15.65	141.15	2.01	267.9
19	85.44	10.09	13.17	3.69	16.47	116.62	2.01	266.6
20	85.82	9.27	14.15	3.77	16.19	116.10	2.01	288.7
21	86.02	10.36	12.84	4.50	14.16	115.81	2.01	311.4
22	85.49	11.01	12.21	4.55	14.06	116.53	2.01	290.0
23	85.78	10.55	12.65	3.92	15.70	116.13	2.01	270.7
24	84.97	10.97	12.28	4.54	14.13	117.24	2.01	292.4
25	90.35	12.03	11.08	5.44	12.05	110.26	2.01	316.1
26	89.40	13.09	10.36	5.08	12.74	111.41	2.01	279.0

Missing vial 27.

28	79.88	15.06	9.65	5.94	11.94	124.74	2.01	275.0
29	81.49	15.77	9.22	5.87	11.95	122.28	2.01	269.1
30	84.73	13.83	10.11	5.77	11.89	117.61	2.01	296.2
31	83.40	14.81	9.61	6.01	11.63	119.47	2.01	282.9
32	86.72	14.43	9.66	6.48	10.85	114.90	2.01	297.1
33	87.37	15.82	8.94	5.97	11.45	114.01	2.01	269.0
34	89.11	20.34	7.24	8.27	9.07	111.80	2.01	280.3
35	89.45	19.02	7.62	7.91	9.33	111.36	2.01	291.1
36	94.29	13.99	9.60	6.25	10.72	105.62	2.01	293.7
37	91.28	14.66	9.35	6.15	11.00	109.11	2.01	301.8
38	96.90	14.31	9.32	6.06	10.82	102.77	2.01	301.9
39	95.66	14.37	9.33	6.01	10.95	104.11	2.01	288.6
40	97.62	16.73	8.16	6.84	9.92	102.00	2.01	292.5
41	94.43	16.96	8.18	6.84	10.06	105.45	2.01	278.7
42	98.13	18.57	7.49	7.52	9.26	101.48	2.01	280.0
43	98.31	18.11	7.63	7.65	9.15	101.28	2.01	297.0
44	96.33	19.35	7.30	7.58	9.29	103.36	2.01	277.8
45	96.38	18.59	7.54	7.87	9.06	103.33	2.01	299.4
46	98.88	20.20	6.98	7.84	8.98	100.72	2.01	273.3
47	99.33	20.34	6.93	8.62	8.40	100.23	2.01	301.0
48	88.54	27.00	5.83	11.40	7.38	112.51	2.01	293.0
49	87.60	26.64	5.92	11.46	7.39	113.73	2.01	295.3
50	82.48	26.94	6.02	11.01	7.79	120.82	2.01	288.8
51	80.92	28.20	5.87	11.76	7.54	123.13	2.01	291.9

Missing vial 52.

Blank WCA

19C2 1a

19C2 1b

2a

2b

3a

3b

4a

4b

5a

5b

6a

6b

7a

7b

8a

8b

9a

9b

10a

10b

11a

11b

12a

53	65.40	34.80	5.53	13.11	7.78	152.51	2.01	270.6	22CW2 1a
54	64.15	34.67	5.59	13.56	7.69	155.46	2.01	274.7	22CW2 1b
55	64.79	31.04	6.03	11.04	8.70	153.92	2.01	258.7	" 2a
56	63.50	31.54	6.01	11.37	8.62	157.05	2.01	262.9	" 2b
57	65.54	32.38	5.81	12.19	8.13	152.15	2.01	275.6	" 3a
58	64.22	33.23	5.76	12.92	7.92	155.27	2.01	279.7	" 3b
59	64.74	29.12	6.32	10.66	8.90	154.02	2.01	269.6	" 4a
60	66.08	31.70	5.88	11.99	8.18	150.93	2.01	269.3	" 4b
61	64.76	29.56	6.24	10.33	9.07	154.00	2.01	259.3	" 5a
62	61.32	30.55	6.25	10.87	9.02	162.65	2.01	257.5	" 5b
63	65.26	28.12	6.46	10.29	9.06	152.82	2.01	264.9	" 6a
64	64.97	26.84	6.70	10.37	9.04	153.49	2.01	285.2	" 6b
65	64.35	23.86	7.35	9.50	9.60	154.96	2.01	283.5	" 7a
66	63.41	24.98	7.14	9.50	9.67	157.27	2.01	276.9	" 7b
67	68.38	20.26	8.12	7.92	10.52	145.80	2.01	274.2	" 8a
68	67.72	19.75	8.32	7.69	10.78	147.27	2.01	280.2	" 8b
69	71.85	18.84	8.42	7.80	10.39	138.78	2.01	294.9	" 9a
70	71.78	20.03	8.02	7.95	10.27	138.90	2.01	277.0	" 9b
71	72.79	20.91	7.71	7.62	10.49	136.95	2.01	264.1	" 10a
72	72.31	20.10	7.98	7.62	10.53	137.86	2.01	275.2	" 10b
73	68.74	21.49	7.73	8.17	10.28	145.03	2.01	275.4	" 11a
74	68.80	22.05	7.58	8.22	10.24	144.92	2.01	273.3	" 11b
Missing vial 75.									
76	85.35	21.00	7.19	7.93	9.51	116.73	2.01	271.6	22CW2 12a
77	85.67	20.89	7.21	8.07	9.38	116.28	2.01	278.2	22CW2 12b
78	72.76	27.04	6.34	10.52	8.50	137.02	2.01	280.1	22CZ 1a
79	71.61	26.31	6.51	10.75	8.44	139.21	2.01	291.4	22CZ 1b
80	73.05	27.46	6.25	10.40	8.54	136.46	2.01	281.2	" 2a
81	72.73	28.16	6.15	11.12	8.20	137.06	2.01	283.9	" 2b
82	74.08	28.35	6.07	11.05	8.17	134.54	2.01	283.0	" 3a
83	72.26	28.03	6.19	11.45	8.08	137.97	2.01	298.8	" 3b
84	76.03	32.36	5.45	12.86	7.34	131.08	2.01	280.9	" 4a
85	75.86	31.87	5.51	12.94	7.32	131.40	2.01	283.9	" 4b
86	74.01	32.26	5.52	12.16	7.69	134.67	2.01	274.3	" 5a
87	75.32	33.88	5.29	13.92	7.02	132.32	2.01	288.6	" 5b
88	75.31	33.51	5.33	14.14	6.95	132.34	2.01	291.0	" 6a
89	74.11	36.01	5.10	14.56	6.88	134.53	2.01	285.9	" 6b
90	76.53	35.79	5.05	13.75	7.02	130.25	2.01	275.1	" 7a
91	75.85	36.83	4.97	15.13	6.65	131.41	2.01	291.5	" 7b
92	76.49	35.72	5.06	14.76	6.72	130.32	2.01	290.1	" 8a
93	77.34	33.23	5.30	13.41	7.09	128.85	2.01	283.2	" 8b
94	77.76	36.17	4.98	14.89	6.64	128.18	2.01	294.3	" 9a
95	77.06	35.46	5.07	14.47	6.78	129.32	2.01	282.1	" 9b
96	75.05	35.44	5.13	13.86	7.05	132.81	2.01	277.8	" 10a
97	74.13	36.21	5.08	14.51	6.89	134.47	2.01	282.2	" 10b
98	73.31	42.27	4.58	16.66	6.38	135.98	2.01	276.7	" 11a
99	74.31	39.80	4.75	16.33	6.41	134.14	2.01	284.4	" 11b
100	70.23	42.11	4.68	16.48	6.55	141.97	2.01	279.7	" 12a
101	71.76	42.81	4.58	17.40	6.28	138.95	2.01	287.7	" 12b
Missing vial 102.									
103	0.40	20420.64	2.21	7822.05	3.58	25142.05	1.99	279.2	Spike W1A2-1
104	0.41	20931.86	2.16	8194.62	3.45	25119.07	1.97	284.0	Spike W1A2-2

Calibration Information

Software Version IC: 2.11
 Software Version EC: 1.31
 Instrument Model: Tri-Carb 3100TR
 Instrument Serial Number: 405314
 3H Chi Square: 11.12 Date Processed: 8/3/06 5:39:35 AM
 14C Chi Square: 26.59 Date Processed: 8/3/06 5:39:35 AM
 3H E^2/B (1-18.6 keV): 280.10 Date Processed: 8/3/06 5:39:35 AM
 14C E^2/B (4-156 keV): 543.72 Date Processed: 8/3/06 5:39:35 AM
 3H Efficiency (0-18.6 keV): 65.20 Date Processed: 8/3/06 5:39:35 AM
 14C Efficiency (0-156 keV): 96.40 Date Processed: 8/3/06 5:39:35 AM
 IPA Background Date Processed: 8/3/06 5:39:35 AM
 3H Background CPM (0-18.6 keV): 15.17 Date Processed: 8/3/06 5:39:35 AM
 14C Background CPM (0-156 keV): 21.98 Date Processed: 8/3/06 5:39:35 AM
 3H Calibration DPM: 285000
 3H Reference Date: 10/29/99
 14C Calibration DPM: 134100

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Continued Testing for 19PB and 22PC with and without Calcite Sorption Experiments

Background: To better understand the interactions between the sediments of 19PB and 22PC with reference solutions, pH adjustments and spikes used in the 19CAL and 22CAL Series 1 and 2 sorption experiments small scale sorption experiments will be conducted to test individual aspects of the experiment process. These small scale experiments will be identified as phases (i.e. Phase 1, Phase 2 etc.). Each Phase will have a specific aspect of the experimental process tested.

The background sections of each of the initial entries will identify which phase of experiments is being conducted as well as what alterations to methodology are taking place.

Objective: To investigate Np sorption on a NC-EWDP-19PB 0305 Consolidated Alluvium and NC-EWDP-22PC 1005 Consolidated Alluvium (with and without calcite) using simulated water (with calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB

Samples 19PB-3 Simulated Groundwater with Ca – 800/20
NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
NC-EWDP-22PC-1005-Sonic Core Composite – 742/8
NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Personnel initially assigned to task: Marla Roberts and Sandra Watson

Phase 1: Testing the repeatability of pH targets using no pH adjustments. The pH target is determined to be the pH of the reference solution (19PB-3 Simulated Groundwater w/ Ca)

16 Aug 2006 MR

Initial Entry – 19PB and 22PC with and without Calcite Sorption Experiments – Testing Repeatability of pH Targets using Np-237 on Consolidated Alluvium with 19PB Simulated Waters with Calcium

Personnel initially assigned to task: Marla Roberts and Sandra Watson

Objective: To investigate Np sorption on a NC-EWDP-19PB 0305 Consolidated Alluvium and NC-EWDP-22PC 1005 Consolidated Alluvium (with and without calcite) using simulated water (with calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB at a single pH.

Background: This is the first of a series of tests (phases) on the 19PB and 22PC with and without calcite sediments. This particular test (Phase 1) is examining the repeatability of sorption results given a specific pH. In this case no pH adjustments are made to the exp. solns. The initial pH is determined to be the pH of the 19PB-3 Simulated Groundwater with Ca – 800/20.

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-3 Simulated Groundwater with Ca – 800/20
NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
NC-EWDP-22PC-1005-Sonic Core Composite – 742/8
NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Reagents Neptunium Spike # 61A-1 (369/100) - 60ppm Np-237 at pH of 5.63
Neptunium Spike # 61A (369/ ~~95~~) - ~~100~~ ppm Np-237 at pH of
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
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Equipment: Orion meter 920A (sn 039518)
Thermo Orion combination electrode 8103BN (ID 3I)
New Brunswick Scientific Gyrotory shaker model G33 (sn 290127990)
Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
Mettler Toledo XP205DR (sn 1126461033)
Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
Centrifuge Tube Holder (Aluminum)

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

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Experimental Steps for Phase 1 Testing of 19PB and 22PC with and without Calcite Sediments- Testing Repeatability of Sorption Results Given Single pH

Personnel involved: Marla Roberts and Sandra Watson

Background: See 811/50

Initial Entry: 811/51

Note: All data (masses, etc.) recorded on 811/54

Step 1: Equilibration of Simulated Waters used in Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions

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

Procedure

Aeration of simulated water conducted since 19 June 2006- see 800/20

Step 2: Addition of Solids to Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experiments

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

Sample info NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
 NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
 NC-EWDP-22PC-1005-Sonic Core Composite - 742/8
 NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Procedure

Four sets of four polycarbonate centrifuge tubes were labeled as follows: 19W T1-8, 19WO T1-8, 22W T1-8 and 22WO T1-8. The mass of each tube (with cap) was measured and recorded on 811/54-55. An individual experimental tube was uncapped and a funnel made from weighing paper was inserted in the top. Prior to aliquot removal the jar containing the solid was gently rolled to ensure an unbiased aliquot. Approximately 0.1 g of solid was transferred to a piece of tared weighing paper on the balance. The solid was transferred into the appropriate experimental tube 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 811/54-55.

The centrifuge tubes labeled with a "W" indicate the solid contains calcite and the aliquot was removed from a sampling containing calcite (NC-EWDP-19PB-0305-Sonic Core Composite and NC-EWDP-22PC-1005-Sonic Core Composite). The centrifuge tubes labeled with a "WO" indicate the solid does not contain calcite and was removed from a treated sample (NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite and NC-EWDP-22PC-1005-Sonic Core Composite w/o Calcite). See 742/25 for further information about calcite removal. The "T" was used to signify this is a test. "19" and "22" indicate well numbers for the sediment.

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Step 3: Addition of Simulated Water Solution to Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experiment

Objective: To add simulated water to centrifuge tubes as preparation for sorption experiment.

Sample Info: 19PB-3 Simulated Groundwater with Calcium 800/20

Procedure

About 30mL (10mL pipet) of the simulated water was added to the experimental pc tubes. The mass was recorded on 811/54-55.

The sample were left over night to equilibration. All pc tubes were reweighed and the masses recorded 811/54-55.

Step 4: Measuring pH of 19PB Simulated water

Objective: Determine initial pH of exp. solutions

Sample Info: 19PB-3 Simulated Groundwater with Calcium 800/20

Procedure:

The pH of 19PB-3 Simulated Groundwater with Calcium 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 set points and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

Temperature: 18.6
 Set Points: 4.00, 7.02, 10.08
 Slope: 97.7
 pH Measured: 8.31

Step 5: Addition of Neptunium Spike to Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions

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

Reagent: Np Spike # 61A-1 (369/100)
 Np Spike # 61A (369/95) 270 ⁴⁰⁰ MR

Procedure

Added 100µL (100µL eppendorf pipet) to each experimental solution and recorded the mass of the experimental solution on 811/54-55. See Note for spike information.

****See 811/57 for explanation of testing period.**
 NOTE: 2 spikes were used in Phase 1. All exp. tubes labeled T1-T4 were spiked using Np Spike #61A. All tubes labeled T5-T8 were spiked using Np Spike # 61A1

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Sample ID	Mass (g) of Empty Tube	Mass (g) of Tube & Sediment	Mass (g) of Tube, Sediment & Water	Mass (g) After Equilibration	Mass (g) After Spike	Mass (g) Before LSA	pH
19W-T1	22.260101	22.36218	52.768446	52.75809	52.85903	52.77824	8.45
19W-T2	22.06505	22.16566	52.62386	52.61438	52.71509	52.67792	8.46
19W-T3	22.49247	22.59856	52.74748	52.73316	52.83266	52.76866	8.31
19W-T4	22.26600	22.37428	52.35924	52.34838	52.44937	52.36562	8.47
19WO-T1	22.25736	22.36066	52.78022	52.77100	52.87288	52.82304	8.51
19WO-T2	22.28784	22.39378	52.70806	52.69410	52.79532	52.74941	8.47
19WO-T3	22.57074	22.61672	52.90219	52.88397	52.98578	52.89887	8.38
19WO-T4	22.33973	22.44168	51.86153	51.84726	51.94864	51.88694	8.32
22W-T1	22.08015	22.18639	52.43057	52.41869	52.71933	52.61822	8.26
22W-T2	22.18468	22.28703	52.55533	52.54492	52.64576	52.58570	8.30
22W-T3	22.28137	22.38973	52.80218	52.79171	52.89272	52.78723	8.44
22W-T4	22.25470	22.35976	52.79629	52.77949	52.87989	52.78349	8.32
22WO-T1	22.08352	22.18549	52.22241	52.21152	52.31227	52.25249	8.37
22WO-T2	22.17033	22.27467	52.42570	52.41618	52.51697	52.44939	8.27
22WO-T3	22.24902	22.35205	52.52543	52.49524	52.59578	52.53635	8.29
22WO-T4	22.30173	22.40441	52.58999	52.57970	52.67990	52.59717	8.32
Date:	16 Aug 06	17 Aug 06	17 Aug 06	18 Aug 06	18 Aug 06	23 Aug 06	23 Aug 06

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Sample ID	Mass (g) of Empty Tube	Mass (g) of Tube & Sediment	Mass (g) of Tube, Sediment & Water	Mass (g) After Equilibration	Mass (g) After Spike	Mass (g) Before LSA	pH
19W-T5	22.27868	22.38191	52.81111	52.79934	52.90011	52.83933	8.49
19W-T6	22.26300	22.36925	52.73716	52.72403	52.82495	52.75445	8.46
19W-T7	22.25484	22.35791	52.79587	52.78250	52.88336	52.74794	8.40
19W-T8	22.10971	22.21593	52.66706	52.65294	52.75353	52.59984	8.37
19WO-T5	22.05785	22.16614	52.63146	52.62209	52.72285	52.25960	8.57
19WO-T6	22.48044	22.58457	53.00710	52.99226	53.09272	52.92134	8.50
19WO-T7	22.15751	22.25797	52.74028	52.72961	52.83020	52.76472	8.48
19WO-T8	22.03708	22.14025	52.49318	52.48295	52.58344	52.50403	8.39
22W-T5	22.24680	22.34991	52.87462	52.86500	52.96574	52.90907	8.49
22W-T6	22.32189	22.42854	52.75024	52.72399	52.82480	52.74688	8.46
22W-T7	22.52499	22.62819	53.09973	53.08669	53.18749	53.11103	8.40
22W-T8	22.28443	22.39277	52.70441	52.69594	52.79939	52.73513	8.56
22WO-T5	22.17235	22.27722	52.40307	52.39443	52.49448	52.40556	8.50
22WO-T6	22.29048	22.39485	52.53535	52.52590	52.62678	52.56455	8.50
22WO-T7	22.28356	22.39043	52.63368	52.62461	52.72501	52.63370	8.48
22WO-T8	22.26284	22.36850	52.57379	52.56414	52.66455	52.57337	8.63
Date:	16 Aug 06	17 Aug 06	17 Aug 06	18 Aug 06	18 Aug 06	18 Aug 06	18 Aug 06

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Step 6: Liquid Scintillation and pH Analyses of Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions—Np-237 in one type of simulated groundwater (with Ca) at a single pH

Task initially assigned to: Marla Roberts and Sandra Watson

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

Background: ~~851/50~~ 811/50 24 Aug 06 MR
Initial Entry: ~~851/51~~ 811/51 24 Aug 06 MR

Sample Info: Four types of experiments were run. Experiments based on consolidated alluvium substrate with and without calcite from NC-EWDP-19PB and NC-EWDP-22PC using 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 test phase, the experimental solution number, and the a/b duplicate designator (example - 19WO T1a). One simulated water blank solution was analyzed and labeled as, Blank wCa. Since two different spike were used, each spike was analyzed in duplicate and labeled according (Spike 61A-5, 61A-6, 61A1-23 and 61A1-24).

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.250 mL (250 μ L eppendorf pipet) of 0.02 M HNO₃ added. The mass of all of the LSA vials with acid was recorded 811/58-59.

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to LSA aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (811/54-55). The tubes containing the experimental solutions were placed in the tared aluminum centrifuge holder and mass recorded.

For experimental solutions, 0.5 mL (500 μ L eppendorf pipet) of the experimental solution was transferred into the appropriate LSA vial. For the simulated water blank, 0.5 mL (500 μ L eppendorf pipet) of simulated water was added to the LSA vial. For the neptunium spike solutions, 0.1 mL (100 μ L eppendorf pipet) of each spike was added to the appropriate LSA vial. The mass of all of the LSA vials was recorded after the addition of the appropriate sample solution (811/58-59).

Next, 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.

Temp: 20.1°C Set Pts.: $\begin{matrix} 4.00 \\ 7.01 \\ 10.03 \end{matrix}$ Slope: 98.7

24 Aug 06 MR

The pH probe was inserted directly into the the tube. The pH was recorded (811/54-55). The pH meter was challenged with one of the calibration standards after all of the sample pHs were measured. The challenge solution type and measurement was recorded.

Challenge: w/ 7.00 measured 6.99

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.

****Note from 811/53 \rightarrow to increase the CO₂ exchange, the caps of all experimental tubes were left loosely capped throughout entire experiment. On the weekdays at the beginning of each business day the caps were tightened so that each tube could be vortex for ~30 seconds. After vortexing the caps were loosened and all tubes placed back on an activated gyrator.**

Note: During the course of this experiment the supply of 19PB-3 Simulated Water w/ Ca was exhausted. This experiment used only 'batch 3', however a new batch of water was made. See 811/65 for specifics.

24 Aug 06 MR

24 Aug 06 MR

Sample ID	Mass (g) of LSA vial & acid	Mass (g) of LSA vial, acid & sample
Blank w/Ca	7.70779	8.20638
19W-T1a	7.78467	8.28127
19W-T1b	7.65371	8.15207
19W-T2a	7.58854	8.08600
19W-T2b	7.65974	8.15931
19W-T3a	7.67365	8.17061
19W-T3b	7.63433	8.13323
19W-T4a	7.63603	8.12954
19W-T4b	7.63584	8.13454
19WO-T1a	7.62471	8.12225
19WO-T1b	7.61989	8.11944
19WO-T2a	7.70774	8.20267
19WO-T2b	7.62726	8.12578
19WO-T3a	7.70608	8.20780
19WO-T3b	7.61904	8.11852
19WO-T4a	7.68076	8.17956
19WO-T4b	7.61266	8.10958
22W-T1a	7.71923	8.21239
22W-T1b	7.60859	8.09552
22W-T2a	7.70548	8.20036
22W-T2b	7.65939	8.15906
22W-T3a	7.64070	8.13798
22W-T3b	7.71999	8.22027
22W-T4a	7.60600	8.10243
22W-T4b	7.59768	8.09669
22WO-T1a	7.68916	8.18423
22WO-T1b	7.61398	8.11216
22WO-T2a	7.62626	8.12266
22WO-T2b	7.62039	8.11791
22WO-T3a	7.68795	8.18317
22WO-T3b	7.64695	8.14286
22WO-T4a	7.64497	8.13947
22WO-T4b	7.64342	8.14087
Date:	22 Aug 06	24 Aug 06

24 Aug 06 MR

Sample ID	Mass (g) of LSA vial & acid	Mass (g) of LSA vial, acid & sample
19W-T5a	7.70120	8.19760
19W-T5b	7.73543	8.23538
19W-T6a	7.70458	8.20107
19W-T6b	7.61816	8.11752
19W-T7a	7.68463	8.18109
19W-T7b	7.63384	8.13209
19W-T8a	7.57535	8.07574
19W-T8b	7.69553	8.19285
19WO-T5a	7.72135	8.21765
19WO-T5b	7.745869	8.24346
19WO-T6a	7.62367	8.12060
19WO-T6b	7.63585	8.13485
19WO-T7a	7.57144	8.06889
19WO-T7b	7.59321	8.09095
19WO-T8a	7.65982	8.15699
19WO-T8b	7.76498	8.25793
22W-T5a	7.69434	8.18934
22W-T5b	7.65847	8.15668
22W-T6a	7.64265	8.13633
22W-T6b	7.63652	8.13446
22W-T7a	7.67974	8.17690
22W-T7b	7.63503	8.13411
22W-T8a	7.63985	8.13693
22W-T8b	7.67023	8.16664
22WO-T5a	7.66642	8.16082
22WO-T5b	7.61090	8.10695
22WO-T6a	7.64358	8.14190
22WO-T6b	7.64617	8.14478
22WO-T7a	7.61226	8.10623
22WO-T7b	7.61699	8.11474
22WO-T8a	7.70587	8.20253
22WO-T8b	7.64976	8.14694
Spike 61A-5	7.64900	7.74946
Spike 61A-6	7.67218	7.77251
Spike 61A1-23	7.68036	7.78130
Spike 61A1-24	7.71367	7.81464
Date:	22 Aug 06	24 Aug 06

24 Aug 06 MR

Preparation of Cation and Anion QA Solutions for 19CAL Series 1 and 22CAL Series 1 with and without Calcite Sorption Experiment

Personnel Involved: Sandra Watson

* Note cation and anion QA solutions made for Phase 1 testing Not Series 1

Objective: To prepare QA solutions to include with 19CAL Series 1 and 22CAL Series 1 with and without Calcite cation and anion samples sent to Div 01 for analyses.

Reagents: Spex Certiprep Instrument Calibration Standard 3 (cat # CL-CAL-3, lot # 32-108AS)
Spex Certiprep IC Instrument Check Standard 2 (cat # ICMIX2-100, lot # 28-164AS)
Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)Supplies: Volumetric pipets
Volumetric flasks
HDPE bottles
Squirt bottle for water
Kimwipes**Procedure**

Added 20 mL (volumetric pipet) of cation mix (Spex CL-CAL-3) into a 1000 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 1 L HDPE bottle and labeled Cation Std, date, initials, and SN ref. The cation dilution factor is 50.

Added 10 mL (volumetric pipet) of anion mix (Spex ICMIX2-100) into a 500 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 500 mL HDPE bottle and labeled Anion Std, date, initials, and SN. The anion dilution factor is 50.

Table of Target Conc (ppm) of QA standards for samples

Species	Conc (ppm) in Spex Standard	Dilution Factor	Target Conc (ppm) of QA Standard
K	1000	50	20
Ca	1000	50	20
Na	1000	50	20
Mg	1000	50	20
HPO ₄	600	50	12
Br	400	50	8
NO ₃	400	50	8
SO ₄	400	50	8
Cl	200	50	4
F	100	50	2

24 Aug 06 MR

24 Aug 06 MR

Sampling of Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions for Div 01 Cation and Anion Analyses

Task initially assigned to: Marla Roberts and Sandra Watson

Objective:

To characterize Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions water chemistry at the conclusion of the sorption experiments by analyses of cations and anions.

Initial Entry: ~~851/51~~ 811/51 24 Aug 06 MR**Samples and Reagents:****NOTE:** Not all exp. solns are being sent for chemistry. Two samples from each set of 4 exp. soln. are being sent to Div. 01 for analyses. See legend on 811/63 for specific exp. soln. IDs.

Experimental solutions: 2 from 19W T1-4, 2 from 19WO T1-4, 2 from 22W T1-4, 2 from 22WO T1-4, 2 from 19W T5-8, 2 from 19WO T5-8, 2 from 22W T5-8, 2 from 22WO T5-8 (811/54-55).

Total exp. solns sent: 16

QA solutions: cation and anion QA std (811/60)

Equipment:100 mL pp beakers
Filters - Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 μ m pore size
10mL syringe, BD# 309604-04
30mL pp bottles
60mL pp bottles
Eppendorff pipette and tips**Note:**

The cation and anion analyses samples were collected after the pH analyses was conducted (811/54-55)

Background:

Experimental solution were to be characterized by cation and anion analyses. Due to limited volume of each experimental solution (about 30mL), anion sample were generated by adding Type 1 water to small aliquots removed from the cation samples. The anion samples Div 01 analyzes are diluted and do not directly represent the concentration in the experimental solutions. Dilution factor will be determined by mass not volume.

Procedure:

A 10cc syringe was used to collect as much sample as possible from the centrifuge tube. 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. The filter was set aside in a contained location and the syringe was again inserted into the centrifuge tube and the experimental solution was withdrawn. The filter was again placed on the syringe and the sample was dispensed into the labeled pp bottle. The filter was then properly discarded with in the solid radioactive waste bin. This process was repeated for each of the experimental solutions.

24 Aug 06 MR

The first step in generating the anion samples was recording the mass of appropriately labeled 30 mL pp bottles (811/63). Using an eppendorff pipet, about a 5 mL aliquot of each cation sample was transferred to the appropriate anion bottle. The new mass of each anion bottle was measure and recorded. Approximately, 15mL of Type 1 water was then transferred to each anion bottle. The new mass of each anion bottle was measured and recorded. The dilution factor was about four and determined by mass for each sample.

Each cation sample was preserved by adding 70 μ L (eppendorff pipet) of 1:1 nitric acid.

A total 5 QA samples were generated: 2 duplicate anion, 2 duplicate cation and 1 anion sample labeled as a cation (1922-4C) and preserved with 70 μ L (eppendorff pipet) of 1:1 nitric acid. This QA was included to test the sensitive of the Div 01 equipment at detecting phosphorus and phosphate. Aliquots of cation and anion were decanted into appropriately labeled 30mL pp bottles. See 811/63 for legend.

The following describes the Div 01 sample ID scheme. The first number (19 or 22) represents the well which the solid material was collected from. The "W or WO" indicate whether the solid material was 'with or without' calcite. The "T" signifies this is a test experiment. All samples sent were numbered 1-16 with a "C" or "A" for either cation or anion analyses.

In addition, sample ID 1922-1C/1A was included for the analyses of a new batch of water (19PB-4 Simulated Groundwater with Calcium). See 811/63 *24 Aug 06 MR*. Also, IDs 1922-2C, 1922-3C, 1922-4C, 1922-5A and 1922-6A were used for the cation and anion QA samples respectively. The following is a complete legend relating CNWRA IDs with the Div 01 IDs.

24 Aug 06 MR

24 Aug 06 MR

CNWRA Sample ID	Div 01 Sample ID-Cations	Div 01 Sample ID-Anions
19W-T1	19WT-1C	19WT-1A
19W-T2	19WT-2C	19WT-2A
19W-T6	19WT-3C	19WT-3A
19W-T7	19WT-4C	19WT-4A
19WO-T2	19WOT-5C	19WOT-5A
19WO-T3	19WOT-6C	19WOT-6A
19WO-T6	19WOT-7C	19WOT-7A
19WO-T7	19WOT-8C	19WOT-8A
22W-T2	22WT-9C	22WT-9A
22W-T3	22WT-10C	22WT-10A
22W-T6	22WT-11C	22WT-11A
22W-T7	22WT-12C	22WT-12A
22WO-T3	22WOT-13C	22WOT-13A
22WO-T4	22WOT-14C	22WOT-14A
22WO-T6	22WOT-15C	22WOT-15A
22WO-T7	22WOT-16C	22WOT-16A
19PB-4 Simulated Groundwater with Calcium	1922-1C	1922-1A
QA-cation1	1922-2C	na
QA-cation2	1922-3C	na
QA-cation3 <i>anion 3</i>	1922-4C	na
QA-anion1	na	1922-5A
QA-anion2	na	1922-6A

24 Aug 06 MR

24 Aug 06 MR

Div 01 Sample ID- Anions	Mass (g) Empty Bottle	Mass (g) bottle & exp. soln.	Mass (g) bottle, exp. soln. & Type 1 water
19WT-1A	8.45066	13.33959	28.69003
19WT-2A	8.34778	13.41239	28.83442
19WT-3A	8.44062	13.57810	29.11861
19WT-4A	8.39433	13.51543	28.83877
19WOT-5A	8.34912	13.47439	28.76716
19WOT-6A	8.43936	13.50773	28.82150
19WOT-7A	8.34755	13.40319	28.80042
19WOT-8A	8.36883	13.49071	28.79906
22WT-9A	8.39897	13.52197	28.78719
22WT-10A	8.45683	13.56149	28.85364
22WT-11A	8.41397	13.48461	28.75202
22WT-12A	8.39674	13.24900	28.60859
22WOT-13A	8.47533	13.58414	28.87450
22WOT-14A	8.34898	13.46073	28.69468
22WOT-15A	8.36913	13.42579	28.67553
22WOT-16A	8.39221	13.42575	28.65349

24 Aug 06 MR

24 Aug 06 MR

Preparation of 19PB Shallow String Simulated Groundwater with Calcium (4th Batch)**Task Assigned To:** Marla Roberts and Sandra Watson**Objective:** To prepare simulated groundwater (from stock solutions) that can be used for sorption experiments.**Background:** 19PB Shallow String Simulated Groundwater with Calcium is created by combining Stock Solutions A, B and C. To achieving the appropriate concentrations as described on 742/27-28, a 40 fold dilution factor is used. Meaning, 25 mL of each stock solution is required per liter of simulated water. The goal is to prepare 4L of 19PB Simulated Water with Calcium. Therefore 100mL of each stock solution is necessary to create 19PB Shallow String Simulated Groundwater with Calcium.**Equipment:**

2 L volumetric flask	Glass gas dispersion tube
1 L volumetric flask	Moisture bottle
100 mL volumetric flask	Pump
4 L HDPE carboy	
100 mL beaker	
50 mL beaker	
Funnel	
Disposable pipet	

Reagents:

Stock Solution A (742/29)
 Stock Solution B (742/31)
 Stock Solution C (742/33)
 Type 1 water

Procedure: 2 L (via 2L volumetric flask) of Type 1 water was added to the 4L carboy. 100mL (volumetric flask with funnel) of Stock Solution A was collected. Taken to mark using disposable pipet and 50 mL beaker (filled with Stock Solution A). The 100 mL was transferred to 4L carboy. Following the same steps, 100mL of Stock Solution B and Stock Solution C were added to the 4L carboy. After Stock Solution A, B and C was added to the 4L carboy, the total volume of the carboy was 2300mL. The carboy was filled with an additional 1700 mL of Type 1 water. The carboy was swirled by hand. The carboy was labeled 19PB-4 Simulated Groundwater without Calcium. The '4' is to indicate this is the fourth batch of 19PB Simulated Groundwater with Calcium (see background 742/35). Finally, the carboy was set-up with an aeration device. A glass gas dispersion tube was inserted into the 4L carboy with a 'moisture bottle' between the carboy and the pump. The carboy opening is covered with a cut-out pieced of bench paper.

25 Aug 06 MR

Delivery of 19W and 19WO & 22W and 22WO Phase I testing of pH to Div 01 for cation and anion analyses

Initial entry: 8/11/51

Total Samples: 39

16 - cation
4 19W, 4 19WO, 4 22W 4 22WO

16 - anion
4 19W, 4 19WO, ~~22~~ 22W, 4-22WO
25 Aug 06 MR

5 - QA
3 cation
2 anion

2 - Simulated groundwater (9PB-4)
Sample and QA collection: 8/11/61-62
2 Nov 06 MR

Sample legend: 8/11/63

QA target concentrations: 8/11/60

Chain of custody on following pages:
chain

25 Aug 06 MR

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Relinquished by (Print/Signature)	Received by (Print/Signature)	Date	Time
19WT-1C	8/11/06		2		1	Analysis of water by ICD one must clean	Marla Roberts <i>Marla Roberts</i>	25 Aug 06	1100
19WT-2C									
19WT-3C									
19WT-4C									
19WOT-5C									
19WOT-6C									
19WOT-7C									
19WOT-8C									
22WT-9C									
22WT-10C									

Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: <u>4 weeks</u>	SwRI Contact <u>Mike Dammann</u>	REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)
SwRI Project: <u>200608 01 242</u>	Received by SwRI Lab: <i>[Signature]</i>	Date: <u>8/15/06</u> Time: <u>1100</u>
Relinquished by (Print/Signature): <i>[Signature]</i>	Received by (Print/Signature): <i>[Signature]</i>	Date: _____ Time: _____
Relinquished by (Print/Signature): _____	Received by (Print/Signature): _____	Date: _____ Time: _____
Relinquished by (Print/Signature): _____	Received by (Print/Signature): _____	Date: _____ Time: _____
Relinquished by (Print/Signature): _____	Received by (Print/Signature): _____	Date: _____ Time: _____

Bradley Werling
8-57
Division 00

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 Rinseate
ES - Environmental Sample
FB - Field Blank
FD - Field Duplicate
MS - Matrix Spike
MSD - Matrix Spike Dup
TB - Trip Blank

Temp: 22.0°C Therm #: 021
Comments: RADIOACTIVE
AP 237 @ 8.5 x 10⁻⁷ Mgr 1.8 x 10⁻⁴ uCi/mL
SN 8/11/06

25 Aug 06 MR

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 weeks

Client: Bradley Werling
B-57
DIVISION 20

Shipper Name/Address: Bradley Werling
B-57
DIVISION 20

Client Purchase Order/Other ID: _____ Site/Zone ID: _____

Analyses Requested: _____

SwRI Contact: Mike Dammann

REMARKS
 Preservation
 a = HCl to pH <2
 b = HNO₃ to pH <2
 c = H₂SO₄ to pH <2
 d = NaOH to pH >12
 e = Cool (4°C±2°C)
 f = Other (specify)

POP IS applies

POC - B Werling
 6565

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Relinquished by (Print/Signature)	Date	Time	SwRI Project#
19 WT-11C	8/24/06		3		1	Relinquished by (Print/Signature) <u>Marla Roberts</u>	25 Aug 06	1100	2006002.01.242
19 WT-12C						Received by (Print/Signature)			
19 WT-13C						Relinquished by (Print/Signature)			
19 WT-14C						Received by (Print/Signature)			
19 WT-15C						Relinquished by (Print/Signature)			
19 WT-16C						Received by (Print/Signature)			
19 WT-17C						Relinquished by (Print/Signature)			
19 WT-18C						Received by (Print/Signature)			
19 WT-19C						Relinquished by (Print/Signature)			
19 WT-20C						Received by (Print/Signature)			
19 WT-21C						Relinquished by (Print/Signature)			
19 WT-22C						Received by (Print/Signature)			
19 WT-23C						Relinquished by (Print/Signature)			
19 WT-24C						Received by (Print/Signature)			

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 @ 8.5x10⁻⁷ M or 1.8x10⁻⁴ MPM
SU 8/11/06

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

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

Page ___ of ___

25 Aug 06 MR

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 weeks

Client: B. Werling
B.57
DIV 20

Shipper Name/Address: B. Werling
B.57
DIV 20

Client Purchase Order/Other ID: _____ Site/Zone ID: _____

Analyses Requested: _____

SwRI Contact: Mike Dammann

REMARKS
 Preservation
 a = HCl to pH <2
 b = HNO₃ to pH <2
 c = H₂SO₄ to pH <2
 d = NaOH to pH >12
 e = Cool (4°C±2°C)
 f = Other (specify)

POP IS applies

POC - B Werling
 6565

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Relinquished by (Print/Signature)	Date	Time	SwRI Project#
19 WT-1A	8/24/06		3		1	Relinquished by (Print/Signature) <u>Marla Roberts</u>	25 Aug 06	1100	2006002.01.242
19 WT-2A						Received by (Print/Signature)			
19 WT-3A						Relinquished by (Print/Signature)			
19 WT-4A						Received by (Print/Signature)			
19 WT-5A						Relinquished by (Print/Signature)			
19 WT-6A						Received by (Print/Signature)			
19 WT-7A						Relinquished by (Print/Signature)			
19 WT-8A						Received by (Print/Signature)			
22 WT-9A						Relinquished by (Print/Signature)			
22 WT-10A						Received by (Print/Signature)			

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 @ 8.5x10⁻⁷ M or 1.8x10⁻⁴ MPM
SU 8/11/06

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

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

Page ___ of ___

25 Aug 06 MR

Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks		Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
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	
Client Name/Address B Werling B.57		Site/Zone ID	
Client DIV 20		Analyses Requested	
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type
22WOT-11A	8/24/06		W
22WOT-13A			
22WOT-14A			
22WOT-15A			
22WOT-16A			
1922-1A			
1922-5A			
1922-6A			
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 E 8.5 x 10 ⁻⁷ M or 1.4 x 10 ⁻⁴ µCi/mL	
SWRI Project#		Received by SwRI Lab (Signature)	
20-06000-01-242		[Signature]	
Date	Time	Date	Time
25 Aug 06	1100		
Relinquished by (Print/Signature)		Relinquished by (Print/Signature)	
[Signature]		[Signature]	
Received by (Print/Signature)		Received by (Print/Signature)	
[Signature]		[Signature]	
Relinquished by (Print/Signature)		Relinquished by (Print/Signature)	
[Signature]		[Signature]	
Received by (Print/Signature)		Received by (Print/Signature)	
[Signature]		[Signature]	
Relinquished by (Print/Signature)		Relinquished by (Print/Signature)	
[Signature]		[Signature]	
Remarks		Remarks	
a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)		POP 15 Applies	
SwRI Contact		SwRI Contact	
Mike Dammann		Mike Dammann	
Remarks		Remarks	
POP 15 Applies		POP 15 Applies	
Rec. - B Werling		Rec. - B Werling	
105.605		105.605	

29 Aug 2006 MR

Step 7: Liquid Scintillation Analyses Results for Phase 1 Testing of 19PB and 22PC with and without Calcite Experimental Solutions

Task initially assigned to: Marla Roberts

Objective

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

Initial Entry 811/51

LSA sample preparation 811/56-57

Sample Info

Blank (1 sample from simulated water with calcium): w Ca 811/56-57

Neptunium spikes (4 samples) - Spike 61A-5, Spike 61A-6, Spike 61A1-23 and Spike 61A1-24 (811/56-57)

Four experiments derived from 4 solids and 1 type of simulated water
 19W T 1-8 (16 samples, with calcite) using simulated water with calcium
 19WO T 1-8 (16 samples, without calcite) using simulated water with calcium
 22W T 1-8 (16 samples, with calcite) using simulated water with calcium
 22WO T 1-8 (16 samples, without calcite) using simulated water with calcium

Total number of LSA samples: 69

Results

Printout of LSA analyses following:

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8/23/06 2:26:51 PM

QuantaSmart (TM) - 1.31 - Serial# 405314

Page # 1

SNC Protocol

Calibration Information

Software Version IC: 2.11
 Software Version EC: 1.31
 Instrument Model: Tri-Carb 3100TR
 Instrument Serial Number: 405314
 3H Chi Square: 16.96 Date Processed: 8/23/06 2:26:50 PM
 14C Chi Square: 14.38 Date Processed: 8/23/06 2:26:50 PM
 3H E²/B (1-18.6 keV): 284.21 Date Processed: 8/23/06 2:26:50 PM
 14C E²/B (4-156 keV): 563.73 Date Processed: 8/23/06 2:26:50 PM
 3H Efficiency (0-18.6 keV): 64.99 Date Processed: 8/23/06 2:26:50 PM
 14C Efficiency (0-156 keV): 96.76 Date Processed: 8/23/06 2:26:50 PM
 IPA Background Date Processed: 8/23/06 2:26:50 PM
 3H Background CPM (0-18.6 keV): 14.90 Date Processed: 8/23/06 2:26:50 PM
 14C Background CPM (0-156 keV): 21.92 Date Processed: 8/23/06 2:26:50 PM
 3H Calibration DPM: 285000
 3H Reference Date: 10/29/99
 14C Calibration DPM: 134100

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QuantaSmart (TM) - 1.31 - Serial# 405314

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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\20060823_1426.results
 Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.049
 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): 200.00
 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-

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: 16.96 Date Processed: 8/23/06 2:26:50 PM
 14C Chi Square: 14.38 Date Processed: 8/23/06 2:26:50 PM
 3H E²/B (1-18.6 keV): 284.21 Date Processed: 8/23/06 2:26:50 PM
 14C E²/B (4-156 keV): 563.73 Date Processed: 8/23/06 2:26:50 PM
 3H Efficiency (0-18.6 keV): 64.99 Date Processed: 8/23/06 2:26:50 PM
 14C Efficiency (0-156 keV): 96.76 Date Processed: 8/23/06 2:26:50 PM
 IPA Background Date Processed: 8/23/06 2:26:50 PM

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3H Background CPM (0-18.6 keV): 14.90 Date Processed: 8/23/06 2:26:50 PM
14C Background CPM (0-156 keV): 21.92 Date Processed: 8/23/06 2:26:50 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100

Cycle 1 Results

Table with columns: S# Count Time, CPMA, A:2S%, CPMB, B:2S%, CPMA, alpha2S%, SIS. Includes rows for Cycle 1 Results, Missing vial 2, Missing vial 11, Missing vial 20, Missing vial 29, Missing vial 38, Missing vial 47, and various sample measurements with handwritten annotations like 'Blank w/ka', '19W T1a', '22W T1a', etc.

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Table with columns: S#, CPM, A:2S%, B:2S%, CPMA, alpha2S%, SIS, and additional columns for counts and percentages. Includes rows for samples 53-79 and various handwritten annotations like '19W T7b', '22W T5a', 'Spike L01A5', etc.

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QuantaSmart (TM) - 1.31 - Serial# 405314

Page # 1

SNC Protocol

Calibration Information

Software Version IC: 2.11

Software Version EC: 1.31

Instrument Model: Tri-Carb 3100TR

Instrument Serial Number: 405314

3H Chi Square: 23.60 Date Processed: 8/26/06 5:56:29 AM

14C Chi Square: 20.33 Date Processed: 8/26/06 5:56:29 AM

3H E²/B (1-18.6 keV): 289.12 Date Processed: 8/26/06 5:56:29 AM

14C E²/B (4-156 keV): 552.85 Date Processed: 8/26/06 5:56:29 AM

3H Efficiency (0-18.6 keV): 65.10 Date Processed: 8/26/06 5:56:29 AM

14C Efficiency (0-156 keV): 96.49 Date Processed: 8/26/06 5:56:29 AM

IPA Background Date Processed: 8/26/06 5:56:29 AM

3H Background CPM (0-18.6 keV): 14.68 Date Processed: 8/26/06 5:56:29 AM

14C Background CPM (0-156 keV): 21.30 Date Processed: 8/26/06 5:56:29 AM

3H Calibration DPM: 285000

3H Reference Date: 10/29/99

14C Calibration DPM: 134100

29 Aug 06 MR

to Sept 06
to A 6 Sept 06 MR

Continued Testing for 19PB and 22PC with and without Calcite Sorption Experiments

Background: To better understand the interactions between the sediments of 19PB and 22PC with reference solutions, pH adjustments and spikes used in the 19CAL and 22CAL Series 1 and 2 sorption experiments small scale sorption experiments will be conducted to test individual aspects of the experiment process. These small scale experiments will be identified as phases (i.e. Phase 1, Phase 2 etc.). Each Phase will have a specific aspect of the experimental process tested.

The background sections of each of the initial entries will identify which phase of experiments is being conducted as well as what alterations to methodology are taking place.

Phase 1: 811/50

Objective: To investigate Np sorption on a NC-EWDP-19PB 0305 Consolidated Alluvium and NC-EWDP-22PC 1005 Consolidated Alluvium (with and without calcite) using simulated water (with calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB

- Samples
- 19PB-4 Simulated Groundwater with Ca - 811/65
 - NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
 - NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
 - NC-EWDP-22PC-1005-Sonic Core Composite - 742/8
 - NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Personnel initially assigned to task: Marla Roberts and Sandra Watson

Phase 2: Adjusting the pH of the reference solution initially. Each experimental solution will have no additional pH adjust. The only adjustment made is to the starting groundwater reference solution (~400mL of 19PB-4 simulated groundwater with Ca)

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Initial Entry - 19PB and 22PC with and without Calcite Sorption Experiments - Using Np-237 on Consolidated Alluvium with 19PB Simulated Waters with Calcium
Phase 2: Adjusting Initial Reference Solution pH

Personnel initially assigned to task: Marla Roberts and Sandra Watson

Objective: To investigate Np sorption on a NC-EWDP-19PB 0305 Consolidated Alluvium and NC-EWDP-22PC 1005 Consolidated Alluvium (with and without calcite) using simulated water (with calcium) based on the chemistry from water collected from the shallow string of drillhole NC-EWDP-19PB at a single pH.

Background: This is the second of a series of tests (phases) on the 19PB and 22PC with and without calcite sediments. This particular test (Phase 2) is examining the repeatability of sorption results given an adjusted initial reference solution pH. In this case no pH adjustments are made to the exp. solns, the only adjustments made were to the reference solution prior to beginning the experiment.

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-4 Simulated Groundwater with Ca - 811/65
- NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
- NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
- NC-EWDP-22PC-1005-Sonic Core Composite - 742/8
- NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

Reagents

- Neptunium Spike # 61A (369/95) - 100ppm Np-237 at pH of
- 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)
- 0.4 M HNO_3 (696/195) for pH adjustment
- 0.02 M HNO_3 (SN 696/29-30) for LSA matrix
- Type 1 water (Barnstead/Thermolyne model D11901 sn1190010979691)

Equipment:

- Orion meter 920A (sn 039518)
- Thermo Orion combination electrode 8103BN (ID 3I)
- New Brunswick Scientific Gyrotory shaker model G33 (sn 290127990)
- Fisher Marathon 21K centrifuge model D7209 (sn 15930025)
- Mettler Toledo XP205DR (sn 1126461033)
- Packard Tricard 3100TR Liquid Scintillation Analyzer model B2505 (sn 405314)
- Centrifuge Tube Holder (Aluminum)

Supplies:

Pipettes and tips	Stirs bars
Repipettor for transfer of scintillation cocktail	Kimwipes
Polycarbonate centrifuge tubes with caps (50 ml capacity)	Squirt bottle for water
7 ml scintillation vials	Plasticware and glassware as needed
Weighing paper	burette

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Experimental Steps for Phase 2: Adjusting Initial Reference Solution pH of 19PB and 22PC with and without Calcite Sorption Experiments

Personnel involved: Marla Roberts and Sandra Watson

Background: See 811/77
 Initial Entry: 811/78

Note: All data (masses, etc.) recorded on 811/81

Step 1: Adjusting pH of Simulated Waters used in Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions

Objective: To adjust the pH of reference solution (simulated water) used to create the experimental solutions.

Procedure:

400mL of 19PB-4 Simulated Groundwater with Ca (811/65) was decanted into a glass beaker. Using a burette 0.4 M HNO_3 (696/195) was incrementally added. The target for the reference solution was between 7.0 and 7.5 pH. Initially acid was added by .5 mL increments. However, after each addition the solution was allowed to equilibrate for 1 hour. The pH was re-measured and adjustments made to the amount of acid added. A total of 1.65mL of acid was added.

Prior to measuring pH- 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 set points and slope were recorded. The acceptable range for the slope is 92-102% from page 5 of Orion Ross pH Electrode Instruction Manual, 2003.

Temperature: 20.6°C
 Set Points: 4.00, 7.01, 10.06
 Slope: 97.8
 pH Measured: 7.43

Note: pH meter unable to calibrate for pH buffer 4.00. pH buffer 4 was manual entered into meter.

Challenge: w/ 7. measured: 7.01

Step 2: Addition of Solids to Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption Experiments

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

Sample info

- NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
- NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
- NC-EWDP-22PC-1005-Sonic Core Composite - 742/8
- NC-EWDP-22Pc-1005-Sonic Core Composite w/o Calcite- 742/25

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Procedure

Four sets of four polycarbonate centrifuge tubes were labeled as follows: 19W-T9-11, 19WO T9-11, 22W T9-11 and 22WO T9-11. The mass of each tube (with cap) was measured and recorded on 8/11/81. An individual experimental tube was uncapped and a funnel made from weighing paper was inserted in the top. Prior to aliquot removal the jar containing the solid was gently rolled to ensure an unbiased aliquot. Approximately 0.1 g of solid was transferred to a piece of tared weighing paper on the balance. The solid was transferred into the appropriate experimental tube 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 8/11/81

The centrifuge tubes labeled with a "W" indicate the solid contains calcite and the aliquot was removed from a sampling containing calcite (NC-EWDP-19PB-0305-Sonic Core Composite and NC-EWDP-22PC-1005-Sonic Core Composite). The centrifuge tubes labeled with a "WO" indicate the solid does not contain calcite and was removed from a treated sample (NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite and NC-EWDP-22PC-1005-Sonic Core Composite). See 742/25 for further information about calcite removal. The "T" was used to signify this is a test. "19" and "22" indicate well numbers for the sediment.

Step 3: Addition of Simulated Water Solution to Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption Experiment

Objective: To add simulated water to centrifuge tubes as preparation for sorption experiment.

Sample Info: 19PB-4 Simulated Groundwater with Calcium 8/11/65

Procedure

About 30mL (10mL pipet) of the pH adjusted simulated water was added to the experimental pc tubes. The mass was recorded on 8/11/81

Step 5: Addition of Neptunium Spike to Phase 1 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions

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

Reagent: Np Spike # 61A (369/95)

Procedure

Added 100µL (100µL eppendorf pipet) to each experimental solution and recorded the mass of the experimental solution on 8/11/81-55.

All exp. soln. tubes remained loosely capped throughout entire experiment. Each morning all tubes vortexed ~30 seconds, then placed back on activated gyrator loosely capped.

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Sample ID	Mass (g) of Empty Tube	Mass (g) of Tube & Sediment	Mass (g) of Tube, Sediment & Water	Mass (g) After Equilibration	Mass (g) After Spike	Mass (g) Before LSA	pH
19W-T9	22.53796	22.64120	52.96736		53.06666	52.88168	7.7
19W-T10	22.32893	22.43141	52.77414		52.87454	52.77954	7.69
19W-T11	22.20287	22.30378	52.74407		52.84399	52.66730	7.79
19WO-T9	22.55879	22.66219	53.04652		53.14689	52.99280	7.85
19WO-T10	22.16011	22.26352	52.72480		52.82543	52.75345	7.86
19WO-T11	22.10726	22.21057	52.71343		52.81371	52.73481	7.79
22W-T9	22.07044	22.17454	52.98721		53.08740	52.95875	7.56
22W-T10	22.04122	22.14476	52.94794		53.04812	52.88872	7.58
22W-T11	22.31998	22.41008	52.63495		52.73539	52.63660	7.60
22WO-T9	22.53079	22.63081	52.62079		52.72111	52.62186	7.84
22WO-T10	22.31068	22.41438	52.56650		52.66684	52.58291	7.80
22WO-T11	22.08210	22.18677	52.28233		52.38101	52.32099	7.84
Date:	8 Sept 06	8 Sept 06	8 Sept 06	8 Sept 06	8 Sept 06	13 Sept 06	13 Sept 06

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Step 6: Liquid Scintillation and pH Analyses of Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions- Np-237 in one type of simulated groundwater (with Ca) with an adjusted pH

Task initially assigned to: Marla Roberts and Sandra Watson

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

Background: 811/77

Initial Entry: ~~851/78~~ 811/78 13 Dec 06 MR

Sample Info: Four types of experiments were run. Experiments based on consolidated alluvium substrate with and without calcite from NC-EWDP-19PB and NC-EWDP-22PC using 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 (Spike 61A-7 & Spike 61A-8). Experimental solutions were analyzed in duplicate and labeled to identify the sorption solid phase and test phase, the experimental solution number, and the a/b duplicate designator (example - 19WO T9a). One simulated water blank solution was analyzed and labeled as, Blank wCa.

Each LSA vial (experimental solution, water blank, and neptunium spikes) had 0.250 mL (250 μ L eppendorf pipet) of 0.02 M HNO₃ added. The mass of all of the LSA vials with acid was recorded 811/84.

The experimental solutions were centrifuged for about 5 minutes at 9200 RPM prior to LSA aliquot transfer. The mass of the experimental solutions was recorded before removing the LSA aliquots (811/81). The tubes containing the experimental solutions were placed in the tared aluminum centrifuge holder and mass recorded.

For experimental solutions, 0.5 mL (500 μ L eppendorf pipet) of the experimental solution was transferred into the appropriate LSA vial. For the simulated water blank, 0.5 mL (500 μ L eppendorf pipet) of simulated water was added to the LSA vial. For the neptunium spike solutions, 0.1 mL (100 μ L eppendorf pipet) of the spike was added to the appropriate LSA vial. The mass of all of the LSA vials was recorded after the addition of the appropriate sample solution (811/84).

Next, 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.

Temp: 20.6°C Set Pts.: $\begin{matrix} 4.00 \\ 7.01 \\ 10.08 \end{matrix}$ Slope: 97.3

*See note 811/79, step 1 about pH probe

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The pH probe was inserted directly into the the tube. The pH was recorded (811/81). The pH meter was challenged with one of the calibration standards after all of the sample pHs were measured. The challenge solution type and measurement was recorded.

Challenge:

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.

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Sample ID	Mass (g) of LSA vial & acid	Mass (g) of LSA vial, acid & sample
Blank w/Ca	7.57847	8.07997
19W-T9a	7.63858	8.13569
19W-T9b	7.72006	8.22350
19W-T10a	7.61898	8.11515
19W-T10b	7.69187	8.19331
19W-T11a	7.63167	8.12742
19W-T11b	7.63420	8.13525
19WO-T9a	7.68012	8.17747
19WO-T9b	7.66419	8.16655
19WO-T10a	7.69540	8.19220
19WO-T10b	7.65066	8.15355
19WO-T11a	7.66617	8.16416
19WO-T11b	7.61180	8.11321
22W-T9a	7.75481	8.25337
22W-T9b	7.70654	8.20708
22W-T10a	7.61881	8.11722
22W-T10b	7.62327	8.12653
22W-T11a	7.62013	8.11847
22W-T11b	7.65241	8.15543
22WO-T9a	7.63217	8.13114
22WO-T9b	7.65075	8.15230
22WO-T10a	7.60264	8.10125
22WO-T10b	7.63431	8.13777
22WO-T11a	7.64192	8.14174
22WO-T11b	7.68526	8.18681
Spike 61A-7	7.68783	7.77914
Spike 61A-8	7.64772	7.74781
Date:	13 Sept 06	13 Sept 06

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**Step 7: Sampling of Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption
Experimental Solutions for Div 01 Cation and Anion Analyses**

Task initially assigned to: Marla Roberts and Sandra Watson

Objective:

To characterize Phase 2 Testing of 19PB and 22PC with and without Calcite Sorption Experimental Solutions water chemistry at the conclusion of the sorption experiments by analyses of cations and anions.

Initial Entry: 811/78

Samples and Reagents:

NOTE: Not all exp. solns are being sent for chemistry. Two samples from each set of 3 exp. soln. are being sent to Div. 01 for analyses. See legend on 811/87 for specific exp. soln. IDs.

Experimental solutions: 2 from 19W T9-11, 2 from 19WO T9-11, 2 from 22W T9-11, and 2 from 22WO T9-11,

Total exp. solns sent: 28

Experimental Solutions: 16

QA solutions: 2 cation and 2 anion QA std (811/60)

UTSA samples: 8 (see note at end of step 7 for further information)

Equipment:

100 mL pp beakers

Filters - Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 μ m pore size

10mL syringe, BD# 309604-04

30mL pp bottles

Eppendorff pipette and tips

Note:

The cation and anion analyses samples were collected after the pH analyses was conducted (811/81)

Background:

Experimental solution were to be characterized by cation and anion analyses. Due to limited volume of each experimental solution (about 30mL), anion sample were generated by adding Type 1 water to small aliquots removed from the cation samples. The anion samples Div 01 analyzes are diluted and do not directly represent the concentration in the experimental solutions. Dilution factor will be determined by mass not volume.

Procedure:

A 10cc syringe was used to collect as much sample as possible from the centrifuge tube. 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. The filter was set aside in a contained location and the syringe was again inserted into the centrifuge tube and the experimental solution was withdrawn. The filter was again placed on the syringe and the sample was

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dispensed into the labeled pp bottle. The filter was then properly discarded with in the solid radioactive waste bin. This process was repeated for each of the experimental solutions.

The first step in generating the anion samples was recording the mass of appropriately labeled 30 mL pp bottles (811/88). Using an eppendorff pipet, about a 5 mL aliquot of each cation sample was transferred to the appropriate anion bottle. The new mass of each anion bottle was measure and recorded. Approximately, 15mL of Type 1 water was then transferred to each anion bottle. The new mass of each anion bottle was measured and recorded. The dilution factor was about four and determined by mass for each sample.

Each cation sample was preserved by adding 70µL (eppendorff pipet) of 1:1 nitric acid.

A total of 4 QA samples were generated: 2 duplicate anion, 2 duplicate cation. Aliquots of cation and anion were decanted into appropriately labeled 30mL pp bottles. See 811/87 for legend.

The following describes the Div 01 sample ID scheme. The first number (19 or 22) represents the well which the solid material was collected from. The "W or WO" indicate whether the solid material was 'with or without' calcite. The "T" signifies this is a test experiment. All samples sent were numbered 9 & 10 to correspond to the experimental solution the aliquot came from, with a "C" or "A" for either cation or anion analyses.

IDs 1922-1C, 1922-2C, 1922-3C, and 1922-4A were used for the cation and anion QA samples respectively. The following is a complete legend relating CNWRA IDs with the Div 01 IDs.

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CNWRA Sample ID	Div 01 Sample ID-Cations	Div 01 Sample ID-Anions
19W-T9	19WT-9C	19WT-9A
19W-T10	19WT-10C	19WT-10A
19WO-T9	19WOT-9C	19WOT-9A
19WO-T10	19WOT-10C	19WOT-10A
22W-T9	22WT-9C	22WT-9A
22W-T10	22WT-10C	22WT-10A
22WO-T9	22WOT-9C	22WOT-9A
22WO-T10	22WOT-10C	22WOT-10A
QA-cation1	1922-1C	na
QA-cation2	1922-2C	na
QA-anion1	na	1922-3A
QA-anion2	na	1922-4A

Included in this delivery to Div 01. 8 samples were included from UTSA. The 8 samples were ground water samples from an unknown location. 4 samples were sent for cation analyses. These 4 samples were preserved w/ 90% of 1:1 nitric acid. The remaining 4 samples were sent for analyses. The sample IDs are as follows, see CoC 811/89.

<u>cation</u>	<u>anion</u>
AD-C	AD-A
LO-C	LO-C ^{13 Dec 04 MR} LO-A
TM-C	TM-C ^{13 Dec 04 MR} TM-A
HT-C	HT-C ^{13 Dec 04 MR} HT-A

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Div 01 Sample ID-Anions	Mass (g) Empty Bottle	Mass (g) bottle & exp. soln.	Mass (g) bottle, exp. soln. & Type 1 water
19WT-9A	8.33708	13.27239	28.522252
19WT-10A	8.17996	13.28418	28.54693
19WOT-9A	8.21071	13.31655	28.57808
19WOT-10A	8.19172	13.02146	28.29223
22WT-9A	8.19390	13.29769	28.56176
22WT-10A	8.31374	13.40311	28.60829
22WOT-9A	8.16607	13.23915	28.53091
22WOT-10A	8.24694	13.20025	28.46150

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Delivery of Phase 2 19W, 19WO, 22W and 22WO experimental solutions to Div 01 for cation and anion analyses

Initial Entry: 8/11/78

Total Samples: 28

8 - cation: 2-19W, 2-19WO, 2-22W, 2-22WO
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8 - anion: 2-19W, 2-19WO, 2-22W, 2-22WO

4 - QAs
 2 cation
 2 anion

8 - UTSA samples: 8/11/86

Sample + QA collection: 8/11/85

Sample legend: 8/11/87

QA target conc: 8/11/86 14 Sept 2006 MR

Chain of custody on following pages:

14 Sept 06 MR

14 Sept 2006 MR

Shipper Name/Address Bradley Werling B-57 Div. 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input checked="" type="checkbox"/> 3 Weeks Other: <u>4 weeks</u>	
Client Div. 20		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact Mike Dammann	
Sample ID 19WT-9C 19WT-10C 19WOT-9C 19WOT-10C 22WT-9C 22WT-10C 22WOT-9C 22WOT-10C 1922-1C 1922-2C		Sample Collection Date (mm/dd/yy) 9/14/06		Sample Collection Time Matrix Type Sample Type # of Containers		REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) PAP 15 applies *Min. 1ppm for potassium POC-B. Werling X 6565	
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°C Therm #: 27 KE Comments: RADIOACTIVE NP 237 @ 8.5x10 ⁻⁷ M or 1.4x10 ⁻⁴ µCi/ml SN 8/11/88		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		SWRI Project#: 20.06002.01.242	
Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		Received by SWRI Lab: (Signature) Date 9/14/06	
Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		Samples Disposed: Date 11/30		Samples Disposed by:	

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

Page ___ of ___

Shipper Name/Address Bradley Werling B-57 Div. 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input checked="" type="checkbox"/> 3 Weeks Other: <u>4 weeks</u>	
Client Div. 20		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact Mike Dammann	
Sample ID AD-C LO-C TM-C HT-C		Sample Collection Date (mm/dd/yy) 9/14/06		Sample Collection Time Matrix Type Sample Type # of Containers		REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) PAP 15 applies *Min. 1ppm for potassium POC-B. Werling X 6565	
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°C Therm #: 27 Comments: RADIOACTIVE NP 237 @ 8.5x10 ⁻⁷ M or 1.4x10 ⁻⁴ µCi/ml SN 8/11/88		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		SWRI Project#: 20.06002.01.242	
Sample Types: D - Duplicate ER - Equipment Rinse ES - Environmental Sample FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		Received by SWRI Lab: (Signature) Date 9/14/06	
Relinquished by (Print/Signature) Received by (Print/Signature)		Date 9/14/06		Samples Disposed: Date 11/30		Samples Disposed by:	

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Shipper Name/Address Bradley Werling B-57 Div. 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client Div. 20		Client Purchase Order/Other ID Site/Zone ID				SwRI Contact Mike Dammann	
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		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature)		REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH >12 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) <i>None</i>	
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Date	Time
19WT-9A	9/11/06		W		1	9/11/06	
19WT-10A							
19WOT-9A							
19WOT-10A							
22WT-9A							
22WOT-10A							
22WOT-9A							
1922-3A							
1922-4A							
Temp: <i>22C</i> Therm #: <i>27</i>		Comments: <i>Radio active</i> <i>1p 237 @ 85x10⁻³M or 1.4x10⁻³µCi/ml</i> <i>SN 8/11/88</i>		SwRI Project#: <i>20.06002.01.242</i> Received by SwRI Lab: (Signature) <i>Ken P. [Signature]</i> Date, Time: <i>9/14/06 1130</i> Samples Disposed: Date Time Samples Disposed by:			

14 SEPT 2006 MR

Shipper Name/Address Bradley Werling B-57 Div. 20		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute® Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client Div. 20		Client Purchase Order/Other ID Site/Zone ID				SwRI Contact Mike Dammann	
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		Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature) Received by (Print/Signature) Relinquished by (Print/Signature)		REMARKS Preservation a = HCl to pH <2 b = HNO ₃ to pH <2 c = H ₂ SO ₄ to pH >12 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) <i>None</i>	
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Date	Time
AD-A	9/11/06		W		1	9/11/06	
LO-A							
TM-A							
HT-A							
Temp: <i>22C</i> Therm #: <i>27</i>		Comments: <i>Radio active</i> <i>1p 237 @ 85x10⁻³M or 1.4x10⁻³µCi/ml</i> <i>SN 8/11/88</i>		SwRI Project#: <i>20.06002.01.242</i> Received by SwRI Lab: (Signature) <i>Ken P. [Signature]</i> Date, Time: <i>9/14/06 1130</i> Samples Disposed: Date Time Samples Disposed by:			

19 Sept 2006 MR

**Step 8: Liquid Scintillation Analyses Results for Phase 2
Testing of 19PB and 22PC with and without Calcite
Experimental Solutions**

Task initially assigned to: Marla Roberts

Objective

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

Initial Entry 811/78

LSA sample preparation 811/82-83

Sample Info

Blank (1 sample from simulated water with calcium): w Ca 811/82-83

Neptunium spikes (2 samples) – Spike 61A-7, Spike 61A-8 (811/82-83)

Four experiments derived from 4 solids and 1 type of simulated water
19W T 9-11 (6 samples, with calcite) using simulated water with calcium
19WO T 9-11 (6 samples, without calcite) using simulated water with calcium
22W T 9-11 (6 samples, with calcite) using simulated water with calcium
22WO T 9-11 (6 samples, without calcite) using simulated water with calcium

Total number of LSA samples: 27

Results

Printout of LSA analyses following:

19 Sept 2006 MR

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9/13/06 1:08:40 PM

QuantaSmart (TM) - 1.31 - Serial# 405314

Page # 1

SNC Protocol

Calibration Information

Software Version IC: 2.11

Software Version EC: 1.31

Instrument Model: Tri-Carb 3100TR

Instrument Serial Number: 405314

3H Chi Square: 9.97 Date Processed: 9/13/06 1:08:39 PM

14C Chi Square: 17.93 Date Processed: 9/13/06 1:08:39 PM

3H E²/B (1-18.6 keV): 310.36 Date Processed: 9/13/06 1:08:39 PM

14C E²/B (4-156 keV): 583.57 Date Processed: 9/13/06 1:08:39 PM

3H Efficiency (0-18.6 keV): 65.20 Date Processed: 9/13/06 1:08:39 PM

14C Efficiency (0-156 keV): 97.01 Date Processed: 9/13/06 1:08:39 PM

IPA Background Date Processed: 9/13/06 1:08:39 PM

3H Background CPM (0-18.6 keV): 13.77 Date Processed: 9/13/06 1:08:39 PM

14C Background CPM (0-156 keV): 20.58 Date Processed: 9/13/06 1:08:39 PM

3H Calibration DPM: 285000

3H Reference Date: 10/29/99

14C Calibration DPM: 134100

19 Sept 2006 MR

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\20060913_1308.results

Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Pa_Np_Exp_AB\Np_Pa_AB.050

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): 200.00

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: 9.97 Date Processed: 9/13/06 1:08:39 PM

14C Chi Square: 17.93 Date Processed: 9/13/06 1:08:39 PM

3H E^2/B (1-18.6 keV): 310.36 Date Processed: 9/13/06 1:08:39 PM

14C E^2/B (4-156 keV): 583.57 Date Processed: 9/13/06 1:08:39 PM

3H Efficiency (0-18.6 keV): 65.20 Date Processed: 9/13/06 1:08:39 PM

14C Efficiency (0-156 keV): 97.01 Date Processed: 9/13/06 1:08:39 PM

IPA Background Date Processed: 9/13/06 1:08:39 PM

3H Background CPM (0-18.6 keV): 13.77 Date Processed: 9/13/06 1:08:39 PM

14C Background CPM (0-156 keV): 20.58 Date Processed: 9/13/06 1:08:39 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
1	200.00	19.50	3.20	3.27	7.81	0.54	19.25	168.6 <i>Blank w/cu</i>
B								
Missing vial 2.								
3	39.48	18.89	10.95	7.08	14.91	252.75	2.00	276.5 <i>19WT9a</i>
4	38.69	18.26	11.35	7.74	14.18	257.95	2.00	282.5 <i>19WT9b</i>
5	38.01	21.56	10.07	7.83	14.19	262.55	2.00	261.6 " <i>10a</i>
6	38.52	22.65	9.64	8.67	13.18	259.12	2.00	264.5 " <i>10b</i>
7	39.22	20.25	10.41	7.28	14.68	254.46	2.00	263.6 " <i>11a</i>
8	38.59	21.18	10.13	7.84	14.07	258.67	2.00	264.0 " <i>11b</i>
Missing vial 9.								
10	39.65	24.81	8.89	9.39	12.35	251.69	2.00	265.3 <i>19WOT9a</i>
11	39.35	25.15	8.83	10.45	11.57	253.62	2.00	291.0 <i>19WOT9b</i>
12	39.37	25.58	8.72	10.03	11.87	253.46	2.00	273.4 " <i>10a</i>
13	38.50	24.24	9.17	9.24	12.64	259.23	2.00	273.4 " <i>10b</i>
14	40.10	25.21	8.74	8.79	12.81	248.89	2.00	251.8 " <i>11a</i>
15	38.79	21.07	10.15	8.53	13.28	257.31	2.00	276.5 " <i>11b</i>
Missing vial 16.								
17	39.77	20.35	10.31	7.91	13.79	250.98	2.00	287.1 <i>22WT9a</i>
18	39.54	19.97	10.48	8.26	13.44	252.37	2.00	293.1 <i>22WT9b</i>
19	39.19	21.96	9.79	8.23	13.53	254.70	2.00	266.9 " <i>10a</i>
20	39.27	22.72	9.53	9.05	12.70	254.16	2.00	288.5 " <i>10b</i>
21	38.65	22.41	9.70	7.98	13.90	258.19	2.00	257.3 " <i>11a</i>
22	38.27	24.71	9.06	9.19	12.73	260.81	2.00	266.8 " <i>11b</i>
Missing vial 23.								
24	39.56	22.30	9.63	9.24	12.49	252.27	2.00	296.2 <i>22WOT9a</i>
25	38.96	21.56	9.95	8.28	13.52	256.26	2.00	276.5 <i>22WOT9b</i>
26	38.43	24.78	9.02	9.19	12.70	259.75	2.00	272.9 " <i>10a</i>
27	39.41	24.27	9.06	9.56	12.23	253.20	2.00	285.0 " <i>10b</i>
28	39.96	25.06	8.79	9.89	11.89	249.79	2.00	279.5 " <i>11a</i>
29	40.19	24.86	8.82	10.26	11.58	248.33	2.00	287.8 " <i>11b</i>
Missing vial 30.								
31	0.66	12725.95	2.18	4181.57	3.81	15266.13	1.99	252.4 <i>Spike 101A-7</i>
32	0.59	14068.63	2.20	4723.84	3.79	17058.78	1.99	255.1 <i>Spike 101A-8</i>

19 Sept 2006 MR

5 OCT 2006 MR

Preparation of Aliquots from September 2006 NC-EWDP Groundwater Samples for Div 01 Cation and Anion Analyses

Task Initially Assigned To: Marla Roberts and Sandra Watson

Objective: To prepare aliquots for cation and anion analyses that are intended to help characterize the groundwater in the Yucca Mountain vicinity and aid in the investigation concerning radionuclide sorption.

Sample Collection: 333/76-80

see 8/11/11 Jan 07 MR 110

Background: In the field 2 liters of filtered water was collected in 2 separate HDPE bottles. One of the two bottles was also preserved with 1:1 nitric acid. Although the water visually appeared clear, overnight a thin layer of particulates settled out. As a result, prior to being sent to Div 01 for analyses, the aliquots removed for analyses will be filtered again. See 333/76-80 for further information on field sampling parameters/conditions.

Samples: Three cation aliquots from well NC-EWDP-13P. One aliquot from the filtered acidified (FA) sample, one from the filtered unacidified (FUA) sample and one from the raw sample taken directly from the well (no filtering at time of acquisition).
 11 Jan 07 MR UFWA

Three anion aliquots from well NC-EWDP-13P. One aliquot from the filtered acidified sample, one from the filtered unacidified and one from the raw sample taken directly from the well (no filtering at time of acquisition).
 11 Jan 07 MR UFWA

Supplies: 60 mL pp bottles
30 mL (cc) syringes, BD #309604-04
Filters - Whatman #6874-2504, lot # 11026, 25mm dia and 0.45 µm pore size
100 mL glass beakers
parafilm

23 Oct 06 MR Raw changed to UFWA

Procedure: From each of the 3 samples (FA, FUA and Raw) a cation and anion aliquot will be taken and sent to Div 01 for analyses.

Approximately 60 mL of the FA NC-EWDP-13P well sample was decanted into a 100 glass beaker. A 30cc syringe was used to collect as much sample as possible from the beaker. Next a filter was attached to the end of the syringe. The sample was dispensed into a labeled 60mL pp bottle. The label contained sample ID, date, initials. This process was repeated for an anion sample.
 23 Oct 06 MR UFWA

The same process listed above was used to collect cation and anion samples from both FUA and Raw field samples. The 60 mL cation aliquot taken from the raw field sample was preserved with ~180 µL of 1:1 nitric (7/42/79). The following table contains sample IDs

Table 1: Legend related CNWRA and Div 01 sample IDs for NC-EWDP-13P-0906 samples.

CNWRA ID	Div 01 Cation	Div 01 Anion
NC-EWDP-13P-0906-FA	0906C-1	0906A-1
NC-EWDP-13P-0906-FUA	0906C-2	0906A-2
NC-EWDP-13P-0906-Raw*	0906C-3	0906A-3

**Note: See 8/11/110 for details on sample collection.
 *UFWA 23 Oct 06 MR

5 OCT 2006 MR

Preparation of Cation and Anion QA Solutions

Personnel Involved: Sandra Watson

Objective: To prepare QA solutions to include with Oct 2006 NC-EWDP cation and anion samples sent to Div 01 for analyses.

Reagents: Spex Certiprep Instrument Calibration Standard 3 (cat # CL-CAL-3, lot # 29-19AS)
Spex Certiprep IC Instrument Check Standard 2 (cat # ICMIX2-100, lot # 28-164AS)
Type 1 water (Barnstead/Thermolyne mode ID11901 sn1190010979691)

Supplies: Volumetric pipets
Volumetric flasks
HDPE bottles
Squirt bottle for water
Kimwipes

Procedure

Added 10 mL (volumetric pipet) of cation mix (Spex CL-CAL-3) into a 1000 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 1 L HDPE bottle and labeled Cation Std, date, initials, and SN ref. The cation dilution factor is 100.

Added 10 mL (volumetric pipet) of anion mix (Spex ICMIX2-100) into a 500 mL volumetric flask and filled to mark with type 1 water and mixed thoroughly. Decanted into a 500 mL HDPE bottle and labeled Anion Std, date, initials, and SN. The anion dilution factor is 50.

Table of Target Conc (ppm) of QA standards for samples

Species	Conc (ppm) in Spex Standard	Dilution Factor	Target Conc (ppm) of QA Standard
K	1000	100	10
Ca	1000	100	10
Na	1000	100	10
Mg	1000	100	10
HPO4	600	50	12
Br	400	50	8
NO3	400	50	8
SO4	400	50	8
Cl	200	50	4
F	100	50	2

5 OCT 2006 MR

Preparation of QA standards for
September 2006 NC-EWDP-BP
sampling event

Preparation of standards: 811/99

cation aliquot collected in 60 mL
pp bottles ^{500 mL} MR bottle from
Cation Std. 1006 (811/99) for
inclusion w/ NC-EWDP-BP
samples sent to Div 01. Cation
QA aliquot decanted into
60 mL pp bottle.

Anion aliquot collected in 60 mL
pp bottle from anion std. 1006
(811/99) for inclusion w/
NC-EWDP-BP samples sent
to Div 01. Anion QA aliquot
decanted into 60 mL pp bottle.

Target values for QAs: 811/99

QA Std. labels: 0906C-4 → cation
0906A-4 → anion

5 OCT 06 MR

5 OCT 2006 MR

Delivery of NC-EWDP-13P Sept 2006
Groundwater Samples to Div 01
for Cation + Anion Analyses

Collection of samples: 333/76-80

Samples: 3 cation (FA, FUA, UFUA)
3 anion (FA, FUA, UFUA)
2 QAs. (anion + cation)

Sample legend: 811/98

QA legend: 811/100

QA target concentrations: 811/99

Chain of custody on following pages:

5 OCT 06 MR

5 OCT 2006 MR

Brad Werling
Bld. 57
Dir 01

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 weeks

SWRI Contact: Mike Dammann

Client Purchase Order/Other ID: _____ Site/Zone ID: _____

Analyses Requested:

Sample ID	Sample Collection Date (m/d/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
0906A-1	10-05-06		W		1
0906A-2			I		
0906A-3			I		
0906A-4			I		

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: 22.00C Therm #: 027

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

Relinquished by (Print/Signature): Marla Roberts
Received by (Print/Signature): Marla Roberts

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Remarks:
a = HCl to pH <2
b = HNO₃ to pH <2
c = H₂SO₄ to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify) none

POP 15 applies
Min 1 ppm for potassium
POC - B Werling x 10565

SWRI Project#: 20.00002.01.242
Received by SWRI Lab (Signature): _____
Date: 10/06/06 Time: 1354
Samples Disposed: _____
Date: _____ Time: _____
Samples Disposed by: _____

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

Page _____ of _____

5 OCT 2006 MR

Brad Werling
Bld. 57
Dir 20

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

Requested Turnaround:
 2 Weeks
 3 Weeks
 Other: 4 weeks

SWRI Contact: Mike Dammann

Client Purchase Order/Other ID: _____ Site/Zone ID: _____

Analyses Requested:

Sample ID	Sample Collection Date (m/d/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
0906C-1	10-05-06		W		1
0906C-2			I		
0906C-3			I		
0906C-4			I		

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: 22.00C Therm #: 027

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

Relinquished by (Print/Signature): Marla Roberts
Received by (Print/Signature): Marla Roberts

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Relinquished by (Print/Signature): _____
Received by (Print/Signature): _____

Remarks:
a = HCl to pH <2
b = HNO₃ to pH <2
c = H₂SO₄ to pH <2
d = NaOH to pH >12
e = Cool (4°C±2°C)
f = Other (specify)

POP 15 applies
Min 1 ppm for potassium
POC - B Werling x 10565

SWRI Project#: 20.00002.01.242
Received by SWRI Lab (Signature): _____
Date: 10/06/06 Time: 1354
Samples Disposed: _____
Date: _____ Time: _____
Samples Disposed by: _____

11 OCT 2006 MR

Surface Area Analyses of 19PB and 22PC with and without Calcite Sonic Core Samples**Personnel initially involved:** Marla Roberts and Sandra Watson**Objectives:** To characterize alluvium in the Forty Mile Wash area of the Yucca Mountain region by determining the surface area of a composite sample from wells 19PB and 22PC as well as comparing the results with treated (calcite removed) sub-samples.**Samples:** NC-EWDP-19PB-0305-Sonic Core Composite - 696/64-66
NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite- 742/25
NC-EWDP-22PC-1005-Sonic Core Composite - 742/8
NC-EWDP-22PC-1005-Sonic Core Composite w/o Calcite- 742/25**Previous Key Entries for these analyses:**
Na-Acetate Treatment:**Equipment:** Coulter Surface Area Analyzer model SA 3100, serial number 46020
Mettler Electronic Balance model AE240, serial number 101237**Reagents:** NIST silicon nitride surface area standard 1899 (10m²/g)
Helium - Zero grade - 99.995%
Nitrogen - UHP grade - 99.999%
Liquid nitrogen
DOW Corning high vacuum grease
Type I water**Supplies:** Loading funnel
3cc glass surface area sample tubes with glass inserts and tube caps
Weighing paper
Spatulas**Procedure**

Prior to using the surface area analyzer, the O-rings were checked for integrity and replaced with new ones if brittle or cracked. The O-rings were lubricated with high vacuum grease.

At the beginning and end of the analyses the SAA was challenged with the NIST 1899 standard. This standard is of known surface area and is expected to have a surface area similar to the samples. The surface area analyzer can outgas three different samples at a time but can only perform the surface area determination one sample at a time.

The mass of the empty glass sample tube with glass stirrer and cap was recorded. To prevent biasing due to grain settling within the container, the container will be gently rolled by hand prior to aliquot removal. An appropriate amount (between 1.7 to 2.0 g) of sample was added to a tared weigh paper and the mass recorded. The aliquot was transferred into a glass sample tube

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with a loading funnel on top. The loading funnel was removed. The glass stirrer was inserted back into the sample tube and the tube cap was replaced. This process was repeated for two other samples since three samples can be outgassed at one time.

The following sample information was entered into the surface area analyzer data acquisition software prior to outgassing: sample ID, operator, and sample mass.

The tube caps were removed and the samples were inserted into the surface area analyzer for outgassing under the following conditions: outgas temperature of 200°C and outgas time of 360 minutes. Once the outgas process was complete, the samples could be removed. Each sample tube was promptly recapped and the total mass after outgassing is recorded. The sample mass after the outgassing process, referred to as the outgas sample mass, was calculated by subtracting the mass of the empty sample tube with glass stirrer and cap from the total mass.

The surface area analyses proceeded one sample at a time. An outgassed sample was uncapped and reloaded into the analyzer. The sample mass information in the data acquisition software was updated with the outgas sample mass. Liquid nitrogen was added to the analyzer and the analysis automatically starts. Surface area was analyzed by N-BET (Brunauer-Emmett-Teller with nitrogen as a carrier). At the completion of the analyses a hardcopy of the results was generated and placed in the scientific notebook. The process was repeated for any remaining outgassed samples.

After the analysis was complete, the sample aliquot was discarded. The sample glass tubes, inserts and caps were cleaned with type I water and oven dried. The whole process was repeated until all samples had been analyzed.

Sample Legend:

Sample ID	Surface Area Analyses Sample ID
NC-EWDP-19PB-0305-Sonic Core Composite	19W
NC-EWDP-19PB-0305-Sonic Core Composite w/o Calcite	19WO
NC-EWDP-22PC-1005-Sonic Core Composite	22W
NC-EWDP-22PC-1005-Sonic Core Composite w/o Calcite	22WO
NIST - 1899 Silicon Nitride	1899

3-15-07

BIO

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Surface Area Analyses of 19PB
and 22PC With & Without Calcite
Sonic Core Samples

- see initial entry: 811/104-105
- see sample legend for complete
Sample ID: 811/105

Before Outgassing - Initial

<u>Sample ID</u>	<u>Mass(g) of tube, cap & insert</u>	<u>Aliquot mass(g)</u>
1899	29.49952	^{11 OCT 06 MR} 1.714 1.05140
22W	29.48020	1.71411
22W0	28.73324	1.73943

See note: 811/109
18 OCT 06 MR

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17 OCT 2006 MR

Surface Area Analyses of 19PB
and 22PC With & Without Calcite Sonic
Core Samples

- see initial entry: 811/104-105
- see sample legend for complete
Sample ID: 811/105

After Outgassing

<u>Sample ID</u>	<u>Total Mass(g)</u>	<u>Outgassed Sample Mass(g)*</u>
1899	30.54977	1.05025
22W	^{10/17/06} 30.31.07830	1.5981
22W0	30.36872	1.63548

See note: 811/109
18 OCT 06 MR

* calculated

17 OCT 06 MR

17 Oct 06 MR

Surface Area Analyses of 19PB & 22PC With & Without Calcite Sonic Core Samples

- see initial entry: 811/104-105
- see complete sample ID - sample legend: 811/105

17 Oct 06 MR
 Before Outgassing - ~~HA~~ Initial

Sample ID	Mass(g) of tube, cap + HA ^{19PB} insert	Aliquot Mass(g)
19W	29.90298	1.74136
19W0	29.07567	1.73943

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~~1899~~

see note: 811/109
 18 Oct 06 MR

17 Oct 06 MR

18 Oct 06 MR

Surface Area Analyses of 19PB & 22PC With & Without Calcite Sonic Core Samples

- see initial entry: 811/104-105
- see sample legend for complete sample ID: 811/105

After Outgassing

Sample ID	Total Mass(g)	Outgassed Sample Mass(g)*
19W	31.53640	1.63342
19W0	30.69830	1.62263

*calculated
 Note:

*** all samples listed on 811/106-109 will be discarded. The LN₂ dewar used in the surface area analyzer was broken. SAA analyses will resume when the new dewar arrives. The same procedure described on 811/104-105 will be followed, however new ~~HA~~ ^{19PB} aliquots will be taken from the composite samples.

18 Oct 06 MR

25 OCT 06 MR

**Early Warning Drilling Program Groundwater Sampling Event
September 2006**

Personnel Involved: Miriam Jucket and Marla Roberts

Objective: To characterize groundwater and collect aliquots for use in sorption experiments in the Yucca Mountain vicinity in order to investigate radionuclide transport issues.

Samples: CNWRA personnel sampled a single well at the September 2006 sampling event. At the well, groundwater characterization will include field analyses of water temperature, air temperature, conductance, dissolved oxygen, oxidation-reduction potential, pH and alkalinity. Sample aliquots will be collected for subsequent lab analyses for cations, anions, stable isotope (hydrogen and oxygen) and inorganic carbon.

Background: This entry is a summary of the methods and procedures used during the sampling event. The purpose of this entry is to augment the in-field entries on 333/76-81.

Equipment: Hydrolab miniSonde 4A Water Quality Multiprobe (sn 41080)
Hydrolab Surveyor 4A Water Quality Data Display (sn S3123)
Masterflex E/S Model 07571-00 sample pump
Masterflex Easy-load L/S Model 7518-02 head

Reagents: 1:1 nitric acid (742/74)
Quinhydrone 98% (Acros Organics, Lot # AO15118301)
Type 1 water

Supplies: Millipore GWSC04510 disposable groundwater sampling capsule 0.45µm
L/S 24 tygon tubing
4 Liter pp bottles
2 Liter pp bottles
1 Liter HDPE bottles (ICChem N319-1000 certified 300 series)
500mL HDPE bottle (ICChem N319-0500 certified 300 series)
250 mL glass amber bottles (ICChem 349-0250 certified 300 series)
40 mL glass amber VOA bottles (ICChem T246-0040 certified series)
Hach digital titrators with J-hooks
Parafilm
Labels
Gloves
Squirt bottle
Sharpies
Pens
Pipettor and 5mL tips

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Disposable pipettes
Scissors
Kim-wipes
Trash bags
Zip locks
Batteries
Calculator
Flashlight
First Aid Kit
Ice crest
Duct Tape

Procedure:

The primary source for pH, ORP, conductivity and dissolved oxygen field data is the inline probe. The probe is challenged for pH. See 333/76-77 for challenge information. The inline probe collected data from the well for about 10 minutes at 30 second intervals. The data was stored in the Surveyor 4 data unit (filename-13P092806). Some readings from the inline probe were recorded (333/76-81) at the time of sampling.

Groundwater samples were collected in one 4 liter pp bottle and one 2 liter pp bottle. Each bottle was rinsed several times with sample water before being filled. Sample types, quantities and treatments for subsequent laboratory analyses are listed in Table 1. The sample IDs will be documented in the in-process entries. Sample IDs listed on following pages. The groundwater for the alkalinity titration was filter (0.45µm) before analyses.

Filtering was conducted using a sample pump, L/S 24 tygon tubing, and disposable Millipore groundwater filters. The bottles for sample collection for subsequent lab analyses were not rinsed with sample before filling because they were certified. Preservation consisted of 3 mL (oxford pipet) of 1:1 nitric into 1 liter of solution. Samples were labeled, parafilm, ziplocked, and placed in cooler. The samples were transferred into a refrigerator once back at the hotel, as space permitted.

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Table 1: Sample Types and Volumes Collected for Each Groundwater Zone During the September 2006 Early Warning Drilling Program Sampling Event.

Sample Volume and Container Type	Filtration	Preservation	Analyses
Two 40mL, Amber glass	Filtered, 0.45µm	None	Inorganic Carbon Analyses (in duplicate)
250mL, Amber glass	Filtered, 0.45µm	None	Stable Isotope Ratio (H/D and ¹⁶ O and ¹⁸ O)
500mL, High Density Polyethylene	Not filtered	None	Anions, cations, Trace metals
1 Liter High Density Polyethylene	Filtered, 0.45µm	1:1 HNO ₃ trace metal grade	Cations, trace metals
1 Liter High Density Polyethylene	Filtered, 0.45µm	none	Anions, trace metals

Local Well Number: NC-EWDP-13P

Well Info.: 2" PVC casing total depth = 476.1 ft. below ground surface (bgs)
 Depth to water = 432.73 ft from top of the PVC casing
 Pumping rate = 0.4- 0.5 gpm
 Pumped well over 3 well volumes (20 gallons). Started pumping on 27 Sept 06 at 1259, estimate pump stopped at 0500 on the 28 Sept 06.
 Restarted pump on 28 Sept 06 at 0913 and pumped continuously until all teams finished sampling at 1504.

Sample IDs	Volume	Sample Treatment/ID Key
NC-EWDP-13P-0906-FA	1L	F- filtered
NC-EWDP-13P-0906-FUA	1L	A- acidified
NC-EWDP-13P-0906-UFUA	500mL	U- unacidified
NC-EWDP-13P-0906-S-FUA	250mL	S- stable isotope
NC-EWDP-13P-0906-IC-FUA	2 x 40mL	IC- inorganic carbon

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Surface Area Analyses of 19PB and 22PC with and without Calcite Sonic Core Samples. - Continued.

See note: 811/109
 initial entry: 811/104
 Sample ID legend: 811/105

Before Outgassing - Initial

Sample ID	Mass(g) of tube, cap + insert	Aliquot Mass(g)
19W	29.19335	1.73526
19W0	28.45684	1.72011
1899	29.48441	1.03158

2 Nov 06 MR

3 Nov 06 MR & SW

Surface Area Analyses of 19PB and 22PC with and without Calcite Sonic Core Samples

see note: 811/109
 initial entry: 811/104
 sample ID legend: 811/105

After Outgassing

<u>Sample ID</u>	<u>Total Mass(g)</u>	<u>Outgassed Sample Mass*</u>
19W	30.83131	1.63796
19W0	30.09408	1.63724
1899	30.50802	1.02361

* calculated.

7 Nov 06 MR

10 Nov 06 MR & SW

Surface Area Analyses of 19PB and 22PC with and without Calcite Sonic Core Samples

see note: 811/109
 initial entry: 811/104
 sample ID legend: 811/105

Before Outgassing- Initial

<u>Sample ID</u>	<u>Mass(g) of tube, cap + insert</u>	<u>Aliquot Mass(g)</u>
22W	29.19555	1.77073
22W0	29.87974	1.76294
1899-2*	28.727 ^{SW 11/10/06} 28.72604	1.01571

* NIST Standard 1899 was run again at the end of the SAA group. To avoid confusion, this second analysis is labeled 1899-2.

3-15-07 SW

13 NOV 06 MR + SW

Surface Area Analyses of 19PB and 22PC with and without Calcite Sonic Core Samples

See note: 8/11/109
 initial entry: 8/11/104
 Sample ID Legend: 8/11/105

After Outgassing

Sample ID	Total Mas (g)	Outgassed ** Sample Mas (g)
22V	30.85161	1.65606
22W0	31.54001	1.66027
1899-2*	29.72587	0.99983

*see sample ID note 8/11/115
 8/11/110 13 NOV 06 MR

** calculated

13 NOV 06 MR

13 NOV 06 MR + SW

Surface Area Analyses Results - 19W

initial entry: 8/11/104

Coulter SA 3100 Surface Area and Pore Size Analyzer
 Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	19W	Start Date	05/30/04
Customer	CNWRA	Start Time	17:59:48
Operator	SW	Elapsed Time	46 min
Sample Wt	1.63796 g	Outgas Time	0 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	18.912 sq.m/g
Micropore Surface Area	0.981 sq.m/g
Micropore Volume	0.00016 ml/g
Correlation Coefficient	0.99953

t-Plot Surface Area Report

t-Plot Surface Area	18.912 sq.m/g
Total Surface Area	19.894 sq.m/g
Micropore Surface Area	0.981 sq.m/g
Micropore Volume	0.00016 ml/g

Film Thickness Range	0.35 to 0.50
Slope	12.2251
Intercept	0.10145
Correlation Coefficient	0.99953

Film Thickness (nm)	Vads (cc/g STP)
0.3518	4.376
0.3676	4.589
0.3815	4.768
0.3947	4.939
0.4084	5.108
0.4226	5.285
0.4355	5.439
0.4612	5.740
0.4871	6.030

Before outgassing:
 8/11/113
 After outgassing:
 8/11/114

Isotherm Data

Freespace Calculation

Slope	0.0188
Intercept	0.0112
Correlation Coefficient	0.99996

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
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Surface Area Analyses - 19W
continued

0.0000	0.000	0.001	743.58
0.0001	0.267	0.092	743.61
0.0003	0.541	0.212	743.53
0.0006	0.851	0.416	743.59
0.0010	1.166	0.753	743.63
0.0016	1.495	1.189	743.46
0.0025	1.841	1.874	743.45
0.0039	2.201	2.903	743.41
0.0061	2.563	4.553	743.21
0.0102	2.912	7.567	743.32
0.0165	3.213	12.259	743.27
0.0247	3.467	18.358	743.20
0.0301	3.599	22.374	743.28
0.0361	3.726	26.801	743.34
0.0430	3.854	31.933	743.21
0.0499	3.972	37.076	743.28
0.0546	4.045	40.557	743.20
0.0599	4.122	44.549	743.33
0.0703	4.257	52.234	743.15
0.0801	4.376	59.537	743.29
0.0997	4.589	74.127	743.20
0.1182	4.768	87.834	743.21
0.1367	4.939	101.612	743.25
0.1567	5.108	116.473	743.13
0.1782	5.285	132.364	742.99
0.1979	5.439	147.019	742.92
0.2379	5.740	176.733	742.94
0.2782	6.030	206.618	742.83
0.3190	6.318	236.922	742.68
0.3578	6.592	265.748	742.63
0.3997	6.871	296.827	742.54
0.4413	7.153	327.686	742.63

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13 NOV 06 MR & SW

Surface Area Analyses Results - 19W0
initial entry: 8/11/104

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	19W0	Start Date	05/30/04
Customer	CNWRA	Start Time	20:01:22
Operator	SW	Elapsed Time	41 min
Sample wt	1.63724 g	Outgas Time	0 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	11.640 sq.m/g
Micropore Surface Area	0.560 sq.m/g
Micropore Volume	0.00015 ml/g
Correlation Coefficient	0.99987

t-Plot Surface Area Report

t-Plot Surface Area	11.640 sq.m/g
Total Surface Area	12.200 sq.m/g
Micropore Surface Area	0.560 sq.m/g
Micropore Volume	0.00015 ml/g

Film Thickness Range	0.35 to 0.50
Slope	7.5240
Intercept	0.09746
Correlation Coefficient	0.99987

Film Thickness (nm)	Vads (cc/g STP)
0.3520	2.753
0.3680	2.870
0.3817	2.969
0.3955	3.069
0.4093	3.171
0.4229	3.274
0.4362	3.379
0.4626	3.575
0.4888	3.784

Before outgassing:
8/11/113

After outgassing:
8/11/114

Isotherm Data

Freespace Calculation

Slope	0.0177
Intercept	0.0028
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
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Surface Area Analyses - 19W0
continued

0.0000	0.000	0.001	748.99
0.0000	0.272	0.025	748.98
0.0001	0.545	0.086	748.93
0.0004	0.856	0.309	748.93
0.0011	1.196	0.826	748.93
0.0022	1.518	1.673	748.94
0.0050	1.832	3.744	748.95
0.0122	2.094	9.121	748.94
0.0248	2.295	18.591	748.93
0.0320	2.376	23.951	748.91
0.0395	2.450	29.576	748.95
0.0488	2.528	36.576	749.00
0.0579	2.597	43.344	749.05
0.0632	2.637	47.325	749.05
0.0701	2.685	52.507	749.03
0.0804	2.753	60.206	749.06
0.1003	2.870	75.106	748.99
0.1185	2.969	88.793	749.00
0.1380	3.069	103.341	748.98
0.1581	3.171	118.406	748.94
0.1785	3.274	133.706	748.93
0.1990	3.379	149.017	748.94
0.2400	3.575	179.740	748.87
0.2808	3.784	210.252	748.84
0.3168	3.977	237.273	748.85
0.3591	4.209	268.910	748.81
0.4004	4.444	299.832	748.76
0.4406	4.669	329.899	748.72

13 Nov 06 MR

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Surface Area Analyses Results - 1899
Initial entry: 8/11/04

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	1899	Start Date	05/31/04
Customer	CNWR	Start Time	17:11:52
Operator	SW	Elapsed Time	28 min
Sample wt	1.02361 g	Outgas Time	0 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	9.367 sq.m/g
Micropore Surface Area	1.131 sq.m/g
Micropore Volume	0.00043 ml/g
Correlation Coefficient	0.99999

t-Plot Surface Area Report

t-Plot Surface Area	9.367 sq.m/g
Total Surface Area	10.498 sq.m/g
Micropore Surface Area	1.131 sq.m/g
Micropore Volume	0.00043 ml/g

Film Thickness Range	0.35 to 0.50
Slope	6.0550
Intercept	0.27591
Correlation Coefficient	0.99999

Film Thickness (nm)	Vads (cc/g STP)
0.3525	2.409
0.3679	2.504
0.3818	2.588
0.3960	2.674
0.4098	2.757
0.4235	2.840
0.4364	2.918
0.4619	3.071
0.4878	3.231

Isotherm Data

Freespace Calculation

Slope	0.0192
Intercept	0.0018
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
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Before outgassing:
8/11/04 MR
113

After outgassing:
8/11/04

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Surface Area Analyses - 1899
continued

0.0000	0.000	0.001	747.53
0.0001	0.438	0.062	747.58
0.0006	0.865	0.440	747.53
0.0029	1.314	2.140	747.53
0.0090	1.667	6.692	747.53
0.0231	1.959	17.276	747.57
0.0311	2.056	23.285	747.59
0.0394	2.136	29.421	747.65
0.0496	2.218	37.117	747.62
0.0596	2.286	44.558	747.77
0.0653	2.323	48.851	747.75
0.0726	2.364	54.263	747.82
0.0809	2.409	60.495	747.88
0.1000	2.504	74.817	747.92
0.1186	2.588	88.755	748.09
0.1386	2.674	103.700	748.14
0.1589	2.757	118.858	748.18
0.1794	2.840	134.243	748.28
0.1993	2.918	149.118	748.28
0.2390	3.071	178.866	748.39
0.2793	3.231	209.055	748.44
0.3186	3.389	238.462	748.46
0.3601	3.563	269.511	748.52
0.3989	3.729	298.620	748.58
0.4402	3.910	329.525	748.59

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Surface Area Analyses Results - 22W
Initial entry: 811/104

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	22W	Start Date	06/04/04
Customer	CNWRA	Start Time	18:08:12
Operator	SW	Elapsed Time	44 min
Sample Wt	1.65606 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	16.549 sq.m/g
Micropore Surface Area	6.934 sq.m/g
Micropore Volume	0.00300 ml/g
Correlation Coefficient	0.99959

t-Plot Surface Area Report

t-Plot Surface Area	16.549 sq.m/g
Total Surface Area	23.484 sq.m/g
Micropore Surface Area	6.934 sq.m/g
Micropore Volume	0.00300 ml/g

Film Thickness Range	0.35 to 0.50
Slope	10.6978
Intercept	1.93834
Correlation Coefficient	0.99959

Film Thickness (nm)	Vads (cc/g STP)
0.3526	5.688
0.3679	5.870
0.3800	6.006
0.3951	6.175
0.4085	6.321
0.4226	6.471
0.4358	6.612
0.4609	6.868
0.4878	7.136

Before outgassing: 811/115

After outgassing: 811/116

Isotherm Data

Freespace Calculation

Slope	0.0182
Intercept	0.0031
Correlation Coefficient	1.00000

Isotherm Data Table

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Surface Area Analyses -22W
Continued

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	750.51
0.0001	0.267	0.044	750.53
0.0001	0.536	0.081	750.49
0.0002	0.848	0.118	750.50
0.0003	1.197	0.233	750.51
0.0003	1.537	0.249	750.54
0.0005	1.968	0.406	750.50
0.0008	2.344	0.567	750.56
0.0013	2.737	0.961	750.56
0.0022	3.150	1.615	750.58
0.0036	3.576	2.710	750.59
0.0061	4.007	4.583	750.62
0.0100	4.353	7.497	750.66
0.0166	4.648	12.448	750.67
0.0254	4.891	19.073	750.75
0.0312	5.013	23.435	750.75
0.0374	5.129	28.107	750.80
0.0448	5.243	33.672	750.87
0.0522	5.351	39.218	750.91
0.0571	5.416	42.876	750.96
0.0626	5.487	46.977	751.00
0.0701	5.573	52.620	751.04
0.0810	5.688	60.847	751.04
0.1001	5.870	75.164	751.06
0.1162	6.006	87.268	751.09
0.1374	6.175	103.176	751.05
0.1570	6.321	117.901	751.07
0.1780	6.471	133.706	751.04
0.1983	6.612	148.967	751.04
0.2373	6.868	178.209	750.99
0.2794	7.136	209.775	750.93
0.3179	7.383	238.765	750.96
0.3589	7.638	269.481	750.86
0.4001	7.891	300.340	750.73
0.4398	8.136	330.124	750.67

14 NOV 06 MR

14 NOV 06 MR SW

Surface Area Analyses Results -22W0
Initial entry: 8/11/04

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	22W0	Start Date	06/04/04
Customer	CNwRA	Start Time	20:46:39
Operator	SW	Elapsed Time	38 min
Sample wt	1.66027 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	10.571 sq.m/g
Micropore Surface Area	0.858 sq.m/g
Micropore Volume	0.00029 ml/g
Correlation Coefficient	0.99999

t-Plot Surface Area Report

t-Plot Surface Area	10.571 sq.m/g
Total Surface Area	11.429 sq.m/g
Micropore Surface Area	0.858 sq.m/g
Micropore Volume	0.00029 ml/g

Film Thickness Range	0.35 to 0.50
Slope	6.8333
Intercept	0.18900
Correlation Coefficient	0.99999

Film Thickness (nm)	Vads (cc/g STP)
0.3520	2.596
0.3684	2.708
0.3808	2.790
0.3953	2.889
0.4092	2.986
0.4233	3.079
0.4371	3.175
0.4614	3.343
0.4877	3.523

Before outgassing:
8/11/115

After outgassing:
8/11/116

Isotherm Data

Freespace Calculation

Slope	0.0159
Intercept	0.0022
Correlation Coefficient	1.00000

14 NOV 06 MR SW

Surface Area Analyses
continued.

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	754.51
0.0000	0.271	0.021	754.52
0.0001	0.540	0.079	754.54
0.0004	0.848	0.308	754.57
0.0012	1.183	0.909	754.51
0.0026	1.496	1.965	754.51
0.0063	1.796	4.747	754.55
0.0166	2.057	12.538	754.56
0.0304	2.227	22.918	754.60
0.0371	2.292	28.002	754.60
0.0453	2.361	34.188	754.63
0.0554	2.436	41.842	754.61
0.0612	2.476	46.150	754.62
0.0698	2.533	52.642	754.60
0.0803	2.596	60.585	754.59
0.1008	2.708	76.056	754.61
0.1173	2.790	88.521	754.57
0.1377	2.889	103.882	754.61
0.1579	2.986	119.160	754.51
0.1791	3.079	135.127	754.54
0.2004	3.175	151.195	754.53
0.2382	3.343	179.744	754.51
0.2791	3.523	210.613	754.51
0.3195	3.708	241.039	754.44
0.3587	3.891	270.601	754.49
0.3993	4.091	301.206	754.39
0.4403	4.287	332.115	754.33

14 NOV 06 MR

14 NOV 06 MR SW

Surface Area Analyses Results - 1899-2
initial entry: 8/11/104

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	1899-2	Start Date	06/04/04
Customer	CNWRA	Start Time	21:38:03
Operator	SW	Elapsed Time	28 min
Sample Wt	0.99983 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	9.746 sq.m/g
Micropore Surface Area	1.102 sq.m/g
Micropore Volume	0.00040 ml/g
Correlation Coefficient	0.99999

t-Plot Surface Area Report

t-Plot Surface Area	9.746 sq.m/g
Total Surface Area	10.847 sq.m/g
Micropore Surface Area	1.102 sq.m/g
Micropore Volume	0.00040 ml/g

Film Thickness Range	0.35 to 0.50
Slope	6.2997
Intercept	0.26053
Correlation Coefficient	0.99999

Film Thickness (nm)	Vads (cc/g STP)
0.3523	2.478
0.3680	2.579
0.3820	2.669
0.3960	2.756
0.4101	2.844
0.4231	2.927
0.4365	3.010
0.4623	3.171
0.4878	3.333

Before
outgassing:
8/11/115
After
outgassing:
8/11/116

Isotherm Data

Freespace Calculation

Slope	0.0189
Intercept	0.0013
Correlation Coefficient	1.00000

Isotherm Data Table

14 NOV 06 MR SW

Surface Area Analyses
continued

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	756.03
0.0001	0.449	0.046	756.07
0.0005	0.884	0.383	756.16
0.0027	1.352	2.004	756.15
0.0088	1.717	6.654	756.17
0.0230	2.012	17.420	756.21
0.0311	2.110	23.488	756.22
0.0391	2.191	29.573	756.25
0.0495	2.276	37.415	756.23
0.0595	2.349	45.027	756.26
0.0652	2.386	49.327	756.27
0.0725	2.431	54.847	756.21
0.0807	2.478	61.055	756.24
0.1003	2.579	75.829	756.25
0.1190	2.669	89.965	756.24
0.1386	2.756	104.825	756.25
0.1592	2.844	120.422	756.26
0.1789	2.927	135.299	756.28
0.1994	3.010	150.767	756.24
0.2396	3.171	181.213	756.30
0.2792	3.333	211.180	756.33
0.3185	3.500	240.890	756.25
0.3595	3.675	271.917	756.31
0.4001	3.851	302.623	756.33
0.4396	4.032	332.510	756.34

14 NOV 06 MR

14 Nov 2006 Notebook review completed.

PO

16 NOV 06 MR SW

Removal of Aliquots from Sonic Core Intervals (<#35) Used to Create 19PB and 22PC Sonic Core Composite Samples

Personnel involved: Marla Roberts and Sandra Watson

Objectives:

To remove aliquots from the less than (<) #35 mesh from the intervals used to created the 19PB and 22PC Sonic Core Composite samples for use in future sorption experiments..

Samples:

Intervals used for the NC-EWDP-19PB-0305- Sonic Core Composite

NC-EWDP-19PB-373.6-374.6 <#35 Mesh A	SN 628/37-43
NC-EWDP-19PB-387.2-388.9 <#35 Mesh A	SN 628/37-43
NC-EWDP-19PB-408.5-409.9 <#35 Mesh A	SN 628/37-43
NC-EWDP-19PB-418.4-419.6 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-424.6-428.7 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-441.2-442.9 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-457.2-460.7 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-470.5-474.3 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-503.4-506.0 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-547.6-549.6 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-584.1-587.4 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-595.2-596.2 <#35 Mesh A	SN 628/55
NC-EWDP-19PB-631.1-633.8 <#35 Mesh A	SN 628/55

Intervals used for the NC-EWDP-22PC-1005- Sonic Core Composite

NC-EWDP-22PC-0305 464.9-466.5 <#35	SN 631/76
NC-EWDP-22PC-0305 486.9-488.5 <#35	SN 631/88
NC-EWDP-22PC-0305 502.5-504.2 <#35	SN 631/88
NC-EWDP-22PC-0305 522.1-523.5 <#35	SN 631/78
NC-EWDP-22PC-0305 539.2-540.8 <#35	SN 710/8
NC-EWDP-22PC-0305 555.3-556.8 <#35	SN 710/9
NC-EWDP-22PC-0305 571.3-573.0 <#35	SN 631/88
NC-EWDP-22PC-0305 588.3-589.9 <#35	SN 631/88
NC-EWDP-22PC-0305 610.1-611.0 <#35	SN 631/84
NC-EWDP-22PC-0305 625.2-626.7 <#35	SN 631/92
NC-EWDP-22PC-0305 640.3-641.4 <#35	SN 631/74
NC-EWDP-22PC-0305 658.3-659.7 <#35	SN 631/92
NC-EWDP-22PC-0305 675.9-677.3 <#35	SN 631/94
NC-EWDP-22PC-0305 686.1-687.4 <#35	SN 710/2-3
NC-EWDP-22PC-0305 715.0-717.1 <#35	SN 710/9
NC-EWDP-22PC-0305 735.7-736.4 <#35	SN 631/74
NC-EWDP-22PC-0305 760.3-761.6 <#35	SN 710/9

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Equipment:

Sartorius Scale - Model: 3808MPS-IV20, (sn 39030006)
Mettler Toledo XP205DR (sn 1126461033)

Supplies:

Weighing Paper
Polystyrene sample containers-various sizes

Basic Outline Procedure:

To prevent biasing due to grain settling within the container, the container will be gently rolled by hand prior to aliquot removal. An appropriate amount (approximately 50 grams) of sample was added to a tared weigh paper and the mass recorded. The aliquots were then placed in a polystyrene container labeled with the sample ID, initials, SN reference and date. "Sorp" was added to the end of the sample ID to identify it is to be used in future sorption experiments. In the event that a sample interval does not have enough remaining sample to remove 50 grams, the interval will be noted, and not used in this series of sorption experiments.

Table 1: Intervals used for the NC-EWDP-19PB-0305- Sonic Core Composite
Table 2: Intervals used for the NC-EWDP-22PC-1005- Sonic Core Composite

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In process entry.

The total amount (mass(g)) of <#35 sediment per interval was insufficient to remove ~ 50g. Therefore, aliquot masses removed from each interval range from ~10g to ~38g. Aliquot masses can be found on Tables 1 & 2.

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Table 1: Original Sample ID
NC-EWDP-19PB - "Interval" <#35 Mesh A

Sample ID	Aliquot Mass (g)
373.6-374.6	30.20
387.2-388.9	* too little sediment to remove aliquot for sorption experiment
408.5-409.9	30.31
418.4-419.6	10.36
424.6-428.7	15.09
441.2-442.9	30.00
457.2-460.7	30.27
470.5-474.3	20.36
503.4-506.0	* too little sediment to remove aliquot for sorption experiment
547.6-549.6	30.69
584.1-587.4	25.24
595.2-596.2	30.07
631.1-633.8	30.41

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Table 2: Original Sample ID
 NG-EWDP-22PC - "interval" - <#35

Sample ID	Aliquot Mass (g)
464.9-466.5	31.2
486.9-488.5	15.4
502.5-504.2	26.7
522.1-523.5	*too little sediment to extract aliquot for sorption experiments
539.2-540.8	32.9
555.3-556.8	13.9
571.3-573.0	24.7
588.3-589.9	31.2
610.1-611.0	32.2
625.2-626.7	36.0
640.3-641.4	38.1
658.3-659.7	32.4
675.9-677.3	31.0
686.1-687.4	25.3
715.0-717.1	27.8
735.7-736.4	26.8
760.3-761.6	38.3

20 NOV 06 MR

Preparation of Stock Solutions for 19PB Simulated Water with Calcium (19PB-5)

Task Assigned To: Marla Roberts, Sandra Watson

Objective: To prepare stock solutions used to make simulated water based on NC-EWDP-19PB shallow string for use in sorption experiments

Background: New stock solutions are being made for use in the creation of future 19PB Simulated Groundwater with Calcium. Previous stock solutions (742/27) were used to create 19PB-2, 19PB-3 and 19PB-4 Simulated Groundwater.

The amounts and types of compounds used to prepare the simulated water will reflect the composition of 19PB shallow water. The target concentrations for the 19PB shallow simulated water were based on the results of analyses performed on 19PB shallow water samples collected by CNWRA personnel (628/128, 628/197-198, 653/37-48 and 653/67-80). Tables 1 (811/134) represents the concentrations associated with the actual groundwater based on analyses. Table 2 (811/134) represents the composition of stock and final solutions of 19PB Simulated Shallow String Groundwater (with Calcium). The masses reported in Table 3 (811/134) were used to prepare the simulated water stock solution and project to slightly different concentrations. Samples from the simulated 19PB water prepared from these stock solutions as well as the stock solutions themselves will be sent to Division 01 for anion and cation analyses in order to assess the actual concentration of species in solution.

Three (A-C) 1 liter stock solutions will be prepared.

Stock Solution A - Calcium Nitrate, Calcium Sulfate, Calcium Chloride and Magnesium Chloride

Stock Solution B - Potassium Fluoride and Sodium Bicarbonate

Stock Solution C - Sodium Meta-Silicate

Procedure:

Compounds individually added to tared weigh boats. Transferred to 1000 mL beaker with 800mL of Type 1 water. Washed weigh boat with Type 1 water with rinsate added 1000 mL beaker. Stirred on stir plate until everything dissolved (varying time periods, usually under 5 minutes). Transferred into 1000 mL volumetric flask. Beaker rinsed and rinsate added to the volumetric flask. Diluted to mark with Type 1 water. Labeled with appropriate stock solution letter, SN, initials and date.

3-15-07 BW

20 NOV 06 MR

Table 1: Representative chemical composition of NC-EWDP-19PB Shallow String Groundwater based on cation, anion and field analyses.

Species	Target Conc (mg/L) for Simulated Shallow Water
Calcium	14
Magnesium	1.3
Potassium	3.6
Silicon	20
Sodium	52
Chloride	7
Fluoride	1.8
Nitrate	3
Sulfate	29
Bicarbonate	143

Table 2: Target chemical composition of stock and final solutions of 19PB Simulated Shallow String Groundwater (with Calcium)

Species	Target Conc (mg/L) for Prepared Stock Solution (note A)	Target Conc (mg/L) for Prepared Simulated Shallow Water
Calcium	561	14.0
Magnesium	48.6	1.22
Potassium	120	3.00
Silicon	480	12.0
Sodium	2074	51.8
Chloride	248	6.20
Fluoride	58.3	1.46
Nitrate	112	2.79
Sulfate	1120	27.9
Bicarbonate	3420	85.4

Note A: based on a dilution factor of 40 for the stock solutions (25mL into 1L)

Table 3: Compound masses (g) used for preparing stock solutions for 19PB shallow simulated water.

Compound	Mass (g) compound for 1 L of stock solution
NaHCO ₃	4.7046
KF*2H ₂ O	0.2890
Ca(NO ₃) ₂ *4H ₂ O	0.2125
CaSO ₄ *2H ₂ O	1.9972
MgCl ₂ *6H ₂ O	0.4066
CaCl ₂ *6H ₂ O	0.3286
Na ₂ SiO ₃ *9H ₂ O	4.8598

20 NOV 06 MR

Preparation of Stock Solution A

initial entry: 8/1/133

Equipment:

Mettler Toledo XP205DR SN1126461035
 1000mL volumetric flask
 weigh boat
 funnel
 stir bar
 stir plate
 1000mL beaker
 Type I water

Challenge Balance (target = 20.0001g)
 - Before analyses: 19.99997
 - After analyses:

Chemicals:

Ca(NO₃)₂ · 4H₂O: Fisher C109-500 Lot 035087
 CaSO₄ · 2H₂O: sigma aldrich 255548 Lot 0261TB
 CaCl₂ · 6H₂O: Fisher C78-500 Lot 033494
 MgCl₂ · 6H₂O: Fisher M33-500 Lot 006854

Masses (g) added:

calcium nitrate - 0.21055 g
 calcium sulfate - 1.99719 g
 calcium chloride - 0.32894 g
 magnesium chloride - 0.40654 g

2-15-07 BW

20 NOV 06 MR

Procedure:

Calcium sulfate added first stirred for ~4 hours before dissolved. Then added calcium nitrate, calcium chloride and magnesium chloride. All dissolved immediately.

Labeled stock solution A, SN, initials and date

See procedure 811/133 section

~~20 NOV 06 MR~~

20 NOV 06 MR

Preparation of Stock Solution B

initial entry: 811/133

Equipment:

Same as 811/135

Challenge Balance (target = 20.0001g)

- Before analyses:

- After analyses:

Chemicals:

KF · 2H₂O: sigmaaldrich 221872 lot 09516JC

NaHCO₃: Fisher S233-500 lot 006275

Procedure:

KF was added first - dissolved after ~1 minute. Sodium bicarb added next dissolved immediately.

Labeled stock solution B, ^{20 NOV 06} SN, initials and date. ~~MR~~

See procedure section 811/133.

^{20 NOV 06} ~~MR~~ Masses (g) Added:

KF = 0.28890 g

NaHCO₃ = 4.70045

~~3-15-07 BLS~~

20 NOV 06 MR

Preparation of Stock Solution C

initial entry: 811/133

Equipment

same as 811/135

Challenge Balance (target = 20.0001g)

- Before analyses
- After analyses

Chemicals:

Na₂SiO₃ · 9H₂O: Fisher S408-500 Lot 942853A

Mass(g) added:

Sodium meta silicate - 4.85914

Procedure:

Sodium meta silicate dissolved immediately

Labeled Stock Solution C, SN, initials date

See procedure section 811/133

~~20 NOV 06 MR~~22 ^{22 NOV 06} MR
~~20~~ NOV 06 MRPreparation of 19PB Shallow String Simulated Groundwater with Calcium (5th Batch)

Task Assigned To: Marla Roberts

Objective: To prepare simulated groundwater (from stock solutions) that can be used for sorption experiments.**Background:** 19PB Shallow String Simulated Groundwater with Calcium is created by combining Stock Solutions A, B and C. To achieving the appropriate concentrations as described on 811/133-134, a 40 fold dilution factor is used. Meaning, 25 mL of each stock solution is required per liter of simulated water. The goal is to prepare 4L of 19PB Simulated Water with Calcium. Therefore 100mL of each stock solution is necessary to create 19PB Shallow String Simulated Groundwater with Calcium.

Equipment:

2 L volumetric flask	Glass gas dispersion tube
1 L volumetric flask	Moisture bottle
100 mL volumetric flask	Pump
4 L HDPE carboy	
100 mL beaker	
50 mL beaker	
Funnel	
Disposable pipet	

Reagents:

- Stock Solution A (811/135)
- Stock Solution B (811/137)
- Stock Solution C (811/138)
- Type 1 water

Procedure: 2 L (via 2L volumetric flask) of Type 1 water was added to the 4L carboy. 100mL (volumetric flask with funnel) of Stock Solution A was collected. Taken to mark using disposable pipet and 50 mL beaker (filled with Stock Solution A). The 100 mL was transferred to 4L carboy. Following the same steps, 100mL of Stock Solution B and Stock Solution C were added to the 4L carboy. After Stock Solution A, B and C was added to the 4L carboy, the total volume of the carboy was 2300mL. The carboy was filled with an additional 1700 mL of Type 1 water. The carboy was swirled by hand. The carboy was labeled 19PB-5 Simulated Groundwater. The '5' is to indicate this is the fifth batch of 19PB Simulated Groundwater with Calcium (see background 811/133). Finally, the carboy was set-up with an aeration device. A glass gas dispersion tube was inserted into the 4L carboy with a 'moisture bottle' between the carboy and the pump. The carboy opening is covered with a cut-out piece of bench paper.

5 Dec 06 MR
~~20 NOV 06 MR~~ 5 Dec 06 MR

Surface Area Analyses of 19PB Intervals Used to Create 19PB Composite-Spot Check

Personnel initially involved: Sandra Watson and Marla Roberts

Objectives: To characterize alluvium in the Forty Mile Wash area of the Yucca Mountain region by determining the surface area of the sample intervals used to create the composite sample from well 19PB. There are a total of 13 intervals used to create the composite, however, this experiment will only be spot check previous surface area results 631/6-17

Samples: NC-EWDP-19PB-373.6-374.0 <#35 mesh A
NC-EWDP-19PB-408.5-409.9 <#35 mesh A
NC-EWDP-19PB-424.6-428.7 <#35 mesh A
NC-EWDP-19PB-457.2-460.7 <#35 mesh A
NC-EWDP-19PB-503.4-506.0 <#35 mesh A
NC-EWDP-19PB-631.1-633.8 <#35 mesh A

Previous Key Entries for these analyses:
631-6-17

Previous SAA Methodology: outgassing time: 12 hours, outgassing temperature: 350 °C

Equipment: Coulter Surface Area Analyzer model SA 3100, serial number 46020
Mettler Electronic Balance model AE240, serial number 101237

Reagents: NIST silicon nitride surface area standard 1899 (10m²/g)
Helium - Zero grade - 99.995%
Nitrogen - UHP grade - 99.999%
Liquid nitrogen
DOW Corning high vacuum grease
Type I water

Supplies: Loading funnel
3cc glass surface area sample tubes with glass inserts and tube caps
Weighing paper
Spatulas

Procedure

Prior to using the surface area analyzer, the O-rings were checked for integrity and replaced with new ones if brittle or cracked. The O-rings were lubricated with high vacuum grease.

At the beginning and end of the analyses the SAA was challenged with the NIST 1899 standard. This standard is of known surface area and is expected to have a surface area similar to the samples. The surface area analyzer can outgas three different samples at a time but can only perform the surface area determination one sample at a time.

5 Dec 06 MR

The mass of the empty glass sample tube with glass stirrer and cap was recorded. To prevent biasing due to grain settling within the container, the container will be gently rolled by hand prior to aliquot removal. An appropriate amount (between 1.7 to 2.0 g) of sample was added to a tared weigh paper and the mass recorded. The aliquot was transferred into a glass sample tube with a loading funnel on top. The loading funnel was removed. The glass stirrer was inserted back into the sample tube and the tube cap was replaced. This process was repeated for two other samples since three samples can be outgassed at one time.

The following sample information was entered into the surface area analyzer data acquisition software prior to outgassing: sample ID, operator, and sample mass.

The tube caps were removed and the samples were inserted into the surface area analyzer for outgassing under the following conditions: outgas temperature of 200°C and outgas time of 360 minutes. Once the outgas process was complete, the samples could be removed. Each sample tube was promptly recapped and the total mass after outgassing is recorded. The sample mass after the outgassing process, referred to as the outgas sample mass, was calculated by subtracting the mass of the empty sample tube with glass stirrer and cap from the total mass.

The surface area analyses proceeded one sample at a time. An outgassed sample was uncapped and reloaded into the analyzer. The sample mass information in the data acquisition software was updated with the outgas sample mass. Liquid nitrogen was added to the analyzer and the analysis automatically starts. Surface area was analyzed by N-BET (Brunauer-Emmett-Teller with nitrogen as a carrier). At the completion of the analyses a hardcopy of the results was generated and placed in the scientific notebook. The process was repeated for any remaining outgassed samples.

After the analysis was complete, the sample aliquot was discarded. The sample glass tubes, inserts and caps were cleaned with type I water and oven dried. The whole process was repeated until all samples had been analyzed.

~~3-17-07 BW
3-15-07 BW
3-17-07 BW
3-15-07 BW~~

5 Dec 06 MR

Surface Area Analyses of
19PB intervals

initial entry: 811/140
* Sample ID prefix: NC-EWDP-19PB

Before Outgassing - Initial

<u>Sample ID*</u>	<u>Mass(g) of tube, cap & insert</u>	<u>Aliquot Mass(g)</u>
373.6-374.0 <#35	28.64602	1.70113
408.5-409.9 <#35	28.59850	1.70783
424.6-428.7 <#35	29.77101	1.70406

5 Dec 06 MR

6 Dec 06 MR

Surface Area Analyses of 19PB
intervals

initial entry: 811/140
* Sample ID prefix: NC-EWDP-19PB

Before Outgassing - Initial

<u>Sample ID*</u>	<u>Mass(g) of tube, cap & insert</u>	<u>Aliquot Mass(g)</u>
457.2-460.7 <#35	29.41878	1.80193
503.4-506.0 <#35	29.27043	1.84123
631.1-633.8 <#35	29.48646	1.83105

6 Dec 06 MR

7 Dec 06 MR

Surface Area Analyses of 19PB intervals

initial entry: 811/140

*sample ID prefix: NL-EWDP-19PB

After outgassing

<u>Sample ID*</u>	<u>Total Mass(g)</u>	<u>Outgassed** Sample Mass(g)</u>
373.6-374.0 <#35	30.25625	1.61023
408.5-409.9 <#35	30.17184	1.57334
424.6-428.7 <#35	31.40796	1.63695

** calculated

7 Dec 06 MR

8 Dec 06 MR

Surface Area Analyses of 19PB Intervals

initial entry: 811/140

*sample ID prefix: NL-EWDP-19PB

After outgassing

<u>Sample ID*</u>	<u>Total Mass(g)</u>	<u>Outgassed** Sample Mass(g)</u>
457.2-460.7 <#35	31.14461	1.72583
503.4-506.0 <#35	31.01456	1.74413
631.1-633.8 <#35	31.22689	1.74043

** calculated.

8 Dec 06 MR

13 Dec 2006 MR

Surface Area Analysis Results:
NC-EWDP-19PB-424.6-428.7 < #35

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	424-6	Start Date	06/07/04
Customer	CNWRA	Start Time	21:49:22
Operator	SW	Elapsed Time	45 min
Sample wt	1.63695 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	18.601 sq.m/g
Micropore Surface Area	7.885 sq.m/g
Micropore Volume	0.00341 ml/g
Correlation Coefficient	0.99958

t-Plot Surface Area Report

t-Plot Surface Area	18.601 sq.m/g
Total Surface Area	26.486 sq.m/g
Micropore Surface Area	7.885 sq.m/g
Micropore Volume	0.00341 ml/g

Film Thickness Range	0.35 to 0.50
Slope	12.0241
Intercept	2.20435
Correlation Coefficient	0.99958

Film Thickness (nm)	Vads (cc/g STP)
0.3525	6.420
0.3688	6.631
0.3800	6.776
0.3937	6.949
0.4090	7.137
0.4224	7.300
0.4356	7.455
0.4616	7.752
0.4872	8.039

Isotherm Data

Freespace Calculation

Slope	0.0162
Intercept	0.0021

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SAA Results Cont.

Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	776.98
0.0000	0.274	0.027	777.02
0.0001	0.547	0.049	776.98
0.0001	0.864	0.073	777.00
0.0002	1.216	0.165	777.09
0.0002	1.559	0.176	777.15
0.0004	1.995	0.276	777.13
0.0005	2.379	0.383	777.21
0.0010	2.920	0.758	777.21
0.0016	3.345	1.225	777.23
0.0026	3.784	2.034	777.37
0.0043	4.239	3.330	777.40
0.0074	4.696	5.760	777.37
0.0119	5.036	9.284	777.43
0.0189	5.326	14.665	777.42
0.0275	5.570	21.394	777.39
0.0331	5.695	25.696	777.46
0.0393	5.819	30.524	777.49
0.0463	5.944	35.988	777.52
0.0535	6.058	41.608	777.55
0.0583	6.128	45.335	777.63
0.0635	6.203	49.359	777.64
0.0698	6.288	54.313	777.64
0.0809	6.420	62.944	777.66
0.1012	6.631	78.724	777.70
0.1162	6.776	90.340	777.69
0.1353	6.949	105.226	777.76
0.1577	7.137	122.666	777.77
0.1779	7.300	138.340	777.79
0.1980	7.455	153.982	777.82
0.2385	7.752	185.511	777.83
0.2783	8.039	216.476	777.82
0.3176	8.317	247.044	777.82
0.3593	8.605	279.452	777.85
0.3998	8.882	310.945	777.77
0.4399	9.150	342.159	777.79

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Surface Area Analysis Results:
ML-ENDP-19PB-457.2-460.7 < #35

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	457	Start Date	06/07/04
Customer	CNWRA	Start Time	22:54:03
Operator	SW	Elapsed Time	41 min
Sample Wt	1.72583 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	13.984 sq.m/g
Micropore Surface Area	6.404 sq.m/g
Micropore Volume	0.00278 ml/g
Correlation Coefficient	0.99933

t-Plot Surface Area Report

t-Plot Surface Area	13.984 sq.m/g
Total Surface Area	20.387 sq.m/g
Micropore Surface Area	6.404 sq.m/g
Micropore Volume	0.00278 ml/g

Film Thickness Range	0.35 to 0.50
Slope	9.0392
Intercept	1.79514
Correlation Coefficient	0.99933

Film Thickness (nm)	Vads (cc/g STP)
0.3520	4.955
0.3682	5.117
0.3808	5.238
0.3949	5.376
0.4088	5.504
0.4234	5.637
0.4357	5.745
0.4610	5.960
0.4875	6.180

Isotherm Data

Freespace Calculation

Slope	0.0176
Intercept	0.0026

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SAA Results Cont.

Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	778.16
0.0000	0.261	0.031	778.13
0.0001	0.521	0.064	778.07
0.0001	0.824	0.101	778.06
0.0003	1.158	0.217	778.03
0.0003	1.482	0.260	777.96
0.0006	1.895	0.448	777.91
0.0009	2.254	0.719	777.88
0.0017	2.625	1.305	777.82
0.0029	3.013	2.253	777.72
0.0051	3.405	3.999	777.62
0.0096	3.786	7.479	777.57
0.0166	4.062	12.915	777.48
0.0259	4.280	20.158	777.37
0.0318	4.387	24.698	777.34
0.0382	4.490	29.670	777.21
0.0459	4.594	35.695	777.15
0.0535	4.687	41.594	777.07
0.0585	4.743	45.454	777.02
0.0642	4.804	49.897	776.93
0.0701	4.862	54.467	776.90
0.0804	4.955	62.447	776.82
0.1005	5.117	78.082	776.79
0.1173	5.238	91.079	776.75
0.1371	5.376	106.448	776.68
0.1573	5.504	122.144	776.60
0.1794	5.637	139.281	776.54
0.1982	5.745	153.917	776.49
0.2375	5.960	184.369	776.43
0.2789	6.180	216.533	776.41
0.3186	6.387	247.331	776.33
0.3586	6.599	278.360	776.28
0.3989	6.808	309.628	776.26
0.4414	7.026	342.596	776.20

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 Surface Area Analysis Results:
 NG EWDP-19PB-503.4-506.0 <#35

Coulter SA 3100 Surface Area and Pore Size Analyzer
 Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	503	Start Date	06/12/04
Customer	CNWRA	Start Time	17:59:33
Operator	MR	Elapsed Time	53 min
Sample wt	1.74413 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	13.550 sq.m/g
Micropore Surface Area	6.979 sq.m/g
Micropore Volume	0.00304 ml/g
Correlation Coefficient	0.99920

t-Plot Surface Area Report

t-Plot Surface Area	13.550 sq.m/g
Total Surface Area	20.529 sq.m/g
Micropore Surface Area	6.979 sq.m/g
Micropore Volume	0.00304 ml/g
Film Thickness Range	0.35 to 0.50
Slope	8.7589
Intercept	1.96813
Correlation Coefficient	0.99920

Film Thickness (nm)	vads (cc/g STP)
0.3521	5.027
0.3679	5.183
0.3813	5.313
0.3945	5.437
0.4090	5.564
0.4226	5.684
0.4354	5.794
0.4615	6.007
0.4877	6.218

Isotherm Data

Freespace Calculation

Slope	0.0200
Intercept	0.0157
Correlation Coefficient	0.99993

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SAA Results Cont.

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	754.92
0.0001	0.247	0.067	754.90
0.0002	0.502	0.169	754.94
0.0004	0.794	0.315	754.85
0.0005	1.097	0.356	754.87
0.0007	1.460	0.496	754.86
0.0008	1.799	0.611	754.85
0.0012	2.153	0.919	754.83
0.0018	2.527	1.350	754.80
0.0030	2.911	2.264	754.79
0.0049	3.305	3.696	754.79
0.0083	3.695	6.296	754.73
0.0139	3.991	10.455	754.71
0.0218	4.235	16.433	754.66
0.0273	4.358	20.600	754.61
0.0333	4.472	25.164	754.59
0.0404	4.581	30.486	754.54
0.0476	4.681	35.924	754.51
0.0551	4.772	41.538	754.48
0.0600	4.828	45.247	754.44
0.0701	4.930	52.883	754.37
0.0805	5.027	60.716	754.32
0.1000	5.183	75.459	754.28
0.1180	5.313	88.976	754.24
0.1365	5.437	102.973	754.19
0.1576	5.564	118.879	754.15
0.1781	5.684	134.278	754.13
0.1977	5.794	149.098	754.08
0.2383	6.007	179.713	754.04
0.2791	6.218	210.447	753.99
0.3174	6.411	239.293	753.96
0.3595	6.623	271.056	753.94
0.3990	6.819	300.791	753.86
0.4405	7.027	332.100	753.85

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Surface Area Analyses Results:
NL-ENDP-19PB-631.1-633.8 < #35

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	631	Start Date	06/12/04
Customer	CNWRA	Start Time	19:50:34
Operator	MR	Elapsed Time	57 min
Sample Wt	1.74043 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	15.685 sq.m/g
Micropore Surface Area	7.508 sq.m/g
Micropore Volume	0.00330 ml/g
Correlation Coefficient	0.99969

t-Plot Surface Area Report

t-Plot Surface Area	15.685 sq.m/g
Total Surface Area	23.193 sq.m/g
Micropore Surface Area	7.508 sq.m/g
Micropore Volume	0.00330 ml/g

Film Thickness Range	0.35 to 0.50
Slope	10.1392
Intercept	2.13112
Correlation Coefficient	0.99969

Film Thickness (nm)	Vads (cc/g STP)
0.3527	5.690
0.3679	5.855
0.3812	6.000
0.3940	6.134
0.4091	6.290
0.4223	6.424
0.4361	6.559
0.4609	6.802
0.4867	7.051

Isotherm Data

Freespace Calculation

Slope	0.0166
Intercept	0.0057

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SAA Results cont.

Correlation Coefficient 0.99999

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	758.42
0.0001	0.253	0.054	758.47
0.0002	0.511	0.132	758.45
0.0003	0.807	0.260	758.48
0.0004	1.112	0.292	758.54
0.0005	1.478	0.398	758.55
0.0006	1.817	0.443	758.60
0.0008	2.275	0.641	758.65
0.0011	2.655	0.871	758.65
0.0018	3.052	1.353	758.66
0.0029	3.464	2.170	758.64
0.0048	3.887	3.614	758.62
0.0086	4.305	6.556	758.57
0.0143	4.603	10.863	758.56
0.0223	4.852	16.880	758.43
0.0277	4.980	20.978	758.36
0.0342	5.100	25.899	758.25
0.0413	5.216	31.291	758.14
0.0487	5.323	36.902	758.07
0.0565	5.420	42.811	758.02
0.0614	5.481	46.555	757.93
0.0697	5.571	52.820	757.91
0.0812	5.690	61.520	757.84
0.1001	5.855	75.847	757.78
0.1178	6.000	89.282	757.73
0.1357	6.134	102.847	757.67
0.1577	6.290	119.504	757.61
0.1777	6.424	134.610	757.59
0.1987	6.559	150.529	757.51
0.2374	6.802	179.782	757.45
0.2776	7.051	210.277	757.38
0.3185	7.303	241.194	757.31
0.3591	7.554	271.961	757.27
0.3996	7.803	302.620	757.23
0.4405	8.055	333.529	757.20

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Surface Area Analysis Results
 NG-EWDP-19PB-408.5-409.9-#35

Coulter SA 3100 Surface Area and Pore Size Analyzer
 Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	408-5	Start Date	06/07/04
Customer	CNwRA	Start Time	20:11:41
Operator	MR	Elapsed Time	51 min
Sample wt	1.57334 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	28.978 sq.m/g
Micropore Surface Area	12.239 sq.m/g
Micropore Volume	0.00530 ml/g
Correlation Coefficient	0.99956

t-Plot Surface Area Report

t-Plot Surface Area	28.978 sq.m/g
Total Surface Area	41.217 sq.m/g
Micropore Surface Area	12.239 sq.m/g
Micropore Volume	0.00530 ml/g

Film Thickness Range	0.35 to 0.50
Slope	18.7317
Intercept	3.42550
Correlation Coefficient	0.99956

Film Thickness (nm)	Vads (cc/g STP)
0.3521	9.981
0.3682	10.313
0.3803	10.553
0.3943	10.827
0.4081	11.094
0.4220	11.354
0.4357	11.606
0.4605	12.049
0.4867	12.504

Isotherm Data

Freespace Calculation

Slope	0.0180
Intercept	0.0026

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SAA Results Cont.

Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	774.33
0.0000	0.284	0.024	774.30
0.0001	0.568	0.062	774.37
0.0002	0.895	0.128	774.39
0.0002	1.262	0.135	774.43
0.0002	1.671	0.183	774.39
0.0002	2.131	0.192	774.44
0.0003	2.641	0.254	774.48
0.0004	3.066	0.277	774.46
0.0006	3.702	0.427	774.47
0.0007	4.175	0.575	774.52
0.0010	4.675	0.810	774.52
0.0016	5.204	1.217	774.52
0.0024	5.758	1.820	774.57
0.0035	6.336	2.702	774.63
0.0053	6.935	4.133	774.57
0.0086	7.533	6.675	774.65
0.0122	7.894	9.419	774.63
0.0170	8.224	13.195	774.67
0.0232	8.527	17.967	774.69
0.0274	8.695	21.200	774.64
0.0322	8.862	24.975	774.60
0.0375	9.024	29.050	774.64
0.0431	9.181	33.381	774.63
0.0487	9.328	37.709	774.60
0.0548	9.470	42.450	774.59
0.0590	9.564	45.696	774.58
0.0634	9.659	49.125	774.63
0.0702	9.794	54.384	774.56
0.0804	9.981	62.275	774.59
0.1005	10.313	77.846	774.60
0.1165	10.553	90.267	774.54
0.1362	10.827	105.467	774.55
0.1564	11.094	121.101	774.53
0.1771	11.354	137.176	774.47
0.1982	11.606	153.507	774.47
0.2368	12.049	183.354	774.45
0.2775	12.504	214.937	774.43
0.3180	12.943	246.249	774.38
0.3590	13.384	278.015	774.37
0.3992	13.801	309.112	774.33
0.4410	14.234	341.460	774.31

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Surface Area Analysis Results:
NC-FWDP-19PB-373.6-374.0 <#35

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.13
Sample ID	373-6	Start Date	06/07/04
Customer	CNWSA	Start Time	18:44:04
Operator	MR	Elapsed Time	44 min
Sample wt	1.61023 g	Outgas Time	360 min
Profile	BET5-T	Outgas Temperature	200 C

Summary

t-Plot Surface Area Report

t-Plot Surface Area	23.481 sq.m/g
Micropore Surface Area	9.866 sq.m/g
Micropore Volume	0.00427 ml/g
Correlation Coefficient	0.99950

t-Plot Surface Area Report

t-Plot Surface Area	23.481 sq.m/g
Total Surface Area	33.346 sq.m/g
Micropore Surface Area	9.866 sq.m/g
Micropore Volume	0.00427 ml/g

Film Thickness Range	0.35 to 0.50
Slope	15.1781
Intercept	2.76024
Correlation Coefficient	0.99950

Film Thickness (nm)	Vads (cc/g STP)
0.3517	8.065
0.3684	8.345
0.3808	8.543
0.3945	8.763
0.4079	8.975
0.4226	9.195
0.4365	9.397
0.4593	9.731
0.4860	10.105

Isotherm Data

Freespace Calculation

Slope	0.0176
Intercept	0.0018
Correlation Coefficient	1.00000

Isotherm Data Table

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SAA Results cont.

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	769.55
0.0000	0.280	0.019	769.59
0.0001	0.560	0.043	769.61
0.0001	0.882	0.062	769.66
0.0002	1.242	0.134	769.71
0.0002	1.645	0.137	769.74
0.0003	2.092	0.206	769.82
0.0003	2.485	0.242	769.83
0.0005	3.043	0.404	769.88
0.0008	3.479	0.592	769.94
0.0012	3.938	0.899	770.00
0.0019	4.419	1.427	770.10
0.0029	4.925	2.216	770.17
0.0046	5.444	3.516	770.23
0.0077	5.974	5.921	770.31
0.0115	6.325	8.853	770.35
0.0171	6.640	13.161	770.41
0.0244	6.918	18.781	770.47
0.0291	7.067	22.385	770.45
0.0344	7.214	26.523	770.50
0.0405	7.358	31.243	770.58
0.0469	7.494	36.155	770.68
0.0533	7.622	41.043	770.68
0.0577	7.706	44.444	770.69
0.0626	7.792	48.218	770.72
0.0705	7.922	54.341	770.74
0.0800	8.065	61.660	770.74
0.1008	8.345	77.662	770.71
0.1173	8.543	90.391	770.74
0.1365	8.763	105.204	770.66
0.1560	8.975	120.241	770.65
0.1781	9.195	137.292	770.72
0.1994	9.397	153.650	770.67
0.2348	9.731	180.945	770.64
0.2765	10.105	213.075	770.60
0.3198	10.479	246.434	770.63
0.3591	10.821	276.659	770.51
0.3984	11.153	306.968	770.44
0.4421	11.508	340.592	770.44

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END OF SCIENTIFIC NOTEBOOK 811

CONTINUED IN SCIENTIFIC NOTEBOOK 836

