308 --- Q200204170002
Scientific Notebook # 309

21
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PAUL BERTETTI - 522-5228

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Investigator	signature	Page initial
Paul Bertetti	Paul Bertetti	PB
ROBERT CHERRINGTON	RC	RC
Bradley Werling	Bradley M Werling	BMW
James D. Pinkerton	James D. Pinkerton	JP

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3/18/99 PB

Preparation of calcite for use in Np sorption experiments

The initial substrate to be used for the next set of Np sorption experiments will be calcite. Two forms of calcite will be used during the investigation in order to examine the possibility of changes in sorption behavior due to the source of calcite. Although the samples of calcite will differ in purity, it is hoped that the magnitude of sorption observed will depend primarily on differences in surface area between samples. Experiments that investigate coprecipitation of Np w/ calcite will be conducted (started) after initial data from the sorption experiments are collected.

To start, experimental solutions will be adjusted so that they are in equilibrium with the calcite substrate. Ideally this will prevent precipitation / dissolution reactions from masking the sorption behavior of Np on the calcite.

Two forms of calcite will be used initially. One will be a purchased "reagent grade" form of $CaCO_3$ while the other will be purchased as iceland spar xtls from a mineral supply company (OJ minerals - on order). Reagent grade $CaCO_3$ was ordered from Fisher Scientific. Six 500-gram aliquots were ordered, each with a separate lot no. and packaging.

In their studies of the sorption of divalent metals on calcite, Zachara et al. (1991) recommend modifying reagent grade calcite by "aging" or recrystallization to produce a more uniform particle size and a more stable surface.

3/18/99 PB

To do this, they recommend storing the calcite reagent in 0.02 m $NaHCO_3$ (aq) for 30 days, a procedure from Reddy and Nancollas (1971). They used 1 L of solution for 500 g of $CaCO_3$. I will use the same ratio but in smaller batches. 125 g in ~~3.6 L~~ 3.5 L of solution.

Formula wt. of $NaHCO_3$ = 84.01

to make ~~3.6~~ 3.5 L of 0.02 m $NaHCO_3$:

$$\frac{0.02 \text{ moles } NaHCO_3}{L} \cdot 3.5\,L \cdot \frac{84.01\ g}{\text{mole } NaHCO_3} = 5.8807\ g\ NaHCO_3$$

=> 5.88 g of $NaHCO_3$ required for each 3.5 L of solution.

To approximate the mixture - I will use the Mettler 4600 to weigh the solution in order to better approximate 3.5 L rather than measure liquid volume.

reagents: Fisher $NaHCO_3$ Lot no. 936893
Fisher $CaCO_3$ Lot no. 986396
nanopure H_2O

In a 600 mL beaker, 5.99 g $NaHCO_3$ was added to ~300 mL of H_2O. The solution was mixed and added to a 2 gal (~4 L) PP bottle (wide mouth). The 2 gal bottle had been tared so that a total of 3500 g of

references: Zachara J.M., Cowan C.E. and Resch C.T. 1991. Sorption of divalent metals on calcite. Geochim. Cosmochim. Acta (55) pp. 1549-1562.
Reddy M.M. and Nancollas G.R. 1971. The crystallization of calcium carbonate 1. Isotopic exchange and kinetics. J. Colloid Inter. Sci. (36) pp. 166-173.

3/18/99 PB

solution were added. A second solution of 5.85 g $NaHCO_3$ in 3500 g solution was also made. ~~Amt~~ 3/19/99 PB 125 g of $CaCO_3$ (measured by weight in a polypropylene beaker) was then added to each 2 gal container. Solid and solution were thoroughly agitated and place on a gyratory shaker at ~80 rpm for 20 minutes.

19 Mar 99 PB

mixed $CaCO_3$ | 0.02 m $NaHCO_3$ solutions by swirling, and rotating on shaker for 20 min.

22 Mar 99 PB

mixed $CaCO_3$ solutions (swirled by hand) mixed on shaker for 20 min. Removed and replaced caps to exchange CO_2 gas in head space. The solutions are supposed to equilibrate w/ atm CO_2.
mixing will continue daily for about 30 days.

PB
9/11/00

7/23/99
pe

Preparation of ^{233}U for Uranoplane Synthesis

Objective: Convert nitrate-form ^{233}U spike into acetate-form in dilute acetic acid. The ^{233}U uranyl acetate will be used for the synthesis of uranoplane. The LSA will be used to quantitate the U.

Equip. + Supplies:
- Orion pH meter 920-A + combination probe (ROSS 8103)
- pH buffers
- Corning hot plate/stirrer
- Mettler 4600 analytical balance
- Mettler 240 analytical balance
- 250 mL glass beaker
- 25 mL volumetric flask
- Eppendorf pipettors with tips
- Oxford pipettors w/ tips
- LSA sample vials (7 mil)
- Ultima Gold AB LSA cocktail - LOT NO. 91-5031
- Kimwipes
- Waste bottle

Reagents:
- 0.1 M HNO_3
- 0.01 M acetic acid - prepared 7/15/99, RC from lot # 971798
- 6 M acetic acid - prepared 7/15/99, RC from lot # 971798
- concentrated acetic acid (glacial) LOT NO. 971798
- ^{233}U spike (nitrate-form) - 50 ppb prepared 8/21/98, A.J.
- ultrapure H_2O

acetic 1B 03/09/00

7/23/99
pe

Procedure:

Overview: An aliquot of ^{233}U spike (as uranyl nitrate in dilute nitric acid) is evaporated to dryness by heating and taken up in glacial acetic acid. Successive evaporation and refluxing in diluted concentrations of acetic acid result in an aliquot of ^{233}U spike as uranyl acetate in dilute acetic acid, a form compatible for addition to an existing uranoplane synthesis recipe.

1. Tared a 250 mL beaker and weighed out 25.0315 g ^{233}U (nitrate-form) spike solution - 50 ppb, prepared 8/21/98 by A.J.

2. Placed beaker containing ^{233}U soln. on a hot plate. Heated slowly to evaporate the solution, swirling occasionally to prevent scale build-up. White crystals formed evenly on bottom surface of beaker.

3. Added approximately 10 mL glacial acetic acid to the nearly dry crystals in the beaker. Stirred + Agitated 7/23/99 solution until all the crystals were dissolved.

4. Took up residue in approximately 6 M acetic acid and warmed mixture until nearly dry.

5. Took up residue in approximately 10 mL of 0.01 M acetic acid and swirled contents until crystals dissolved. Heated solution until near dry.

6. Added another 10 mL approximate of 0.01 M acetic acid and warmed mixture until crystals were dissolved. Poured solution from beaker into 25 mL volumetric flask,

continued →

7/23/99 nc

Procedure – cont'd.

6. cont'd.
rinsed beaker with successive 5 mL aliquots of .01 M acetic acid until vol. flask was filled to mark.

7. Transferred contents of 25 mL vol. flask into a 30 mL polypropylene container and labeled as "^{233}U in 0.01 M acetic acid."

8. Added 0.5 mL of 0.1 M HNO_3 to four LSA sample vials and 10 mL of 0.1 M HNO_3 to one LSA sample vial to be used as the blank.

Weighed the sample vials + HNO_3:

Sample ID	Weight
U-NO_3 1	7.8041 g
U-NO_3 2	7.7768 g
U-Ac 1	7/23/99 nc ~~7.7236 g~~ 7.8234 g
U-Ac 2	7/23/99 nc ~~7.8296 g~~ 7.8587 g
Blank	—

9. Added 0.5 mL of original nitrate from ^{233}U spike to two LSA vials (labeled U-NO_3 1+2) and 0.5 mL acetate from ^{233}U to two LSA vials (labeled U-Ac 1+2).

Weighed sample vials after addition of ^{233}U spikes:

Sample ID	Weight	Wt. of Spike
U-NO_3 1	8.3090 g	0.5049 g
U-NO_3 2	8.2796 g	0.5028 g
7/23/99 U-~~NO_3~~Ac 1	7/23/99 nc ~~7.7236 g~~ 8.2246 g	0.5012 g
U-Ac 2	7/23/99 nc ~~7.8295 g~~ 8.3600 g	0.5013 g

7/23/99 nc

Procedure: cont'd.

7/23/99 ~~4. cont'd~~

10. added 5 mL of Ultima Gold cocktail to all 5 LSA sample vials and placed capped vials in sample cassette for LSA ~~7/23/99~~ 7/23/99 analysis.

12. Calibrated pH meter with 4.0, 7.0, and 10.0 buffers and measured pH of prepared ^{233}U spike in 0.01 N acetic acid.

pH = 3.27 slope = 97.8

7/27/99 nc

Results:

Analysis by Packard 2500 TR/AB Liquid Scintillation Analyzer

24 Jul 1999 14:28 ALPHA/BETA - 1.09
Protocol #:22 U-233 check

nc 7/27/99

Time: 200.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	350	0	0.0	19.02
Region B:	100 -	350	0	2.0	3.10
Region C:	0.0 -	2000	0	0.0	24.93

Quench Indicator: SIS

7/27/99 nc background

S#1 = ~~U-Ac 1~~

S#2 = U-NO_3 1 nc 7/27/99

S#3 = U-NO_3 2

S#4 = U-Ac 1

S#5 = U-Ac 2

S#	TIME	CPMA	CPMB	B:2S%	CPMC	SIS	FLAG
1	200.00	19.02	3.10	8.03	24.93	150.89	B
2	19.04	526.30	522.11	2.01	527.95	604.99	
3	18.77	534.36	529.88	2.01	534.20	602.35	
4	19.42	514.55	512.04	2.01	516.57	608.25	
5	19.33	517.19	514.28	2.01	519.25	607.50	

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 24-Jul-1999 15:52
C14 Eff (0-156 keV) = 96.44 %
C14 CHI SQUARE IPA DATA PROCESSED - 24-Jul-1999 16:03
C14 Chi Square = 16.17
H3 IPA DATA PROCESSED - 24-Jul-1999 16:04
H3 Eff (0-18.6 keV) = 65.25 %
H3 CHI SQUARE IPA DATA PROCESSED - 24-Jul-1999 16:15
H3 Chi Square = 21.61
BKG IPA DATA PROCESSED - 24-Jul-1999 17:15
Bkg (0-18.6 keV) = 16.20 cpm
Bkg (0-156 keV) = 23.13 cpm
C14 E^2/B (1-156 keV) = 526.83
H3 E^2/B (1-18.6 keV) = 262.88

Experiment will need to be duplicated. As from ^{233}U yielded lower cpm alpha results.

7/27/99 AC

Analysis of $CaCO_3$ samples (GC DIV. 20 525/23) for Ca concentration using the Perkin Elmer 3100 Atomic Absorption Spectrometer.

Prepared Ca^{+2} standards for calibrating the A.A. 7/27/99

$$5\ ppm = \frac{5\ mL \times 1{,}000\ ppm\ Ref.\ Std.\ Ca.}{1L\ (1\%\ potassium\ soln)}$$

Pipetted with vol. pipette (class A) 5 mL of Ca reference solution (Lot No. 786835-24, Exp. date Oct 2000) into 1L vol. flask. Filled to mark with 1% K soln. as KCl. (Lot No. 885967)

$$4\ ppm = \frac{40\ mL \times 5\ ppm}{50\ mL}$$

$$2\ ppm = \frac{40\ mL \times 5\ ppm}{100\ mL}$$

$$0.5\ ppm = \frac{10\ mL \times 5\ ppm}{100\ mL}$$

$$0.2\ ppm = \frac{10\ mL \times 5\ ppm}{250\ mL}$$

7/28/99 AC

Results: 7/28/99 Absorbance Concentration (ppm)

Standard (ppm)
0.2
0.5
2.0
4.0
5.0

Sample	Trial 1	Trial 2	Trial 3	Avg.
A	1.3	1.2	1.3	1.3
B	1.3	1.3	1.2	1.3
C	1.4	1.4	1.4	1.4
D	1.2	1.2	1.2	1.2
E	1.2	1.2	1.2	1.2
F	1.1	1.1	1.1	1.1
1	1.0	1.0	1.0	1.0
2	1.0	1.0	1.0	1.0

7/28/99 AC

Results: cont'd.

Concentration

Sample	ppm			Avg.
1-A	0.1	0.1	0.1	0.1
1-B	0.1	0.1	0.1	0.1

Standard Absorbances

Standard ppm	Absorbance		
0.2	.008	.009	.008
0.5	.020	.021	.019
2.0	.070	.069	.071
4.0	.165	.162	.163
5.0	.224	.221	.223

$CaCO_3$ Samples

Sample	Trial 1	Trial 2	Trial 3
A	.047	.048	.049
B	.050	.048	.049
C	.054	.053	.055
D	.047	.047	.046
E	.048	.047	.047
F	.046	.045	.046
7/28/99 1	.045	.046	.044
2	.047	.046	.046
1-A	.008	.008	.008
1-B	.009	.008	.009

Samples were prepared by pipetting (Eppendorf pipetter and plastic tip) 1 mL of each sample and 5 mL of sample 1-A & 1-B into individually labeled 10 mL volumetric flasks, then adding to mark with 1% potassium soln. as KCl.
KCl - Fisher Chemical, Lot No. 885967

7/28/99
pc

pH measurements of $CaCO_3$ solution samples as analyzed using Orion model 920A pH meter and Orion 8103 ross combination electrode w/ ATC probe:

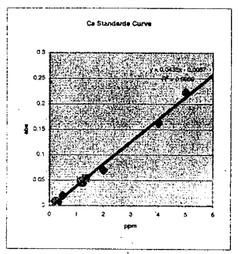
Sample	pH
A	8.348
B	8.382
C	8.363
D	8.321
E	8.382
F	8.334
1	8.384
2	8.438
1-A	9.271
1-B	9.270

Calcium conc. results of A.A. Analysis:

pc 7/28/99

Calcium Concentration in CaCO3 Soln. Samples

Ca++ Standards (ppm)	Absorbance	Average Absorbance
0.2	0.008	
0.2	0.009	
0.2	0.008	0.008333
0.5	0.02	
0.5	0.021	
0.5	0.019	0.02
2	0.07	
2	0.069	
2	0.071	0.07
4	0.165	
4	0.162	
4	0.163	0.163333
5	0.224	
5	0.221	
5	0.223	0.222667



Sample I.D.	Absorbance Trial 1	Trial 2	Trial 3	Average	pH	Measured Conc. (ppm)	Concentration x Dil. Factor
A	0.047	0.048	0.049	0.048	8.348	1.23	12.26027
B	0.05	0.048	0.049	0.049	8.382	1.25	12.48858
C	0.054	0.053	0.055	0.054	8.363	1.36	13.63014
D	0.047	0.047	0.046	0.046667	8.321	1.20	11.95586
E	0.048	0.047	0.047	0.047333	8.382	1.21	12.10807
F	0.046	0.045	0.046	0.045667	8.334	1.17	11.72755
1	0.045	0.046	0.044	0.045	8.384	1.16	11.57534
2	0.047	0.046	0.046	0.046333	8.438	1.19	11.87976
1-A	0.008	0.008	0.008	0.008	9.271	0.31	0.625571
1-B	0.009	0.008	0.009	0.008667	9.27	0.33	0.656012

7/27/99 ~~Results: contd. from pg. 7, Preparation of 2334 for Hamptons Synthesis~~ 7/27/99 pc
pc

8/2/99
pc

Preparation of cation standard solution checks to be analyzed together with calcite samples:

① Tared 100 mL volumetric flask using Mettler PM4600 balance. Using a class A volumetric pipette, added 10 mL Instrument Check Standard 1, lot no. 15-18AS, recorded weight as 10.03 g. Partially filled flask with D.I. water then added 0.2 mL HNO_3 (1+1) to preserve solution. Filled to mark with D.I. water. Labeled flask contents as "Std. #1 soln A".

② Tared 100 mL volumetric flask. Added 5 mL Instrument check Standard 4, lot no. 15-19AS. Weighed as 5.04 g. Partially filled flask w/ D.I. water, added 0.2 mL HNO_3 (1+1). Filled to mark w/ D.I. water. Labeled flask as "Std. #4 soln. A".

③ Tared 100 mL vol. flask. added 10 mL Std. #4 soln A. Weighed as 10.01 g. Partially filled w/ D.I. water, added 0.2 mL HNO_3 (1+1), then filled to mark w/ D.I. water. Labeled as "Std. #4 soln. B".

④ Preparation of Sample Soln. No. 1:
a) added 10 mL ~~Std. #1 soln A~~ 15-15AS to tared ~~100~~ 500 mL vol. flask. Weighed ~~10.00~~ 10.27 g. pc 8/3/99 pc 8/3/99
b) added 5 mL Std 5, lot no. 13-146AS to tared flask. Weighed 5.03 g.
c) added 10 mL Std. #4 soln B to tared flask. Weighed 10.00 g
d) added 1.0 mL HNO_3 (1+1) then filled to mark with D.I. water. Labeled "Sample Soln. #1".

1+1 HNO_3 - Mfg. Fisher Scientific - prepared by P. Bertetti Lot No. 118110 Trace Metal Grade 5/12/99

8/4/99
~~7/29/99~~ RC
RC

⑤ Preparation of sample soln. #2:
a) added 10 mL Std #1 soln. A to tared 1L volumetric flask. Weighed 10.01 g.
b) added 20 mL Std. #4 soln A to tared flask. Weighed 20.03 g
c) added 100 mL ~~Std #~~ alternate Metals III, lot no. 16-87AS, to tared flask. Weighed 100.65 RC 8/4/99
d) added 2.0 mL HNO3 (1+1), then filled to mark with D.I. water. Labeled as Sample Soln. #2.

RC 8/4/99 ⑥

⑥ Preparation of sample soln. # 3:
a) added 100 mL alternate Metals III to tared 500 mL volumetric flask. Weighed ~~100.99 g~~ 101.07 g RC 8/19/99
b) added 10 mL Std #1, lot no. 15-18AS, weighed 10.09 g.
c) added 25 mL Std. #5, lot no. 13-146AS, weighed 25.21 g.
d) added 5 mL Std. #4 soln. A, Weighed 5.01 g.
e) added 1 mL HNO3 (1+1) to flask and filled to mark w/ D.I. water. Labeled flask as Sample Soln. #3.

RC 8/4/99

8/6/99
~~7/27/99~~
RC
OK [illegible]
03/04/02

Summary of Cation concentrations in prepared Stds.

Prepared Cation Standard Solutions from Reference Standards

RC 8/6/99

	Cation Standard Soln. No. 1	Cation Standard Soln No. 2	Cation Standard Soln. No. 3
Analyte	Concentration (ppb)	Concentration (ppb)	Concentration (ppb)
Ag	20.56	9.99	200.79
Al	392.71	9.97	200.39
As	20.56	10.15	204.02
Ba	407.07	10.05	202.00
Be	10.29	9.99	200.79
Cd	10.08	9.92	199.54
Co	103.11	10.07	202.41
Cr	20.99	10.00	200.99
Cu	51.86	10.04	201.80
Mn	30.05	10.04	201.80
Ni	82.30	10.06	202.20
Pb	61.95	10.01	201.19
Sb	122.66	10.04	201.80
Se	10.08	10.07	202.41
Tl	122.12	10.09	202.81
V	102.28	10.01	201.19
Zn	40.34	9.97	200.39
Mo	151.20	10.04	511.24
Sn	99.90	0.00	500.67
Sr	100.70	0.00	504.70
Ti	100.70	0.00	504.70
Th	1.01	10.06	5.03
U	1.01	10.06	5.03
	(ppm)	(ppm)	(ppm)
Ca	10.28	50.14	100.70
K	10.19	10.04	20.17
Mg	10.34	10.06	20.20
Na	10.13	50.48	101.38

8/23/99
RC

A.A. Analysis of Cation Check Standards

Purpose: Determine Ca Concentrations of prepared Cation check standards as analyzed using Perkin Elmer 3100 atomic absorption spectrometer.

Procedure:
① Prepared 1L of 1% ~~LaCl3·7H2O~~ RC 8/16/99 Lanthanum as LaCl3·7H2O by weighing 30g and adding to 1L volumetric flask, then added 2 mL HNO3 as a preservative. Filled to mark w/ DI water.

8/23/99
~~7/27/99~~ rr
rr

Procedure - cont'd.

② Prepared Ca standards from ref. std. by adding to vol. flask and filling to mark with prepared La+HNO3 soln. as follow:

a) 4 ppm Ca std. - 4 mL ref. std (1,000 ppm) to 100 mL
b) 1 ppm Ca std. - 25 mL (4 ppm above) to 100 mL
c) 0.5 ppm Ca std. - 25 mL (4 ppm) to 200 mL
d) 0.2 ppm Ca std. - 10 mL (4 ppm) to 200 mL

③ Diluted Cation Check Stds. to within instrument sensitivity range as follow:

a) Ck Std No. 1 (10.28 ppm) x dil factor 4 = 2.57 ppm
b) Ck Std No. 2 (50.14 ppm) x dil factor 20 = 2.507 ppm
c) Ck Std No. 3 (100.70 ppm) x dil factor 50 = 2.014 ppm

Data:

Ca Standard	Absorbance Trial 1	Trial 2	Trial 3	Trial 4
0.20 ppm	0.008	0.007	0.007	0.008
0.50 ppm	0.023	0.022	0.022	0.023
1.0 ppm	0.049	0.048	0.049	0.050
4.0 ppm	0.178	0.180	0.181	0.180

8/23/99 rr

Cation Ck. Std	Measured Conc. Trial 1	Trial 2	Trial 3	Avg. Conc. ~~x Dil. Factor~~
No. 1	2.61	2.51	2.49	2.54
No. 2	2.57	2.50	2.51	2.53
No. 3	2.04	2.03	2.03	2.03

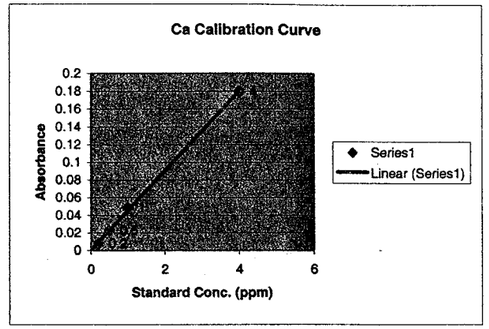
8/23/99
~~7/27/99~~ rr
rr

Summary of A.A. calibration for Ca^{++} and results of Cation Check Standards analyses for Ca concentration:

rr
8/23/99

Cation Check Std. Solns.

Ca++ Standards (ppm)	Absorbance	Average Absorbance
0.2	0.008	
0.2	0.007	
0.2	0.007	0.007333
0.5	0.023	
0.5	0.022	
0.5	0.022	0.022333
1	0.049	
1	0.048	
1	0.049	0.048667
4	0.178	
4	0.18	
4	0.181	0.179667



Sample I.D.	Concentration Trial 1	Trial 2	Trial 3	Measured Conc.	X Dil. Factor (ppm)
1	2.61	2.51	2.49	2.54	10.2
2	2.57	2.5	2.51	2.53	50.6
3	2.04	2.03	2.03	2.03	101

Reagents used for above analysis:

Lanthanum Chloride ($LaCl_3 \cdot 7H_2O$)
Fisher Chemical
Lot No. 915032A

Calcium reference std
SPEX Centiprep
Lot No. CLI-147 CA
Exp. date 5/15/00

HNO_3 - Concentrated Trace Metal Grade
Fisher Chemical
Lot. No. 118110

8/25/99 Well Water
~~8/24/99~~ nc
nc

~~CaCO3~~ nc 8/25/99 samples to be sent to Div. 01 for analysis of cation species. Summary of prepared samples is as follows:

Sample ID	Description 8/25/99 nc ~~Prepared~~	Date ~~Prepared~~ Collected
95*FA-2(4)	Filtered, acidified well NC-EWDP-9S Zone 4	5/19/99
95*UA(4)	Well NC-EWDP-9S Zone 4, unfiltered, acidified	5/19/99
~~9S*U~~ 8/25/99 95*U(4)	Well NC-EWDP-9S Zone 4, unfiltered	5/18/99
1S*U(shallow)	Well NC-EWDP-1S shallow zone, unfiltered	5/18/99
1S*U*A(shallow)	Well NC-EWDP-1S shallow zone, unfiltered, acidified	5/18/99
1S*FA-2(shallow)	Well NC-EWDP-1S shallow zone, filtered, acidified	5/19/99
1S*FA-2(deep)	Well NC-EWDP-1S deep zone, filtered acidfied	5/17/99
1S*U*A(deep)	Well NC-EWDP-1S (deep) 8/25/99 nc deep zone, unfiltered acidified	5/17/99

8/25/99
~~8/24/99~~ nc
nc

Sample ID	Description	Date Collected
1S* U (Deep)	Well NC-EWDP-1S deep zone, unfiltered, not preserved	5/17/99

Summary of samples sent to Div 01 for analyses:

CNWRA
1. Pena Blanca anion 172/12
2. Pena Blanca cation 172/12
3. Std. #5 anion
4. 1S FA-2 (shallow)
5. 1S UA (shallow)
6. 1S ~~UA~~ 8/25/99 U (shallow)
7. 9S FA-2 (4)
8. 9S UA (4)
9. 1S U (deep) anion
10. 9S U (4)
11. 1S U (deep) anion
12. 9S FA-2 (4)
13. 1S UA (deep)
14. 1S U (shallow)
15. 1S FA-2 (shallow)
16. 9S UA (4)
17. Std. #2 309/12
18. 9S U (4)
19. 9S U (4) anion
20. Std. #4
21. 1S FA-2 (deep)
22. 1S UA (deep)
23. Std. #3 309/12
24. Std. #1 309/11
25. 1S U (deep)
26. Std. #1 309/11

(7) (28) (29) (30) (31) (32) (33) (34)

8/25/99
7/27/99 RC
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Listing of standard solns. sent to Div. 01 with well water samples for analysis:

Cation Ch. Std. No. 1 (GC 309/11) CNWRA-STD. 1

Cation Ch. Std. No. 2 (GC 309/12) CNWRA-STD. 2

Cation Ch. Std No. 3 (GC 309/12) CNWRA-STD. 3

Cation Ch. Std. No. 4 S.P.E.X. Certiprep. CNWRA-15-33A5

Anion Ch. Std. No. 5 S.P.E.X. Certiprep CNWRA-16-20A5

~~CNWRA-STD. 4~~ 8/25/99

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8/25/99

9/8/99
RC

Tc Sorption on Clinoptilolite Experiment No. 1

Procedure:

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Tc Sorption on Clinoptilolite
Sorption Experiment - TcC

WRITTEN BY: F.P. Bertetti
REVISION NO.: 0

DATE WRITTEN: June 6, 1994
DATE REVISED: N/A

CONDITIONS:

1. Σ Tc = 300 ppb
2. 0.1 m NaCl matrix, equilibrium with atmospheric CO_2(g); $PCO_2=10^{-3.5}$
3. pH range 3 – 9
4. Initial solution volume = 20 ml, initial clinoptilolite mass = 0.4 g

OBJECTIVES:

1. To investigate the characteristics of Tc sorption on clinoptilolite as a function of solution pH. Experimental data will be correlated with technetium aqueous speciation and compared with results of Tc sorption on γ-alumina and sorption results from published data.
2. To investigate the reproducibility and reversibility of Tc sorption reactions.

EQUIPMENT:

Gyratory shaker
Packard 2505 TR/AB liquid scintillation counter
Orion pH/mV/ISE/°C meter
Combination pH electrode
various Eppendorf micropipettors and Oxford macropipettors for solution transfer
ATC probe
Repipettor for transfer of scintillation cocktail
Analytical balances (Mettler 4600 and 240AE)
α, β and γ survey instruments

SUPPLIES:

40-ml polycarbonate Oak Ridge-type test tubes (acid washed and dried)
FEP beaker (acid cleaned and dried)
Eppendorf and Oxford pipette tips
pH buffer solutions
Ultima-Gold AB liquid scintillation cocktail
7-ml scintillation vials
weighing paper and boats (as necessary)
Na-clinoptilolite
reagent grade NaOH and HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water

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PROCEDURE:

Special considerations:

Experimental solutions and sample containers/vials should be weighed at each step. Do not add or subtract contents without weighing before and after each process. Always record weight of solutions on most sensitive balance available.

When measuring pH, minimize the amount of time the glass electrode is in contact with the Tc bearing solution. Make sure to rinse the electrode thoroughly before measuring another solution. Take care not to introduce lint particles or other foreign objects into the experimental or sample containers.

Sample vials should "rest" in the absence of light for at least 24 hours prior to initial analysis to allow for decay of incident radiation pulses.

Following each sampling period, swipes and frisks of the work area should be performed. If contamination is found, follow the radiological procedures for clean up. Radioactive solutions may not be disposed of without following all radiation safety guidelines. Do not dispose of any solutions without prior approval of the division RSPOC.

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. Use degassed ultrapure water to make the NaOH solutions. The NaOH solutions should be store in tightly capped glass reagent bottles.. Record lot numbers and preparation steps in the scientific notebook. The following stock solutions are required:

1) 0.1 M HCl stock solution
2) 0.01 M HCl stock solution
3) 1.0 M NaOH stock solution
4) 0.1 M NaOH stock solution

B. Experimental Solution Preparation

1. Label twenty-seven (27) 40-ml polycarbonate test tubes (e.g., TcC-pH*i* - where *i* is the approximate pH of each solution, see **Table 1**). Measure and record the weight of each test tube with cap (use Mettler AE240 balance).

2. Add clinoptilolite to each experimental solution container
   a. Transfer ~0.400 ± 0.001 g of Na-clinoptilolite to each test tube.
   b. Measure and record weight of test tube following addition of clinoptilolite.

3. Add Tc solution to each experimental container.
   a. Add ~20 g of the 300 ppb Tc stock solution to each test tube. Use an Oxford pipettor and a 10-ml tip to transfer 2 10-ml aliquots of Tc stock solution to each test tube.
   b. Following addition of the Tc stock solution, measure and record weight of each test tube.

4. Adjust pH of experimental solutions.
   a. Use an Eppendorf micropipettor and associated tip to add HCl or NaOH to each experimental test tube. The concentration and approximate volume of acid or base to be added to each solution is given in Table 1.

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   b. Record the amount and concentration actually added to each container. Mix well by swirling the solutions.
   c. Measure and record weight of each container following pH adjustment.

C. Start Sorption Experiment.

1. Place experimental test tubes in secure holder. Ensure that caps are loosely fitted to allow for gas exchange with atmosphere.

2. Place holder with tubes on a gyratory shaker, and set the shaker speed at ~120 rpm on the continuous setting.

3. Allow the experimental solutions and clinoptilolite to equilibrate for 10 to 14 days.

4. Sample Tc stock solution to determine initial Tc concentration for experimental solutions.
   a. Label (e.g., TcC-Ia and TcC-Ib) and pre-weigh 2, 7-ml liquid scintillation vials.
   b. Using an Eppendorf pipette, withdraw 0.5 ml of the Tc stock solution and transfer to a liquid scintillation vial. Repeat for the second vial. Measure and record weight of both vials following addition of Tc stock solution.
   c. Add 5 ml of Ultima-Gold AB cocktail to each vial. Homogenize samples and set aside for analysis.
   d. Analyze initial Tc samples

D. Measure Equilibration pH and Tc Concentration of Experimental Solutions.

1. Following the 10 to 14 day equilibration period, measure and record weight of each solution container. Be careful to cap solutions tightly before handling.
2. Measure and record the pH of each solution. Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.
3. *If pH measurements indicate the expected values and range given in* ***Table 1****, proceed with sampling for Tc concentration. If pH measurements indicate that solutions may not be in equilibrium with atmosphere, contact the principal investigator for instructions.*
4. Withdraw samples for LSA
   a. For each sample solution label two 7-ml LSA vials (e.g., TcC-3a and TcC-3b). Measure and record the weight of each vial.
   b. From each sample container, withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials. Measure and record weight of each vial after addition of experimental solution sample.
   c. Measure and record the weight of each experimental container following pH and LSA sample withdrawal.
   d. Add 5 ml of Ultima-Gold AB cocktail to each vial, homogenize the vial contents, and set aside for counting.
   e. Record weight of each sample container after pH measurement and sampling for LSC counting.
5. Measure Tc in sample vials using LSA.

If analytical results are deemed satisfactory, reversibility and reproducibility tests will be performed as provided in a separate procedure.

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Table 1. Polycarbonate test tube labels, estimated solution pH, and volume of HCl or NaOH solutions needed for adjustment of pH in 0.1m NaCl solutions with 300 ppb Tc. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated solution pH	Volume of HCl needed, ml	Molarity
TcC-pH3	3.00	0.250	0.10
TcC-pH3.25	3.25	0.150	0.10
TcC-pH3.5	3.50	0.090	0.10
TcC-pH3.75	3.75	0.050	0.10
TcC-pH4	4.00	0.030	0.10
TcC-pH4.25	4.25	0.020	0.10
TcC-pH4.5	4.50	0.125	0.01
TcC-pH4.75	4.75	0.090	0.01
TcC-pH5	5.00	0.075	0.01
TcC-pH5.25	5.25	0.060	0.01
TcC-pH5.5	5.50	0.050	0.01
TcC-pH5.75	5.75	0.045	0.01
TcC-pH6	6.00	0.040	0.01
TcC-pH6.25	6.25	0.025	0.01
TcC-pH6.5	6.50	0.010	0.01
TcC-pH6.75	6.75	0.000	n/a
Sample Label	**Estimated solution pH**	**Volume of NaOH needed, ml**	**Molarity**
TcC-pH7	7.00	0.010	0.10
TcC-pH7.25	7.25	0.020	0.10
TcC-pH7.5	7.50	0.040	0.10
TcC-pH7.75	7.75	0.075	0.10
TcC-pH8	8.00	0.125	0.10
TcC-pH8.25	8.25	0.250	0.10
TcC-pH8.5	8.50	0.450	0.10
TcC-pH8.75	8.75	0.085	1.0
TcC-pH9	9.00	0.165	1.0
TcC-pH9.25	9.25	0.340	1.0
TcC-pH9.5	9.50	0.750	1.0

Preparation of HCl and NaOH solutions as reagents for adjusting the pH of Tc solutions in individual test tube containers.
RC 9/8/99

Equipment + materials:
- Kimax 34/28 bubbler apparatus
- 1-L erlenmeyer flask w/rubber stopper
- Corning heating plate/stirrer
- Tygon tubing
- Metal measuring spatula
- 1-250 mL vol. flask
- 1-100 mL vol. flask
- 4-200 mL vol. flask
- Volumetric pipettes

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Reagents:
- Hydrochloric acid - conc. ACS Plus
  Fisher Chemical
  Lot No. 956110
- *~~not used~~ – Sodium hydroxide - ACS pellets
  RC 9/8/99
  Fisher Chemical
  Lot No. 976631
- D.I. Ultrapure water
- Dilute - 1L analytical conc. 1 unit ea. of 0.1N + 0.02N NaOH
  Lot No. H33121 + D23109

Procedure:

1. Removed CO_2 from approx. 1.3 L of D.I. water by transferring water to a 1-L erlenmeyer flask and placing on a hot plate. Water was brought to boiling and held at 100°C for 20 minutes. Flask was removed from hot plate and was allowed to cool for 10 minutes. A no-hole rubber stopper was placed onto the flask.

2. A Kimax 34/28 bubbler container was filled with approx. 250 mL of D.I. water and a stirring rod was inserted. With the container on a stir plate NaOH pellets were transferred into the container until dissolution became difficult. The glass cap and ceramic frit bubbling tube was fitted on the container and a short piece of tygon tubing was connected between the bubbling apparatus and the filter flask connection.

3. The prepared CO_2 free water was allowed to cool with the CO_2 air scrubbing device connected for approx. 2 hrs. The water was then transferred to a ~~1-L~~ RC 9/8/99 vol. flask containing one unit of 200 mL

9/8/99 RC

Diluted-lt NaOH analytical concentrate, 0.02 N. Flask was filled to mark w/ CO_2 free H_2O. Liquid was transfered to a 250 mL glass stoppered storage container and labeled as 0.02 N NaOH. RC 9/8/99

4. One Dilut-It NaOH analytical concentrate, 0.1 N was added to a 100 mL vol. flask and CO_2 free H_2O was added to the mark. Liquid was transfered to a 100 mL glass storage bottle & labeled as 1.0 N NaOH.

5. ~~Pipetted 25 mL using a vol. pipette into~~ RC 9/8/99 Measured 16.6 mL conc. HCl with a 25mL graduated cylinder and transfered into a 200 mL vol. flask. Rinsed the graduated cylinder with D.I. water into the flask, and then filled to mark w/ D.I. water. Transfered liquid into a 200 mL glass container and labeled as 1.0 N HCl.

6. Pipetted 20 mL ~~of~~ 1.0 N HCl using a vol. pipette into a 200 mL vol. flask and filled to mark w/ D.I. H_2O. Transfered to a 200 mL glass container and labeled as 0.10 N HCl. OK 03/08/02 BB

7. ~~Lipo~~ 9/8/99 Pipetted 20 mL of 0.10 N HCl into a 200 mL vol. flask using a vol. pipette. Filled to mark w/ D.I. water and labeled as 0.01 N HCl.

RC 9/8/99

9/12/99 RC

Data:

Sample I.D	Weight of empty PC tube w/cap (grams) Wt.	Wt. of PC tube w/cap+clino. (grams) Wt
TcC-pH3	22.0507	22.4533
TcC-pH3.25	21.9857	22.3948
TcC-pH3.5	21.9791	22.3847
TcC-pH3.75	21.9595	22.3657
TcC-pH4	22.0549	22.4620
TcC-pH4.25	22.0871	22.4941
TcC-pH4.5	22.1586	22.5607
TcC-pH4.75	22.4019	22.8069
TcC-pH5	22.2646	22.6715
TcC-pH5.25	21.9944	22.4050
TcC-pH5.5	22.0027	22.4100
TcC-pH5.75	22.3917	22.7974
TcC-pH6.	22.4129	22.8192
TcC-pH6.25	22.3939	22.8001
TcC-pH6.5	22.3260	22.7314
TcC-pH6.75	22.3740	22.7809
TcC-pH7	22.3236	22.7296
TcC-pH7.25	21.9992	22.4054
TcC-pH7.5	22.0078	22.4132
TcC-pH7.75	22.0858	22.4919
TcC-pH8	22.2120	22.6173
TcC-pH8.25	22.4057	22.8106
TcC-pH8.5	21.7848 RC 9/12/99	22.3987 RC 9/12/99
TcC-pH8.75	22.0161	22.4222
TcC-pH9	22.2304	22.6353
TcC-pH9.25	22.2477	22.6535
TcC-pH9.5	22.1271	22.5336

9/14/99 RC

Data - contd.

Wt. of PC tube + Cline + Tc spike* (grams)

TcC-pH3	42.3203	TcC-pH 6.25	42.6596
TcC-pH3.25	42.2373	TcC-pH 6.5	42.6094
TcC-pH3.5	42.2450	TcC-pH 6.75	42.6545
TcC-pH3.75	42.1958	TcC-pH 7.0	42.5813
TcC-pH4	42.3084	TcC-pH 7.25	42.2516
TcC-pH4.25	42.3333	TcC-pH 7.5	42.2618
TcC-pH4.5	42.5326	TcC-pH 7.75	42.3628
TcC-pH4.75	42.6612	TcC-pH 8	42.4560
TcC-pH5	42.5070	TcC-pH 8.25	42.6958
TcC-pH5.25	42.2472	TcC-pH 8.5	42.3063
TcC-pH5.5	42.2523	TcC-pH 8.75	42.2563
TcC-pH5.75	42.6390	TcC-pH 9	42.4899
TcC-pH6	42.6507	TcC-pH 9.25	42.5936
		TcC-pH 9.5	42.3928

* Spike 42A(031/289) prepared 6/23/99, $^{99}Tc = 5 \times 10^{-3}$ mCi/g; 2.95×10^{-4} M in 0.1 m NaCl

9/15/99 RC

pH adjustment of Tc solutions in PC tubes:

Sample I.D.	Desired pH	pH soln added	Quantity increments (mL) (~~mL~~) pc 9/14/99
TcC-pH3	3.00	0.10M HCl	9/14/99 rc ~~250~~ 250
TcC-pH3.25	3.25	0.10m HCl	150
TcC-pH3.5	3.50	0.10m HCl	50, 20, 20
TcC-pH3.75	3.75	0.10m HCl	50
TcC-pH4	4.00	0.10m HCl	20, 10
TcC-pH4.25	4.25	0.10m HCl	20
TcC-pH4.5	4.50	0.01M HCl	100, 25
TcC-pH4.75	4.75	0.01M HCl	50, 20, 20
TcC-pH5	5.00	0.01M HCl	75

9/15/99 RC

Data - contd.

Sample I.D.	Desired pH	pH soln added*	Quantity increments (mL)
TcC-pH5.25	5.25	0.01M HCl	50, 10
TcC-pH5.5	5.50	0.01M HCl	50
TcC-pH5.75	5.75	0.01M HCl	25+20
TcC-pH6	6.00	0.01M HCl	20, 20
TcC-pH6.25	6.25	0.01M HCl	25
TcC-pH6.5	6.50	0.01M HCl	10
TcC-pH6.75	6.75	N/A	∅
TcC-pH7	7.00	rc ~~0.01~~ 0.10M NaOH 9/15/99	10
TcC-pH7.25	7.25	0.10M NaOH	20
TcC-pH7.5	7.50	0.10M NaOH	20, 20
TcC-pH7.75	7.75	0.10M NaOH	50, 25
TcC-pH8	8.00	0.10M NaOH	100, 25
TcC-pH8.25	8.25	0.10M NaOH	250
TcC-pH8.5	8.50	0.10M NaOH	250, 200
TcC-pH8.75	8.75	1.0M NaOH	75, 10
TcC-pH9	9.00	1.0M NaOH	100, 25, 20, 20
TcC-pH9.25	9.25	1.0M NaOH	300, ~~100~~ rc 9/15/99 20, 20, 100 rc
TcC-pH9.5	9.50	1.0M NaOH	500, 250

* pH soln. reagents prepared 9/8/99 (309/22)

RC 9/15/99

9/15/99 nc

Data - contd.

Wt. of PC sample tubes after pH adjustment:

Sample I.D.	Weight (grams)
TcC-pH3	42.5701
TcC-pH3.25	42.3784
TcC-pH3.5	42.3349
TcC-pH3.75	42.2486
nc 9/15/99 TcC-pH4	~~42.2466~~ nc 9/15/99 42.3291
TcC-pH4.25	42.3544
TcC-pH4.5	42.6552
TcC-pH4.75	42.7664
TcC-pH5	42.5721
TcC-pH5.25	42.3063
TcC-pH5.5	42.3021
TcC-pH5.75	42.6819
TcC-pH6	42.6886
TcC-pH6.25	42.6859
TcC-pH6.5	42.6200
TcC-pH6.75	42.6550
TcC-pH7	42.5919
TcC-pH7.25	42.2727
TcC-pH7.5	42.3021
TcC-pH7.75	42.4378
TcC-pH8	42.5798
TcC-pH8.25	42.9348
TcC-pH8.5	42.7630
TcC-pH8.75	42.3464
TcC-pH9	42.6577
TcC-pH9.25	43.0571
TcC-pH9.5	43.1606

9/23/99 nc

Preparation of initial Tc spike for ESA analysis:

	Wt. of 7mL scintillation vial w/cap	Wt. of vial + 0.5mL Tc spike
TcC-IA	7.3167 g	7.8246 g
TcC-IB	7.2938 g	7.8001 g

Wt. of Tc 0.5mL spike additions:

TcC-IA = 0.5079 g

TcC-IB = 0.5063 g

10/1/99 nc

Data:

Sample I.D.	Wt. of PC tube after pH adjustment (grams)	Sample I.D.	Wt. of PC tube after pH adj (g)
TcC-pH3	41.9440	TcC-pH7.25	41.8909
TcC-pH3.25	41.9939	TcC-pH7.5	41.9449
TcC-pH3.5	41.8915	TcC-pH7.75	42.0049
TcC-pH3.75	41.8565	TcC-pH8	42.1958
TcC-pH4	41.6320	TcC-pH8.25	42.3512
TcC-pH4.25	41.8808	TcC-pH8.5	42.3263
pp 03/09/00 TcC-pH4.5	42.0637	TcC-pH8.75	41.9533
Tc ~~TcC~~-pH4.75	42.3789	TcC-pH9	42.2456
TcC-pH5	42.1223	TcC-pH9.25	42.5097
TcC-pH5.25	41.9749	TcC-pH9.5	42.7034
TcC-pH5.5	41.6969		
TcC-pH5.75	42.3950		
TcC-pH6	42.3264		
TcC-pH6.25	42.0425		
TcC-pH6.5	42.3161		
TcC-pH6.75	42.3634		
TcC-pH7	41.9916		

nc 10/1/99

10/1/99 RC

Data: cont'd ~~10/1/99 RC 10/1/99~~ PRD 03/08/02

Sample — pH measurement after equil. period (grams)

TcC-pH 3

10/1/99 RC

Calibration of Orion model 920A pH meter prior to taking pH measurements of Tc soln. after equilibrium pH adjustments.

pH buffer 3.00 Fisher Chemical Lot No. 977121-24
pH buffer 7.00 Fisher Chemical Lot No. 986993-24
pH buffer 10.00 Fisher Chemical Lot No. 981233-24

Fresh pH buffers were used. Changed 10/1/99.

2 point calibration, buffers 3.00 and 7.00
slope = 100.2

Sample	pH Value	Sample	pH Value
TcC-pH3	3.74	TcC-pH6	
TcC-pH3.25	4.11	TcC-pH6.25	
TcC-pH3.5	4.56	TcC-pH6.5	
TcC-pH3.75	4.89	TcC-pH6.75	
TcC-pH4	5.56	TcC-pH7	
TcC-pH4.25	5.98	TcC-pH7.25	
TcC-pH4.5	6.26	TcC-pH7.5	
TcC-pH4.75	6.27	TcC-pH7.75	
TcC-pH5	6.43	TcC-pH8	
TcC-pH5.25	6.51	TcC-pH8.25	
TcC-pH5.5		TcC-pH8.5	
TcC-pH5.75		TcC-pH8.75	
		TcC-pH9	
TcC-pH9.5		TcC-pH9.25	

RC 10/1/99

RC 10/1/99

10/4/99 RC

10/1/99 RC

~~Vial I.D. Vial Wt. Vial Wt. + Sample~~ RC 10/1/99

Ran out of time at end of day to finish pH measurements and sampling. Will reweigh all samples.

Sample Name	10/4/99 RC Wt. of PC tube after equil., grams	10/4/99 Ph Measured Value	10/4/99 Wt. of PC tube after sampling, grams
TcC-pH 3	41.8235	3.685	40.7965
TcC-pH 3.25	41.8696	4.721	40.8559
TcC-pH 3.5	41.7416	4.512	40.7207
TcC-pH3.75	41.7512	4.851	40.7283
TcC-pH 4	41.5168	~~5.93~~ 5.343 10/4/99 RC	40.5536
TcC-pH 4.25	41.7623	6.011	40.7382
TcC-pH 4.5	41.9579	6.321	40.9244
TcC-pH 4.75	42.1827	6.344	41.1679
TcC-pH 5	42.0260	6.494	41.0315
TcC-pH 5.25	41.8738	6.473	40.8457
TcC-pH 5.5	41.6055	6.512	40.5780
TcC-pH 5.75	10/4/99 RC 42.2849	6.486	41.3162
TcC-pH 6	42.2621	6.290	41.2383
TcC-pH 6.25	41.9532	6.659	40.9307
TcC-pH 6.5	42.2400	6.425	41.2133 RC 10/4/99
TcC-pH 6.75	42.2839	6.107	41.2466
TcC-pH 7	41.9338	6.920	40.9061
TcC-pH 7.25	41.8017	6.703	40.7839
TcC-pH 7.5	41.8423	7.388	40.7907
TcC-pH 7.75	41.8996	7.603	40.8674
TcC-pH 8	42.1141	10/4/99 RC ~~7.603~~ 7.604	41.0590
TcC-pH 8.25	42.2859	10/4/99 RC ~~7.804~~ 8.048	41.2514
TcC-pH 8.5	42.2139	8.392	41.1898
TcC-pH 8.75	41.8714	8.671	40.8464
TcC-pH 9	42.0816	8.882	41.0662
TcC-pH 9.25	42.3994	9.406	41.3769
TcC-pH 9.5	42.6070	9.942	41.5901

OK 3/08/02 [initials]

10/8/99 [initials]

Data -contd.

Sample Name	Wt. of sample vial A, grams (10/4/99)	Wt. of sample vial B, grams (10/4/99)	Wt. of vial A + Sample, grams (10/4/99)	Wt. of vial B + Sample, grams (10/4/99)
TcC-pH 3	7.2120	7.2225	7.8097	7.8287
TcC-pH 3.25	7.2546	7.2781	7.8528	7.7784
TcC-pH 3.5	7.3624	7.3251	7.8637	7.8270
TcC-pH3.75	7.2887	7.2092	7.7872	7.8106
TcC-pH 4	7.2823	7.3380	7.7789	7.7701
TcC-pH 4.25	7.3917	7.3601	7.8889	7.8594
TcC-pH 4.5	7.2429	7.3251	7.8391	7.8236
TcC-pH 4.75	7.2887	7.3622	7.7857	7.8609
TcC-pH 5	7.2729	7.3093	7.7727	7.8579
TcC-pH 5.25	7.4021	7.2495	7.8991	7.7497
TcC-pH 5.5	7.2744	7.3127	7.7707	7.8128
TcC-pH 5.75	7.2588	7.2598	7.7572	7.7607
TcC-pH 6	7.2170	7.3001	7.7156	7.8009
TcC-pH 6.25	7.2111	7.3186	7.7080	7.8182
TcC-pH 6.5	7.2983	7.2457	7.7960	7.8466
TcC-pH 6.75	7.3281	7.3804	7.8263	7.8820
TcC-pH 7	7.3359	7.3583	7.8334	7.8577
TcC-pH 7.25	7.3975	7.2767	7.8412	7.7725
TcC-pH 7.5	7.3749	7.3304	7.8721	7.8192
TcC-pH 7.75	7.3613	7.3409	7.8593	7.8409
TcC-pH 8	7.2490	7.2863	7.7463	7.7857
TcC-pH 8.25	7.2632	7.2801	7.7595	7.7790
TcC-pH 8.5	7.3261	7.2733	7.8219	7.7716
TcC-pH 8.75	7.2767	7.3645	7.7736	7.8629
TcC-pH 9	7.3113	7.2717	7.8085	7.7712
TcC-pH 9.25	7.2400	7.2604	7.7280	7.7612
TcC-pH 9.5	7.2345	7.3277	7.7224	7.8268

Added A-B Gold cocktail to samples, mixed by hand shaking, and place under LSA darkened cover at 1700, 10/4/99.

10/5/99 [initials]

Data -contd.

Analysis results of Tc/Clino. solutions as analyzed using the Packard 2500 TR/AB liquid scintillator analyzer:

SYSTEM NORMALIZED

[initials] 10/5/99

C14 IPA DATA PROCESSED - 05-Oct-1999 13:39
C14 Eff (0-156 keV) = 96.38 %
C14 CHI SQUARE IPA DATA PROCESSED - 05-Oct-1999 13:50
C14 Chi Square = 12.90
H3 IPA DATA PROCESSED - 05-Oct-1999 13:51

[initials] 10/5/99

05 Oct 1999 13:51 ALPHA/BETA - 1.09 Page #3

H3 Eff (0-18.6 keV) = 65.16 %
H3 CHI SQUARE IPA DATA PROCESSED - 05-Oct-1999 14:01
H3 Chi Square = 16.82
BKG IPA DATA PROCESSED - 05-Oct-1999 15:02
Bkg (0-18.6 keV) = 17.85 cpm
Bkg (0-156 keV) = 26.07 cpm
C14 E^2/B (1-156 keV) = 466.85
H3 E^2/B (1-18.6 keV) = 237.46

06 Oct 1999 07:51 ALPHA/BETA - 1.09 Page #1
Protocol #:35 Tc 99 CPM User : Bob Cherring

Time: 999.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	296	0	2.0	19.68
Region B:	0.4 -	296	0	0.0	19.58
Region C:	0.0 -	2000	0	0.0	25.13

Quench Indicator: SIS
Tc-99 analysis by CPM from 0 to 296 keV
Luminescence Correction On
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: c:\Tc99\PROT.DAT
Count Data Filename: c:\Tc99\SDATA35.DAT
Spectrum Data Drive & Path: c:\Tc99

[initials]

10/5/99 Results:
pc

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	FLAG
1	999.00	19.68	1.43	19.58	1.43	25.13	1.26	116.21	B
2	1.96	5079.81	2.01	5079.91	2.01	[illegible]	2.01	174.03	
3	1.94	5186.61	2.00	5155.88	2.00	[illegible]	2.00	174.41	
4	1.92	5200.63	2.01	5200.73	2.01	[illegible]	2.01	171.67	
5	1.92	5194.38	2.01	5194.48	2.01	5194.68	2.01	174.67	
6	1.91	5220.63	2.01	5220.73	2.01	[illegible]	2.01	174.67	
7	1.91	5242.10	2.00	5242.20	2.00	5245.02	2.00	172.24	
8	1.90	5257.69	2.00	5257.37	2.00	5256.97	2.01	174.34	
9	1.92	5214.70	2.00	5214.80	2.00	5212.89	2.00	171.71	
10	1.89	5291.43	2.00	5291.53	2.00	5289.15	2.01	175.30	
11	2.15	4645.44	2.01	4644.79	2.01	4647.42	2.01	176.58	
12	1.95	5125.45	2.00	5125.14	2.00	5122.56	2.01	176.21	
13	1.94	5140.63	2.01	5139.90	2.01	5141.34	2.01	175.80	
14	1.97	5074.23	2.00	5073.92	2.00	5074.36	2.01	174.89	
15	1.91	5234.77	2.00	5234.87	2.00	5239.78	2.00	173.54	
16	1.93	5190.17	2.00	5190.27	2.00	5187.82	2.00	173.50	
17	1.92	5190.22	2.01	5189.48	2.01	5192.52	2.01	174.91	
18	1.95	5121.55	2.01	5120.22	2.01	5121.53	2.01	175.32	
19	1.76	5672.93	2.01	5673.03	2.01	5672.02	2.01	170.92	
20	1.89	5283.50	2.01	5282.33	2.01	5280.69	2.01	173.72	
21	1.96	5101.24	2.00	5100.52	2.00	5101.40	2.01	173.81	
22	1.88	5312.24	2.01	5311.06	2.01	5311.57	2.01	175.47	
23	1.88	5313.83	2.00	5313.93	2.00	5314.76	2.01	174.60	
24	1.94	5146.30	2.01	5145.99	2.01	5146.00	2.01	173.15	
25	1.91	5236.86	2.00	5236.55	2.00	5239.26	2.00	175.09	
26	1.93	5186.54	2.00	5186.64	2.00	5188.86	2.00	173.63	
27	1.92	5207.40	2.00	5206.67	2.00	5206.12	2.01	172.81	
28	1.86	5361.50	2.01	5361.60	2.01	5363.58	2.01	172.78	
29	1.88	5319.68	2.00	5318.93	2.00	5320.41	2.00	172.74	
30	1.93	5181.36	2.00	5180.21	2.00	5182.64	2.00	171.38	
31	1.95	5120.83	2.01	5120.52	2.01	5123.07	2.01	173.24	
32	1.93	5166.85	2.01	5166.53	2.01	5167.61	2.01	172.68	
33	1.93	5171.51	2.01	5171.20	2.01	5171.76	2.01	173.55	
34	1.90	5272.95	2.00	5272.21	2.00	5278.02	2.00	172.75	
35	1.90	5245.58	2.01	5245.26	2.01	5246.02	2.01	175.84	
36	1.93	5181.88	2.00	5181.98	2.00	5181.08	2.00	174.55	
37	1.94	5145.27	2.01	5144.96	2.01	5143.42	2.01	175.68	

10/5/99
pc

10/5/99 Results - contd.
pc

06 Oct 1999 09:14 ALPHA/BETA - 1.09 Page #2
Protocol #:35 Tc 99 CPM User : Bob Cherrington

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	FLAG
38	1.92	5198.03	2.01	5197.30	2.01	5200.39	2.01	175.81	
39	1.94	5157.64	2.00	5156.50	2.00	5158.89	2.00	172.94	
40	1.91	5218.54	2.01	5217.80	2.01	5217.80	2.01	174.33	
41	1.91	5235.82	2.00	5235.92	2.00	5239.26	2.00	175.70	
42	1.92	5193.34	2.01	5193.02	2.01	5194.14	2.01	175.94	
43	1.94	5162.28	2.00	5162.38	2.00	5163.01	2.00	173.19	
44	1.92	5207.92	2.00	5207.61	2.00	5210.80	2.00	174.43	
45	1.91	5245.77	2.00	5245.87	2.00	5247.12	2.00	174.15	
46	1.92	5191.78	2.01	5191.46	2.01	5192.57	2.01	175.93	
47	1.97	5063.57	2.01	5062.86	2.01	5062.18	2.01	176.75	
48	1.96	5106.85	2.00	5106.54	2.00	5105.48	2.00	173.73	
49	1.94	5133.93	2.01	5133.20	2.01	5137.75	2.01	174.02	
50	1.92	5205.32	2.00	5204.59	2.00	5202.99	2.01	176.49	
51	1.90	5268.22	2.00	5267.47	2.00	5269.60	2.00	173.64	
52	1.96	5082.36	2.01	5082.46	2.01	5083.03	2.01	174.45	
53	1.91	5243.15	2.00	5242.41	2.00	5241.88	2.00	175.94	
54	2.03	4907.91	2.01	4907.22	2.01	4908.86	2.01	174.63	
55	2.00	5005.82	2.00	5005.12	2.00	5006.37	2.00	172.58	
1 MISSING TUBE(S)									
57	1.90	5265.06	2.00	5264.74	2.00	5265.92	2.00	173.60	
58	1.96	5084.40	2.01	5084.50	2.01	5085.07	2.01	171.33	

10/5/99
pc

pc
10/5/99

PB 03/08/02
investigation

10/13/99
RC

Objective: The Tc solutions from the previous Tc/Cyno. experiment are suspect to having reached equilibrium with the added pH buffers after the 14 day stabilization period. An investigation will begin to determine whether air forced into the sample containers during the entire equilibration period is more effective than using the loosely-capped method.

Materials & equipment:

Small aquarium pump - Second Nature Whisper 300
¼" polycarbonate tubing (manifold)
PE tubing (leader lines)
¼" poly check valve
¼" end ~~cap~~ plug RC 10/13/99
Polycarbonate test tubes - 40mL w/caps
Ring stand w/support bar and clamp
Twist ties
1-Liter volumetric flask
Analytical balance - Mettler PM4600

Reagents:

Sodium Chloride
Fisher Chemical
Lot No. 984321

O.I. ultrapure H_2O

HCl Concentrated A.C.S.
Fisher Chemical
Lot. No. 956110

10/12/99
RC

Procedure: An air bubbling device was constructed by taking a piece of ¼" PC tubing and punching small holes through one side at appropriate spacing using the sharp tip of a pair of tweezers. A 6" length of the PE leader tubing was cut for each hole and forced into the PC manifold pipe.

A poly end plug was inserted in one end of the manifold pipe and the other end was connected to the air pump. A flow check valve was placed in-line to prevent an accidental syphoning of sample liquids into the apparatus.

RC ~~Each leader tube end was placed into~~
10/12/99

The manifold pipe was secured to a horizontally mounted support bar which was clamped to a ring support. The manifold was held onto the support bar using twist-ties.

15 - 40 mL PC test tubes were each filled with 20mL of O.I. water and labeled as pH 3.0 to pH 9.5 individually in 0.5 increments. pH buffers were added in amounts according to Table 1 (309/22). A leader tube from the manifold pipe was placed into the liquid of each test tube and the cap was twisted lightly onto each test tube to secure the leader tube. Care has to be taken not to pinch the tube. This was best accomplished by tightening the caps while the air pump was running and observing whether sufficient air bubbles were forming.

The air flow through each leader tube appeared to be about equal for each of the 15 test tubes.

10/13/99 mc

Procedure – cont'd.

pH measurements were taken about 24 hrs. after pH buffers had been added. Tube caps were screwed on tight during the 24 hours. No air had been bubbled into the liquid.

Results of pH measurements:

Sample	pH value
pH 3.0	2.88
pH 3.5	3.34
pH 4.0	3.84
pH 4.5	4.22
pH 5.0	4.42
pH 5.5	4.58
pH 6.0	4.74
pH 6.5	5.25
pH 7.0	7.25
pH 7.5	9.95
pH 8.0	10.79
pH 8.5	11.27
pH 9.0 10/13/99	11.93
pH 9.5 10/13/99	12.49

mc ~~12.48~~ 10/13/99

Prepared a 0.1 N NaCl solution and placed 20 mL into each of 3 pc 10/13/99 test tubes. Added NaOH buffer soln. in appropriate quantities (.309/22) to read pH 8.5, pH 9.0, and pH 9.5. These samples were placed into the test tube rack, replacing the previously prepared pH 3.5, pH 5.0, and pH 6.5 test samples. These samples were labeled pH 8.5 NaCl, pH 9.0 NaCl, and pH 9.5 NaCl, respectively.

Leader tubes were secured and air turned on at 1745.

10/15/99 pc

Data re: pH measurements of solutions equilibrated with bubbling air.

Sample	pH value
8.5 pc 10/15/99	
pH 8.0 NaCl	8.38
pH 9.0 NaCl	8.92
pH 9.5 NaCl	9.65
pH 3.0	2.90
pH 3.5 mc 4.0	3.86
pH 4.5	4.28
pH 5.5	4.65
pH 6.75	4.76

Remaining samples were not measured since the concentration of H^+ probably changed per sample due to evaporation losses. This H_2O loss cannot be determined because the original sample wts. were not measured.

Another experiment will be performed using pH adjusted NaCl soln. (0.1 N) for all samples. Control samples will be included using ~~tightly capped~~ samples, loosely capped samples, ~~bubbled~~ air in the liquid samples, and ~~bubbled~~ air pumped into 10/15/99 the non-liquid occupied top space of the sample container.

Weights will be carefully monitored at each addition/subtraction step performed.

The feeder tube will be included as part of the sample container assembly.

10/18/99 nc

Results: Summary

10/21/99 nc

pH Equilibrium Experiment

10/18/99 nc 10/21/99 nc 10/21/99 nc

Sample I.D.	Wt.of empty container, grams	Wt. after add. of NaCl soln., grams	Wt.after add. of pH buffer, grams	Wt. after 2 day equil. period, grams	pH after 2 day equil. period	Wt. after pH measure., grams
4.0a	22.3093	42.2866	42.3139	39.1665	3.85	39.1108
4.0b	22.4267	42.3685	42.3905	41.4977	4.01	41.4802
4.0c	22.5067	42.4074	42.4367	42.2952	3.93	NA
4.0d	22.2180	42.0923	42.1218	42.1153	3.91	NA
5.0a	22.3202	42.1621	42.2344	38.9252	4.70	38.9252
5.0b	22.3079	42.1320	42.2041	38.6235	4.53	38.6013
5.0c	22.6102	42.4857	42.5601	42.3565	4.53	NA
5.0d	22.2922	42.1260	42.2010	42.1952	4.53	NA
8.5a	22.2648	42.1313	42.5803	41.9508	8.37	41.8428
8.5b	22.1656	42.0155	42.4648	40.0520	8.44	40.0412
8.5c	22.3457	42.2238	42.6746	42.4677	8.62	NA
8.5d	22.4980	42.3521	42.8027	42.7943	11.19	NA
9.0a	22.3048	42.1830	42.3529	40.6853	8.93	40.5776
9.0b	22.2912	42.1428	42.3174	38.9169	9.50	38.8972
9.0c	22.1760	42.0380	42.2067	42.0823	11.00	NA
9.0d	22.4403	42.3359	42.3952	42.3882	11.80	NA
9.5a	22.5852	42.4606	42.5759	39.4407	9.42	39.4407
9.5b	22.3349	42.2475	42.3241	39.7558	10.20	39.7347
9.5c	22.2974	42.1945	42.2475	42.7587	12.21	NA
9.5d	22.2838	42.1648	42.1960	42.9343	12.53	NA

Note: Container weights include test tube, cap and leader tube

a = air bubbled into soln.
b = air exchanged above soln. surface
c = loosely capped container
d = tightly capped container

10/21/99 nc

10/27/99 nc

pH equilibrium experiment:

Data:

Sample	pH meas. after 4 days equil.	pH meas. after 7 day equil	Wt (g)
4.0 b	3.96	10/27/99 ~~nc 4.24~~ 3.64	30.7376
5.0 b	4.46	10/27/99 ~~nc 3.6~~ 4.21	31.7010
8.5 b	8.47	8.58	33.2983
9.0 b	8.99	9.03	35.3462
9.5 b	9.79	9.52	27.9592

Summary of pH Equilibrium Data

pH Equilibrium Experiment

Sample I.D.	Wt.of empty container, grams	Wt. after add. of NaCl soln., grams	Wt. of NaCl soln., grams	Wt. after add. of pH buffer, grams	Wt. of pH buffer soln., grams	Wt. after 3 day equil. period, grams	Wt. loss during equil. period, grams	pH after 3 days equil. period	Wt. after pH measure, grams	Wt. loss due to pH meas., grams	pH after 4 days equil. period	pH after 7 days equil. period	Wt. after pH measure, grams	Wt. loss during equil. period, grams
4.0a	22.3093	42.2866	19.9773	42.3139	0.0273	39.1665	3.1474	3.85	39.1108	0.0557	na	na	na	#VALUE
4.0b	22.4267	42.3685	19.9418	42.3905	0.0220	41.4977	0.8928	4.01	41.4802	0.0175	3.96	3.64	30.7376	10.7426
4.0c	22.5067	42.4074	19.9007	42.4367	0.0293	42.2952	0.1415	3.93	na	#VALUE!	na	na	na	#VALUE!
4.0d	22.2180	42.0923	19.8743	42.1218	0.0295	42.1153	0.0065	3.91	na	#VALUE!	na	na	na	#VALUE!
5.0a	22.3202	42.1621	19.8419	42.2344	0.0723	38.9487	3.2857	4.70	38.9252	0.0235	na	na	na	#VALUE!
5.0b	22.3079	42.1320	19.8241	42.2041	0.0721	38.6235	3.5806	4.53	38.6013	0.0222	4.46	4.21	31.7010	6.9003
5.0c	22.6102	42.4857	19.8755	42.5601	0.0744	42.3565	0.2036	4.53	na	#VALUE!	na	na	na	#VALUE!
5.0d	22.2922	42.1260	19.8338	42.2010	0.0750	42.1952	0.0058	4.53	na	#VALUE!	na	na	na	#VALUE!
8.5a	22.2648	42.1313	19.8665	42.5803	0.4490	41.9508	0.6295	8.37	41.8428	0.1080	na	na	na	#VALUE!
8.5b	22.1656	42.0155	19.8499	42.4648	0.4493	40.0520	2.4128	8.44	40.0412	0.0108	8.47	8.58	33.2983	6.7429
8.5c	22.3457	42.2238	19.8781	42.6746	0.4508	42.4677	0.2069	8.62	na	#VALUE!	na	na	na	#VALUE!
8.5d	22.4980	42.3521	19.8541	42.8027	0.4506	42.7943	0.0084	11.19	na	#VALUE!	na	na	na	#VALUE!
9.0a	22.3048	42.1830	19.8782	42.3529	0.1699	40.6853	1.6676	8.93	40.5776	0.1077	na	na	na	#VALUE!
9.0b	22.2912	42.1428	19.8516	42.3174	0.1746	38.9169	3.4005	9.50	38.8972	0.0197	8.99	9.03	35.3462	3.5510
9.0c	22.1760	42.0380	19.8620	42.2067	0.1687	42.0823	0.1244	11.00	na	#VALUE!	na	na	na	#VALUE!
9.0d	22.4403	42.3359	19.8956	42.3952	0.0593	42.3882	0.0070	11.80	na	#VALUE!	na	na	na	#VALUE!
9.5a	22.5852	42.4606	19.8754	43.2434	0.7828	39.4803	3.7631	9.42	39.4407	0.0396	na	na	na	#VALUE!
9.5b	22.3349	42.2475	19.9126	43.0241	0.7766	39.7558	3.2683	10.20	39.7347	0.0211	9.79	9.52	27.9592	11.7755
9.5c	22.2974	42.1945	19.8971	42.9759	0.7814	42.7587	0.2172	12.21	na	#VALUE!	na	na	na	#VALUE!
9.5d	22.2838	42.1648	19.8810	42.9460	0.7812	42.9343	0.0117	12.53	na	#VALUE!	na	na	na	#VALUE!

Note: Container weights include test tube, cap and leader tube

a = air bubbled into soln.
b = air exchanged above soln. surface
c = loosely capped container
d = tightly capped container

10/27/99 nc

1/12/00
RC

Preparation of 325/450 mesh pulverized clinoptilolite to be used in Tc sorption experiments.

Sample previously pulverized, screened, ultrasonically cleaned, and acid washed by A.V. 266/2.

Removal of carbonates from clino. sample:

Materials + Equipment:

Water bath - Fisher Scientific
50 mL centrifuge tubes (16)
Glass stir rod

Reagents:

Clinoptilolite - Death Valley Junction, Calif. - 77.58 grams
NaOAc buffer (pH 5) 10/1/99 A.V. - 1N
Nanopure H_2O (17.3 megaohm-cm)

Procedure:

1. Approx. 5 grams of clino. was placed in 50 mL centrifuge tubes.

2. Added 1N NaOAc soln. to 50 mL mark.

3. Placed centrifuge tubes in rack and placed in hot water bath at 95°C for about 1 hour.

4. Removed rack of tubes from bath and allowed soln. to cool.

5. Decanted NaOAc soln. into waste container.

Procedure - cont'd.

6. Added nanopure H_2O to 50 mL mark and decanted after stirring with glass stir rod. Repeated two times.

1/13/00
RC

Removal of iron oxides from clinoptilolite sample:

Materials + Equipment:

Water bath - Fisher Scientific
50 mL centrifuge tubes
Glass stir rod
Eppendorf pipettor w/ tips
Mettler analytical balance
Metal spatula
Weigh boat

Reagents:

Na-citrate (0.3M) soln. prepared 1/12/00 by RC
from Lot No. 940621 Fisher Chemical

Sodium dithionite ($Na_2S_2O_4$)
Lot No. 912722 Fisher Chemical

Sodium bicarbonate soln. (1M) ($NaHCO_3$)
Prepared 11/15/99 by A.V. 365/57

Sodium chloride soln. (NaCl) saturated
Prepared 1/13/00 by RC
Lot No. 947723 Fisher Chemical

1/13/00
pc

Procedure:

Prepared 0.3 M Na-citrate soln. by adding 88 grams of Na-citrate to 1-L volumetric flask and adding to mark with nanopure H_2O.

Added magnetic stir bar and placed on stir plate for ~30 minutes.

Poured liter flask contents into a 1-L pp storage bottle and labeled as "0.3 M Na Citrate".

1. Added 20 mL Na-citrate 0.3 M soln. to each centrifuge tube containing ~5 grams clinoptilolite from previous carbonate-removal step.

2. Added 2.5 mL $NaHCO_3$ (1 M) soln. to each tube using a micro-pipettor.

3. Placed rack of centrifuge tubes into hot water bath heated to 77°C.

4. Added 0.5 grams $Na_2S_2O_4$ to one sample tube and stirred for 1 min. using a glass stir rod with a rubber policeman tip. Mixture turned a grey-blue color and gas bubbles formed in the solution. Repeated for all 16 sample tubes.

5. Repeated step #4 two times, adding 0.5 g $Na_2S_2O_4$ each time.

6. Decanted supernatant into waste beaker. Added 10 mL of saturated NaCl soln. and let sit for 5-10 min.

Procedure - cont'd.

7. Decanted NaCl soln. into waste beaker.

8. Added ~30 mL nanopure H_2O to each sample tube and decanted two times.

Observation: Clinoptilolite samples turned a greenish/grey color after the $Na_2S_2O_4$ addition to the sample solution. Type 07 water rinses did not lighten or change the color of the solid.

1/14/00
pc

9. Added 20 mL Na-citrate + 2.5 mL $NaHCO_3$ solutions to each sample container and stirred vigorously for several minutes while heating solution in the water bath (~76°C). Decanted.

10. Repeated step 9 - No color change of solid. The chelating solution (Na-citrate + $NaHCO_3$) remained fairly clear.

11. Repeated iron reduction and chelating steps 1-6 two times. Color of clino. solid lightened to a more greenish and less grey color.

1/17/00
pc

12. Combined solid clinoptilolite from sample tubes into a glass collection jar and labeled COV*UC*WA*RC*RFE.

13. Placed jar containing sample into oven set at 65°C overnight.

1/18/00
pc

14. Dried clino. sample appeared off-white in color.

Procedure - cont'd.

1/18/00 RC

15. Washed sample with acetone through a No 5 filter paper. Rinsed 3 times with nanopure H_2O.

16. Washed sample with 0.1 M HNO_3 through a No. 5 filter paper. Rinsed 4 times using nanopure H_2O.

17. Placed jar containing clino. sample in oven set at 60°C overnight.

1/19/00 RE

18. Dried sample removed from oven, cooled, and lid placed tightly over jar. Sample appears off-white in color.

1/20/00 RE

Clino. crystals were placed under microscope. No contaminants could be observed.

1/24/00 RE

Began heavy liquid separation of iron and heavy contaminants from clinoptilolite sample:

Procedure:

1. Prepared solution of tetrabromoethane and (Fisher Chemical Lot No. 980087) N-N dimethyl formamide (Fisher Chem. Lot No. 981439) from formulation found in lab notebook 365/22:
   measured 130 mL of N-N dimethyl formamide and 264 mL tetrabromoethane using a graduated cylinder and combined in a 500 mL amber glass bottle. Labeled as "Heavy liquid - density = 2.3 g/cc".

2. Transferred approximately 150 mL of heavy liquid mixture into a 250 mL separation funnel. Added about 20 grams of clino.

Procedure - cont'd.

2. cont'd - from CDV*UC*WA*RC*RFe sample into the funnel and capped.

3. The funnel contents were thoroughly mixed by inverting the funnel several times. The mixture was allowed to rest for 30 minutes with the funnel supported in a ring stand apparatus. 1/24/00 RE

4. The bottom half of the funnel liquid was drained rapidly ~~into~~ through a No. 5 Whatman filter paper and funnel apparatus into a 250 mL beaker. The contaminants trapped in the filter paper were disposed of in the trash and the filtrate was reused for cleaning the next portion of the clino. sample. 1/24/00 RE

5. The top liquid/clino. mixture in the funnel was drained into a No. 5 Whatman filter paper apparatus. The filtered clino. ~~was placed~~ was removed in the apparatus until the full clino. sample was treated. The clean heavy liquid was reused for further clino. portions. 1/24/00 RE

1/25/00 RE

6. The above steps were repeated until the entire clino. sample was treated.

7. Acetone was poured over the clino. sample in the filter apparatus and allowed to drain completely through the mineral. Repeated 4 times using about 50 mL acetone for each rinse.

8. The clino. sample was ~~placed~~ rinsed 3 times using D.I. water, then placed in a 400 mL glass beaker. Placed the sample in the drying oven overnight. 1/24/00 RE

1/26/00 pc

Procedure - cont'd.

9. Removed clino. sample from oven and allowed to cool. Weighed sample = 03/03/00 RB

Observation: Very little amounts of contaminants ~~were~~ appeared on the filter paper following the cleaning liquid separation steps. The fine mesh size of the sample particles may prevent adequate separation.

2/1/00 pc

Observed clino. sample under the microscope and sample appears to be clean and uniform. No further cleaning of the sample will be performed.

Preparation of sodium - form clinoptilolite:

Prepared 3N NaCl soln. as per procedure from 266/8. NaCl - Fisher Chemical Lot No. 947723

Procedure:

1. Placed approximately 35g of clino. sample in a 500 mL PP bottle. Two bottles prepared.

2. added 400 mL of NaCl soln. to bottle and placed bottle in a shaker/water bath set at 90°C.

3. Samples were agitated continuously for 14 days. NaCl soln. was replaced in bottle every other day.

2/15/00 pc

4. Filtered the samples and NaCl soln. through a No. 5 Whatman filter paper/funnel into a 500 mL beaker.

Procedure cont'd -

5. Clino. sample was removed from filter and placed in a 250 mL glass beaker. Added nanopure H_2O (approx. 150 mL) and stirred with glass rod. Placed beaker in sonicator for 15 min.

6. Decanted liquid and repeated with 5 washings with nanopure H_2O.

7. Placed clino. sample into glass storage container and placed in drying oven overnight.

2/16/00 pc

8. Sample removed from oven and allowed to cool. Weighed sample = 6.9 grams. Labeled glass container as "CDV * ~~HS~~ UC * WA * RC * RF2 * HL * Naf". pc 2/16/00

2/21/00 pc

Surface area analysis of 325/450 mesh clinoptilolite sample:

pc 2/21/00

~~Wt. of empty tube assembly~~

Tube I.D.	Wt. of empty tube assembly	Sample + tube assembly	Sample Wt.
#1	33.6273 g	34.0764 g	0.4491 g
#2	33.2323 g	33.6851 g	0.4528 g
#9	33.2952 g	34.2273 g	0.9321 g

pc 2/24/00

2/22/00 pe

Results of S.A. analysis of 325/450 mesh clino:

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

pe 2/22/00

Serial No.	w46020	Software Version	2.11
Sample ID	CLINO-1	Start Date	10/21/89
Customer	CNWRA	Start Time	01:24:04
Operator	BOB	Elapsed Time	32 min
Sample Wt	0.4491 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	350 C

Correct date 10/21/99 pe

Summary

Surface Area Report

BET Surface area 20.881 sq.m/g
Correlation Coefficient 0.99969

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

pe 2/22/00

Serial No.	w46020	Software Version	2.11
Sample ID	CLINO-2	Start Date	10/21/89
Customer	CNWRA	Start Time	04:08:27
Operator	BOB	Elapsed Time	31 min
Sample Wt	0.4528 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	350 C

Correct date 10/21/99 pe

Summary

Surface Area Report

BET Surface area 20.788 sq.m/g
Correlation Coefficient 0.99967

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

pe 2/22/00

Serial No.	w46020	Software Version	2.11
Sample ID	CLINO-3	Start Date	10/21/89
Customer	CNWRA	Start Time	05:09:07
Operator	BOB	Elapsed Time	34 min
Sample Wt	0.9321 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	350 C

Correct date 10/21/99 pe

Summary

Surface Area Report

BET Surface area 19.930 sq.m/g
Correlation Coefficient 0.99971

2/24/00 pe

Begin experiment TcA - Technicium 99/clinoptilolite sorption.

Tc Sorption on Clinoptilolite
Sorption Experiment – TcA

Written by: J.D. Prikryl
Revision No.: 0

Date Written: Jan 28, 2000
Date Revised: N/A

CONDITIONS:

1. Σ Tc = 30 ppb
2. 0.01 m NaCl matrix, equilibrium with atmospheric $CO_2(g)$; $PCO_2 = 10^{-3.5}$
3. pH range 2.5 to 9
4. Initial solution volume = 20 ml, initial clinoptilolite mass = 0.5 g

OBJECTIVES:

1. To investigate the characteristics of Tc sorption on clinoptilolite as a function of solution pH.
2. To investigate the effects of Eh on Tc sorption

EQUIPMENT:

Gyratory shaker
Packard 2505 TR/AB liquid scintillation counter
Orion pH/mV/ISE/°C meter
Combination pH electrode
Combination platinum redox/ORP and silver:silver chloride electrodes
ATC probe
Calibrated thermometer
Eppendorf micropipettors
Oxford macropipettors
ATC probe
Repipettor for transfer of scintillation cocktail
Analytical balances (Mettler 4600 and 240AE)

SUPPLIES:

50 ml polycarbonate centrifuge tubes (acid washed and dried)
FEP beaker (acid washed and dried)
1 or 2L FEP bottle
Eppendorf and Oxford pipette tips
pH buffer solutions
Ultima-Gold AB liquid scintillation cocktail
7-ml scintillation vials
weighing paper and boats (as necessary)
Na clinoptilolite
Reagent grade NaOH and HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water
stir bars
Potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$ (lot no. _____)
potassium ferricyanide, $K_3Fe(CN)_6$ (lot no. _____)
potassium chloride, KCl (lot no. _____)
quinhydrone (lot no. _____)
glassware and plasticware as needed

TcA Experiment

2/24/00
ac

PROCEDURE:

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. Use degassed ultrapure water to make the NaOH solutions. The NaOH solutions should be stored in tightly capped glass reagent bottles. Record lot numbers and preparation steps in scientific notebook. The following stock solutions are required:

1. 1.0 M HCl
2. 0.1 M HCl
3. 0.01 M HCl
4. 1.0 M NaOH
5. 0.1 M NaOH
6. 0.01 M NaOH

B. Experimental Solution Preparation

1. Label fourteen (14) 50 ml polycarbonate centrifuge tubes (e.g., TcA-pHi – where i is the approximate pH of each solution, see Table 1). Label one (1) 50 ml polycarbonate tube as TcA-ITc. Measure and record the weight of each tube with cap (use Mettler AE240 balance).

2. Add clinoptilolite to each experimental solution container
   a. Transfer ~0.5 ± 0.001 g of Na clinoptilolite to each test tube.
   b. Measure and record the weight of test tube following addition of clinoptilolite.

3. Prepare 30 ppb Tc solution (total of 300 g)
   a. Tare a 2L FEP bottle on the Mettler 4600 balance.
   b. Add 30 g of the 300 ppb Tc stock solution (spike 43A) to the tared FEP bottle.
   c. Add ultrapure water to the FEP bottle until the balance reads 300 g.

4. Add Tc solution to each experimental container
   a. Add ~20 g of the 30 ppb Tc solution to each TcA-pHi container (use the Mettler 4600 balance to tare the centrifuge tube, then add ~20 g of the Tc solution using an FEP beaker, minor adjustments can be made using a plastic pipette). Remaining solution should be placed in the TcA-ITc tube.
   b. Following addition of the Tc solution, measure and record the weight of each tube.

5. Adjust pH of experimental solutions
   a. Use an Eppendorf micropipettor and associated tip to add HCl or NaOH to each experimental container. The concentration and approximate volume of acid or base to be added to each container is given in Table 1.
   b. Measure and record weight of each container following pH adjustment.

B. Start Sorption Experiment

1. Ensure that caps are loosely fitted on tubes to allow for gas exchange with atmosphere.

2. Place tubes on gyratory shaker; set shaker speed at ~120 rpm.

3. Circulate air into the headspace of each tube for a few hours each day using plastic tubing. This will speed up equilibrium with atmosphere.

4. Allow the experimental solutions and clinoptilolite to equilibrate for 14 days.

TcA Experiment

2/24/00
ac

5. Determine initial Tc concentration of experimental solutions by sampling solution in tube TcA-ITc.

   a. Label (e.g., TcA-Ia and TcA-Ib) and pre-weigh 2, 7 ml scintillation vials.
   b. Using an Eppendorf pipette, withdraw 0.5 ml of solution from container TcA-ITc and transfer to a liquid scintillation vial. Repeat for the second vial. Measure and record weight of both vials after the transfer.
   c. Add 5 ml of Ultima-Gold AB cocktail to each vial. Homogenize samples and set aside for analysis.
   d. Analyze initial Tc samples by LSA.

C. Measure Equilibrium pH and Tc Concentration of Experimental Solutions

1. Following the 14 day equilibrium period, measure and record weight of each solution tube. Be careful to cap solutions tightly before handling.

2. Measure and record the pH of each solution. Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.

3. *If pH measurements indicate the expected values and range given in Table 1, proceed with sampling for Tc concentrations; otherwise, contact principal investigator for instructions.*

4. Sample solutions for Tc concentration
   a. For each sample tube label 2, 7 ml LSA vials (e.g., TcA-3a and TcA-3b). Measure and record weight of each vial.
   b. From each sample tube, withdraw 2, 0.5 ml aliquots of solution and transfer to the appropriately labeled LSA vials. Measure and record weight of each vial after addition of experimental solution.
   c. Measure and record the weight of each experimental tube following pH measurement and LSA sample withdrawal. Place tubes back on gyratory shaker set at ~120 rpm and insure caps are loosely fitted.
   d. Add 5 ml of Ultima-Gold AB cocktail to each vial, homogenize vial contents, and set aside for counting.
   e. Measure Tc in sample vials by LSA.

D. Determine Eh of Experimental Solutions

Measure ORP (oxidation-reduction potential) of experimental solutions by following the method outlined below.

1. Prepare redox reference quinhydrone solutions
   a. Mix 50 ml of pH 4 buffer solution with 0.5 g of quinhydrone in a plastic cup.
   b. Mix 50 ml of pH 7 buffer solution with 0.5 g of quinhydrone in a plastic cup.

   *Be sure that excess quinhydrone is used in each solution so that crystals are always present. These solutions are stable for only 8 hrs. Table 3 in ASTM Method D1498-93 lists the nominal millivolt readings for the reference solutions.

2. Prepare ZoBell's solution
   a. In a 1L volumetric flask add 1.4080 g potassium ferrocyanide, 1.0975 g potassium ferricyanide, and 7.4557 g potassium chloride.
   b. Dissolve in ultrapure water and dilute solution to 1L.

2/24/00
pc

TcA Experiment

c. Store the solution in a dark bottle, label, and refrigerate. The solution is stable for at least 90 days if kept chilled at 4 °C.
d. Table 6.5-3 of the U.S. Geological Survey TWRI Book 9 lists the Eh of ZoBell's solution as a function of temperature.

3. Check response of ORP electrode to the standard redox solutions. Readings should be within 30 mV of the value expected for the standard solution.

If response of ORP electrode is acceptable then proceed to the next step, otherwise contact principal investigator for instructions.

4. Measure ORP of experimental solutions
a. Place a stir bar in each experimental tube to insure agitation of solution during measurement.
b. Measure the ORP of the sample solution by immersing the electrode directly into the sample tube and record the millivolt potential. Also record temperature of experimental solutions using a calibrated thermometer.
c. The Eh of each solution can be calculated using the following formula

Eh = Eobs + Eref

Where Eh is oxidation-reduction potential referred to the hydrogen scale, mV; Eobs is the observed oxidation-reduction potential of the platinum electrode, mV; and Eref is the oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

Table 1. Container labels, estimated solution pH, and volume of HCl or NaOH solutions needed for adjustment of pH in 0.01 NaCl solutions with 30 ppb Tc. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated Solution pH	Volume of HCl Needed, ml	Molarity
TcA-pH2.5	2.5	0.075	1.0
TcA-pH3	3	0.025	1.0
TcA-pH3.5	3.5	0.075	0.1
TcA-pH4	4	0.025	0.1
TcA-pH4.5	4.5	0.080	0.01
TcA-pH5	5	0.035	0.01
TcA-pH5.5	5.5	0.020	0.01
TcA-pH6	6	-	-
Sample Label	**Estimated Solution pH**	**Volume of NaOH Needed, ml**	**Molarity**
TcA-pH6.5	6.5	0.035	0.01
TcA-pH7	7	0.020	0.1
TcA-pH7.5	7.5	0.060	0.1
TcA-pH8	8	0.020	1.0
TcA-pH8.5	8.5	0.065	1.0
TcA-pH9	9	0.225	1.0

2/25/00
pc

TcA Experiment

Preparation of HCl and NaOH solutions used for adjusting pH of sample solutions:

A. Preparation of HCl acid solns:

1. Boiled 800 mL of nanopure H_2O in a 1 liter erlenmeyer flask for about 15 minutes.
2. Let degassed H_2O cool and then transfer liquid to a 1L pp bottle.
3. Prepared 1.0 M HCl soln. by adding 2/25/00 8.33 mL of conc. HCl, using an automatic micro pipettor, to a 100 mL volumetric flask and adding to mark with nanopure H_2O. ~~Degassed~~ as 2/25/00 Trace Metal Grade HCl - Fisher Chemical Lot #. 418110

$$V_1 = \frac{C_2 V_2}{C_1} = \frac{1\,M\,HCl \times 100\,mL}{12\,M\,HCl} = 8.33\,mL$$

4. Prepared 100 mL of 0.1 M HCl soln. by adding 10 mL of prepared 1.0 M HCl, ~~24~~ 2/25/00 using a volumetric glass pipet, to a 100 mL vol. flask and adding to mark with nanopure H_2O.

5. Prepared 100 mL of 0.01 M HCl by adding 10 mL of 0.1 M HCl to a 100 mL vol. flask and adding to mark with nanopure H_2O.

6. Transferred prepared HCl solns. to 125 mL pp bottles and labeled as to concentration.

B. Preparation of NaOH solutions:

1. added one vial of Dilut-It analytical conc. (Lot # H33121) (J.T. Baker, Inc.), to a 100 mL volumetric flask and added to mark with degassed nanopure H_2O. Concentration = 1 M NaOH.

note - 1 vial makes 1 liter of 0.1 N NaOH.

TcA Experiment

Procedure - cont'd.

B. 2. Prepared 0.1M NaOH and 0.01M NaOH solutions by serial dilutions using 10mL pipette to a 100mL vol. flask.

3. Transferred NaOH solns. to 125mL PP bottles and labeled with concentrations.

2/24/00 pc — Clinoptilolite sample added to PC tubes: (Clino. 309/49)

Sample Name	2/24/00 pc Wt. of empty PC tubes, grams	2/24/00 pc Wt. of PC container + clino., grams
TcA-pH 2.5	22.1924	22.7076
TcA-pH 3.0	22.3596	22.8735
TcA-pH 3.5	22.2390	22.7497
TcA-pH 4.0	22.9726	22.4912
TcA-pH 4.5	22.3084	22.8224
TcA-pH 5.0	22.0100	22.5260
TcA-pH 5.5	22.4675	22.9833
TcA-pH 6.0	22.2754	22.7914
TcA-pH 6.5	22.3808	22.8951
TcA-pH 7.0	22.1024	22.6179
TcA-pH 7.5	22.2075	22.7211
TcA-pH 8.0	22.9926	22.5049
TcA-pH 8.5	22.6144	23.1325
TcA-pH 9.0	22.4798	22.9947

9/11/00 PB 2/24/00 pc 2/1 pc

9/11/00 PB 2/24/00 pc 2/1 pc

line outs initialed for pc by PB on 9/11/00

TcA Experiment

Procedure - cont'd.

2/25/00 pc — Preparation of 30ppb Tc^{99} in 0.01M NaCl matrix:

(031/289)

Wt. of 300ppb Tc^{99} (43A spike) = 29.98 g

Wt. of nanopure H_2O + spike = 300.01 g

Liquids weighed directly into a tared 1-L THE pc 2/25/00 bottle and labeled as "30ppb Tc in .01M NaCl matrix."

2/26/00 pc — Preparation of TcA control samples:

Sample I.D.	Wt. of empty vial	Wt. of vial + sample	= Wt. of sample
TcA-I a	7.1671 g	7.6684 g	0.5013 g
TcA-I b	7.1590 g	7.6579 g	0.4989 g

2/26/00 — Addition of Tc^{99} solution and pH adjuster solutions:

pc 2/26/00

Sample Name	Wt. of PC tube after add. of Tc soln., grams	Wt. of PC tube after pH adj., grams
TcA-pH 2.5	42.7986	42.8706
TcA-pH 3.0	42.9063	42.9252
TcA-pH 3.5	42.7635	42.8340
TcA-pH 4.0	43.2124	~~43.2374~~ 43.2348 PB 2/26/00
TcA-pH 4.5	42.8939	42.7652
TcA-pH 5.0	42.5197	42.5496
TcA-pH 5.5	42.9739	42.9908
TcA-pH 6.0	42.7944	42.7943
TcA-pH 6.5	42.9241	42.9520
TcA-pH 7.0	42.6208	42.6354
TcA-pH 7.5	42.7917	42.8445
TcA-pH 8.0	42.5298	42.5488
TcA-pH 8.5	43.1240	43.1864
TcA-pH 9.0	43.1130	43.3460

2/26/00 JL

TcA Experiment

Procedure - cont'd.

pH adjustment of sample solns.

pL 2/26/00

Sample Name	Estimated Solution pH	Volume of HCl Needed, mL	Molarity	Volume of Solution Added, ml
TcA-pH 2.5	2.5	0.075	1.0	0.075
TcA-pH 3.0	3.0	0.025	1.0	0.025
TcA-pH 3.5	3.5	0.075	0.1	0.075
TcA-pH 4.0	4.0	0.025	0.1	0.025
TcA-pH 4.5	4.5	0.080	0.01	0.080
TcA-pH 5.0	5.0	0.035	0.01	0.035
TcA-pH 5.5	5.5	0.020	0.01	0.020
TcA-pH 6.0	6.0	0	—	0

Sample Name	Estimated Solution pH	Volume of NaOH Needed, mL	Molarity	Volume of Solution Added, ml
TcA-pH 6.5	6.5	0.035	0.01	0.035
TcA-pH 7.0	7.0	0.020	0.1	0.020
TcA-pH 7.5	7.5	0.060	0.1	0.060
TcA-pH 8.0	8.0	0.020	1.0	0.020
TcA-pH 8.5	8.5	0.065	1.0	0.065
TcA-pH 9.0	9.0	0.225	1.0	0.200 0.025

Placed sample tubes in a holding rack and placed rack on shaker. Set to 180rpm.

attached 0.05 cm I.D. of tubing to containers through a small hole in container cap for forced aeration.

3/13/00 JL

TcA Experiment

Procedure - cont'd.

Sample Name	3/13/00 JL Wt. of container after equil. period, grams	3/13/00 JL Measured pH value of Sample solution	3/14/00 JL Wt. of container after pH meas. & sampling, grams
TcA-pH 2.5	41.9989	3.56	40.9304
TcA-pH 3.0	42.1046	4.39	41.0318
TcA-pH 3.5	41.7678	5.04	40.6891
TcA-pH 4.0	42.5280	5.24	41.4822
TcA-pH 4.5	42.0337	5.42	40.9895
TcA-pH 5.0	41.8114	5.40	40.7573
TcA-pH 5.5	42.3998	5.45	41.3504
TcA-pH 6.0	41.9803	5.43	40.9399
TcA-pH 6.5	42.0268	5.50	40.9616
TcA-pH 7.0	41.7250	5.67	40.6730
TcA-pH 7.5	41.9190	6.20	40.8466
TcA-pH 8.0	41.7689	7.25	40.7316
TcA-pH 8.5	42.4887	8.23	41.4531
TcA-pH 9.0	42.7017	9.05	41.6654

Performed a 4 point calibration of the Orion 920A pH meter using pH buffers 2.00, 4.00, 7.00, and 9.00. Slope of instrument = 99.6

Buffers used for calibration:

Fisher Chemical Buffer Solns. 2.00 lot no. 996387-24
4.00 lot no. 994523-24
7.00 lot no. 000749-24
9.00 lot no. 994373-24

TcA Experiment

3/14/00
RE

Sampling data:

Sample Name	3/14/00 RE Wt. of empty sample vial, grams	3/14/00 RE Wt. of vial + sample solution, grams
TcA-pH 2.5a	7.2520	7.7564
TcA-pH 2.5b	7.3104	7.8144
TcA-pH 3.0a	7.2824	7.7863
TcA-pH 3.0b	7.3393	7.8419
TcA-pH 3.5a	7.2383	7.7392
TcA-pH 3.5b	7.2980	7.7997
TcA-pH 4.0a	7.3149	7.8150
TcA-pH 4.0b	7.3995	7.9018
TcA-pH 4.5a	7.3264	7.8261
TcA-pH 4.5b	7.2442	7.7467
TcA-pH 5.0a	7.3320	7.8310
TcA-pH 5.0b	7.4040	7.9057
TcA-pH 5.5a	7.3500	7.8481
TcA-pH 5.5b	7.2679	7.7695
TcA-pH 6.0a	7.3212	7.8194
TcA-pH 6.0b	7.3860	7.8865
TcA-pH 6.5a	7.3043	7.8027
TcA-pH 6.5b	7.3436	7.8455
TcA-pH 7.0a	7.2959	7.7932
TcA-pH 7.0b	7.2856	7.7850
TcA-pH 7.5a	7.3397	7.8369
TcA-pH 7.5b	7.2665	7.7686
TcA-pH 8.0a	7.2623	7.7592
TcA-pH 8.0b	7.2955	7.7951
TcA-pH 8.5a	7.3283	7.8238
TcA-pH 8.5b	7.2937	7.7926
TcA-pH 9.0a	7.2977	7.7930
TcA-pH 9.0b	7.3294	7.8259

TcA Experiment

3/15/00
RE

Preparation of quinhydrone soln. and Zobell's soln. for referencing Corning redox combination electrode (platinum): Cat. No. 476516.

Zobell's soln: Recipe taken from U.S. Geological Survey TWRI, Book 9 pg. 5

Potassium ferrocyanide ($K_4Fe(CN)_6 \cdot 3H_2O$)
- Fisher Chemical
- Lot No. 995787
- Received 3/7/00, no exp. date

Potassium ferricyanide ($K_3Fe(CN)_6$)
- Fisher Chemical
- Lot No. 996057
- Received 3/7/00, no exp. date

Potassium Chloride (KCl)
- Fisher Chemical
- Lot No. 885961
- No rec. date, no exp. date

Wt. of $K_4Fe(CN)_6 \cdot 3H_2O$ = 1.4077 g
Wt. of $K_3Fe(CN)_6$ = 1.0925 g
Wt. of KCl = 7.4593 g

Added chemicals to a 1L 3/15/00 volumetric flask and filled to mark with nanopure H_2O, poured contents into colored glass storage bottle and labeled as Zobell's Soln expiration date of 6/15/00.

Quinhydrone soln.:

Wt. of quinhydrone in 50 mL pH 4 buffer = 0.5043 g
Wt. of quinhydrone in 50 mL pH 7 buffer = 0.5056 g

Quinhydrone Lot No. B0010437-2
Mfg. ACROS Organics

3/15/00
pc

TcA Experiment

	mV	C°	*Expected ORP, mV
Zobell's Soln.	230.2	23.3	246 ± 30
quinhydrone pH 4	263.8	22.1	263 ± 30
quinhydrone pH 7	89.4	22.2	86 ± 30

* quinhydrone soln. response ref. ASTM D 1498 table 3
Zobell's soln. response ref. U.S. Geological Survey TWRI Book 9 Table 6.5-3.

Eh Measurement of TcA Sample Solutions (mV)

Sample ID.	Trial 1	Trial 2	Trial 3
TcA-pH 2.5	287.0 ↑	306.8	228.4
TcA-pH 3.0	265.6	267.7	299.4
TcA-pH 3.5	280.8	232.7	266.8 ↓
TcA-pH 4.0	271.3	239.4	251.3
TcA-pH 4.5	260.5 ↓	235.5	241.4
TcA-pH 5.0	267.6	255.1	249.9
TcA-pH 5.5	253.2	229.0 ↓	253.7
TcA-pH 6.0	239.3	241.9	discontinued pc 3/15/00
TcA-pH 6.5	254.0	229.2	
TcA-pH 7.0	221.5	231.0	
TcA-pH 7.5	223.8	204.5	
TcA-pH 8.0	208.3	208.3	
TcA-pH 8.5	183.7	176.6	
TcA-pH 9.0	168.5 ↑	160.4 ↑	
Zobell's soln check	231.2	231.6	
TcA-I	202.4		

mV readings taken at solution temperature of 23.9°C.

a slow drift of mV readings are observed with each sample even after 5-10 minutes of testing.

3/16/00
pc

TcA Experiment

Changing method for obtaining Eh measurements due to inconsistent readings from previous 3 trials of analysis.

Decanted sample soln. from PC tubes into 15 mL PC storage containers, added magnetic stir bar, inserted Eh probe and set container on a stir plate.

Allowed each sample to analyze for approximately 15 minutes to assure a stable mV reading.

Sample ID.	Trial 1 @ 20.8°C	Trial 2 @ 21.0°C	Trial 3
Zobell's ref. soln.	249.0 @ 20.8°C	~~251.8 mV~~ 3/16/00	234.1 @ 20.3°C
TcA-pH 2.5	336.2 @ 20.8°C	351.8 @ 21.0°C	350.0 @ 21.1°C
TcA-pH 3.0	282.9 @ 20.8°C	307.3 @ 21.4°C	306.8 @ 21.2°C
TcA-pH 3.5	250.2 @ 20.9°C	266.6 @ 21.9°C	pc 263.6 @ ~~23.5~~ 21.5°C
TcA-pH 4.0	253.6 @ 20.9°C	261.5 @ 22.5°C 3/16/00	250.5 @ 21.8°C
TcA-pH 4.5	258.8 @ 21.0°C	254.2 @ 20.5°C	246.5 @ 22.3°C
TcA-pH 5.0	267.7 @ 21.0°C	261.0 @ 20.6°C	253.6 @ 22.6°C
TcA-pH 5.5	272.6 @ 20.5°C	271.8 @ 20.4°C	257.9 @ 22.7°C
TcA-pH 6.0	267.4 @ 20.5°C	266.1 @ 20.4°C	245.2 @ 22.7°C
TcA-pH 6.5	280.7 @ 20.5°C	273.1 @ 20.4°C	259.3 @ 22.7°C
TcA-pH 7.0	270.1 @ 20.5°C	262.4 @ 20.4°C	254.5 @ 22.7°C
TcA-pH 7.5	264.4 @ 20.5°C	250.9 @ 20.4°C	248.4 @ 22.7°C
TcA-pH 8.0	243.9 @ 20.4°C	229.0 @ 20.4°C	227.8 @ 22.8°C
TcA-pH 8.5	~~230.4 @ 20.4°C~~ 226.4 @ 20.4°C pc	196.1 @ 20.4°C	191.0 @ 23.1°C
TcA-pH 9.0	3/17/00 179.5 @ 20.4°C	3/17/00 169.2 @ 20.4°C	165.6 @ 23.6°C

pc 3/17/00

3/17/00
pc

3/17/00

TcA Experiment

3/27/00 pc

Data summary:

R Cherrington pc 3/27/00

Eh of Sample Solns.
as a function of temperature

March 27, 2000

Sample I.D.	Measured pH	Trial 1 Potential (mV)	Soln. Temp. (deg. C)	Eh (mV)	Trial 2 Potential (mV)	Soln.Temp. (deg. C)	Eh (mV)	Trial 3 Potential (mV)	Soln.Temp. (deg. C)	Eh (mV)	Average Eh (mV)
pH-2.5	3.56	336.2	20.8	539.2	351.8	21.0	554.8	350.0	21.1	553.0	549.0
pH-3	4.39	282.9	20.8	485.9	307.3	21.4	510.3	306.8	21.2	509.8	502.0
pH-3.5	5.04	250.2	20.9	453.2	266.6	21.9	468.6	263.6	21.5	465.6	462.5
pH-4	5.24	253.6	20.9	456.6	261.5	22.5	462.5	250.5	21.8	452.5	457.2
pH-4.5	5.42	258.8	21.0	461.8	254.2	20.5	457.2	246.5	22.3	448.5	455.8
pH-5	5.40	267.7	21.0	470.7	261.0	20.6	464.0	253.6	22.6	454.6	463.1
pH-5.5	5.45	272.6	20.5	475.6	271.8	20.4	475.8	267.9	22.7	468.9	470.1
pH-6	5.43	267.4	20.5	470.4	266.1	20.4	470.1	245.2	22.7	446.2	462.2
pH-6.5	5.50	280.7	20.5	483.7	273.1	20.4	477.1	259.3	22.7	460.3	473.7
pH-7	5.67	270.1	20.5	473.1	262.4	20.4	466.4	254.5	22.7	455.5	465.0
pH-7.5	6.20	264.4	20.5	467.4	250.9	20.4	454.9	248.4	22.7	449.4	457.2
pH-8	7.25	243.9	20.4	447.9	229.0	20.4	433.0	227.8	22.8	428.8	436.6
pH-8.5	8.23	221.4	20.4	425.4	196.1	20.4	400.1	191.0	23.1	392.0	405.8
pH-9	9.05	179.5	20.4	383.5	169.2	20.4	373.2	165.6	23.6	365.6	374.1

pc 3/27/00

pc 3/27/00

```
Assay Definition-

Assay Description:
Protocol Name:  TC-99 CPM
Add'l Heading:  BETA CPM TC-99
Original Protocol Settings (actual values may have changed):
   Delay Before Burst:  75
   Coincidence Time:  18
   Count Mode:  Normal
   Half Life A:  0.00 Hours
   Half Life B:  0.00 Hours
Assay imported during upgrade 1/25/00.

Assay Type: CPM
Report Name: 99Tc 2% 2sigma
Output Data Path: C:\Packard\Tricarb\Results\Cherrington\99Tc 2% 2sigma
Raw Results Path: C:\Packard\Tricarb\Results\Cherrington\99Tc 2% 2sigma
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Cherrington\99Tc 2% 2sigma\99Tc 2%
2sigma.txt

Count Conditions-

Nuclide: 99Tc
   Quench Indicator: SIS
   External Std Terminator (sec): n/a
   Pre-Count Delay (min): 0.00
Quench Set: n/a
Count Time (min): 180.00
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

Regions        LL        UL  Bkg Subtract  2Sigma % Terminator
A             0.0     300.0       1st Vial                2.00
B             0.4     300.0       1st Vial                0.00
C             0.0    2000.0       1st Vial                0.00

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
A
B
C
```

Cycle 1 Results

	S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	MESSAGES
BLANK	1	180.00	21	3.28	21	3.28	28	2.84	132.00	B
TcA-Ia	2	18.43	522	2.08	522	2.08	523	2.10	167.10	
Ib	3	18.93	508	2.09	508	2.09	508	2.10	165.87	
pH 2.5	4	17.82	541	2.08	541	2.08	540	2.09	163.12	
	5	18.00	535	2.08	535	2.08	535	2.10	163.73	
pH 3.0	6	18.13	531	2.08	531	2.08	531	2.10	164.77	
	7	17.94	537	2.08	537	2.08	537	2.10	163.31	
pH 3.5	8	17.75	543	2.08	543	2.08	545	2.09	163.23	
	9	17.83	540	2.08	540	2.08	539	2.10	164.59	
pH 4.0	10	18.05	533	2.08	533	2.08	534	2.09	165.60	
	11	18.20	529	2.08	529	2.08	529	2.10	166.67	
pH 4.5	12	18.14	531	2.08	531	2.08	530	2.10	165.18	
	13	18.10	532	2.08	532	2.08	532	2.10	162.48	
pH 5.0	14	18.29	526	2.08	526	2.08	526	2.10	164.07	
	15	18.34	525	2.08	525	2.08	525	2.10	165.55	
	16	18.22	528	2.08	528	2.08	528	2.10	163.05	
pH 5.5	17	18.49	520	2.08	520	2.08	520	2.10	162.29	
pH 6.0	18	18.11	532	2.08	532	2.08	531	2.10	165.05	
	19	18.06	533	2.08	533	2.08	534	2.09	163.94	
pH 6.5	20	17.90	538	2.08	538	2.08	538	2.09	165.38	
	21	17.82	541	2.08	541	2.08	541	2.09	163.61	
pH 7.0	22	17.98	536	2.08	535	2.08	536	2.09	163.96	
	23	18.17	530	2.08	530	2.08	530	2.09	164.84	
pH 7.5	24	17.99	535	2.08	535	2.08	535	2.10	164.89	
	25	17.66	546	2.08	546	2.08	544	2.10	166.54	
pH 8	26	18.19	529	2.08	529	2.08	528	2.10	165.35	
	27	18.11	531	2.08	532	2.08	532	2.10	165.53	
pH 8.5	28	18.53	519	2.08	519	2.08	519	2.10	164.06	
	29	17.89	538	2.08	538	2.08	538	2.10	165.88	
pH 9.0	30	18.46	521	2.08	521	2.08	521	2.10	164.85	
	31	18.38	524	2.08	523	2.08	524	2.10	165.76	

TcR Experiment

Test Method for Tc solution reduction
Experiment TcR

Written by: J.D. Prikryl
Revision No.: 0

Date Written: March 30, 2000
Date Revised: N/A

CONDITIONS:

1. Σ Tc = 30 ppb
2. 0.01 m NaCl matrix
3. pH range 2.5 to 9
4. Initial solution volume = 20 ml

OBJECTIVES:

To investigate the effects of reduction on the pH and Eh of Tc solutions.

METHOD:

Bubble N_2 through sample solutions to remove oxygen.

EQUIPMENT:

Gyratory shaker
Orion pH/mV/ISE/°C meter
Combination pH electrode
Combination platinum redox/ORP and silver:silver chloride electrodes
ATC probe
Calibrated thermometer
Eppendorf micropipettors
Oxford macropipettors
Analytical balances (Mettler 4600 and 240AE)
Nitrogen gas (99.999%)

SUPPLIES:

50 ml polycarbonate centrifuge tubes (acid washed and dried)
Eppendorf and Oxford pipette tips
pH buffer solutions
Reagent grade NaOH and HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water
stir bars
Potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$ (lot no. _____)
potassium ferricyanide, $K_3Fe(CN)_6$ (lot no. _____)
potassium chloride, KCl (lot no. _____)
quinhydrone (lot no. _____)
glassware and plasticware as needed

TcR Experiment

PROCEDURE:

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. Use degassed ultrapure water to make the NaOH solutions. The NaOH solutions should be stored in tightly capped glass reagent bottles. Record lot numbers and preparation steps in scientific notebook. The following stock solutions are required:

1. 1.0 M HCl
2. 0.1 M HCl
3. 0.01 M HCl
4. 1.0 M NaOH
5. 0.1 M NaOH
6. 0.01 M NaOH

Note These solutions should be available from earlier sorption experiments.*

B. Experimental Solution Preparation

1. Label five (5) 50 ml polycarbonate centrifuge tubes (e.g., TcR-pHi – where i is the approximate pH of each solution, see Table 1).

2. Prepare 30 ppb Tc solution (total of 100 g) 10.01g (300ppb Tc) 100.02 g ttl
   a. Tare a FEP bottle on the Mettler 4600 balance.
   b. Add 10 g of the 300 ppb Tc stock solution (spike 43A) to the tared FEP bottle.
   c. Add ultrapure water to the FEP bottle until the balance reads 100 g.

   4/11/00

3. Add Tc solution to each experimental container
   a. Add ~20 g of the 30 ppb Tc solution to each TcR-pHi container.

4. Adjust pH of experimental solutions
   a. Use an Eppendorf micropipettor and associated tip to add HCl or NaOH to each experimental container. The concentration and approximate volume of acid or base to be added to each container is given in Table 1.

5. Bubble N_2 through the sample solutions to remove O_2.

C. Measure solution pH

1. Measure and record the pH each solution periodically (every 2 or 3 days). Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.

2. When pH appears to be stable proceed to next step (Eh measurement).

TcR Experiment

4/11/00
pc

pc
4/11/00

D. Measure solution Eh

Measure ORP (oxidation-reduction potential) of experimental solutions by following the method outlined below.

1. Prepare redox reference quinhydrone solutions
   a. Mix 50 ml of pH 4 buffer solution with 0.5 g of quinhydrone in a plastic cup.
   b. Mix 50 ml of pH 7 buffer solution with 0.5 g of quinhydrone in a plastic cup.

   *Be sure that excess quinhydrone is used in each solution so that crystals are always present. These solutions are stable for only 8 hrs. Table 3 in ASTM Method D1498-93 lists the nominal millivolt readings for the reference solutions.

2. Prepare ZoBell's solution
   a. In a 1L volumetric flask add 1.4080 g potassium ferrocyanide, 1.0975 g potassium ferricyanide, and 7.4557 g potassium chloride.
   b. Dissolve in ultrapure water and dilute solution to 1L.
   c. Store the solution in a dark bottle, label, and refrigerate. The solution is stable for at least 90 days if kept chilled at 4 °C.
   d. Table 6.5-3 of the U.S. Geological Survey TWRI Book 9 lists the Eh of ZoBell's solution as a function of temperature.

3. Check response of ORP electrode to the standard redox solutions. Readings should be within 30 mV of the value expected for the standard solution.

   If response of ORP electrode is acceptable then proceed to the next step, otherwise contact principal investigator for instructions.

4. Measure ORP of experimental solutions
   a. Place a stir bar in each experimental tube to insure agitation of solution during measurement.
   b. Measure the ORP of the sample solution by immersing the electrode directly into the sample tube and record the millivolt potential. Also record temperature of experimental solutions using a calibrated thermometer.
   c. The Eh of each solution can be calculated using the following formula

      Eh = Eobs + Eref

      Where Eh is oxidation-reduction potential referred to the hydrogen scale, mV; Eobs is the observed oxidation-reduction potential of the platinum electrode, mV; and Eref is the oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

* Table 1. Container labels, estimated solution pH, and volume of HCl or NaOH solutions needed for adjustment of pH in 0.01 NaCl solutions with 30 ppb Tc in a reducing environment. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated Solution pH	Volume of HCl Needed, ml	Molarity
TcA-pH2.5	2.5	0.100	1.0
TcA-pH4	4	0.025	0.1
TcA-pH5.5	5.5	0.020	0.01
TcA-pH7	7	0.010	0.01
TcA-pH8.5	8.5	–	–

* 0.01N NaCl soln. not used. Substituted by nanopure H_2O.
pc

TcR Experiment

4/12/00
pc

pH measurement of TcR test solutions:

Soln. label	pH
TcR 2.5	2.38
TcR 4.0	4.05
TcR 5.5	6.12
TcR 7.0	6.70
TcR 8.5	6.95
TcR 8.5	8.76 added 20 uL of 0.01N NaOH

4/13/00
pc

pH measurement of TcR test solns:

Soln. label	pH
TcR 2.5	2.34
TcR 4.0	4.04
TcR 5.5	6.10
TcR 7.0	7.00
TcR 8.5	8.71

4/17/00
pc

pH measurement of TcR test soln + Eh measurement:

Soln. label	*pH	mV	interpolated Eh @ 21.6
TcR 2.5	4/17/00 pc ~~2.34~~	2.21	
TcR 4.0	4/17/00 pc ~~4.04~~	4.00	Eh not performed due to unstable pH conditions of samples.
TcR 5.5	4/17/00 pc ~~6.10~~	6.03	
TcR 7.0	6.27		
TcR 8.5	7.35		

* note: test samples had lost some liquid volume due to evaporation caused by the bubbling of N gas in the loosely capped containers.

pH meter calibrated using a 3 pt. buffer soln reading.
slope = 99.7 Buffer Lot No.'s as per 309/59.

TcR Experiment

4/17/00 RL — Zobell's standard soln. @ 21.5°C had a measurement of 234.1 mV. Eh value = 435
Expected Eh value of Zobell's = 441

Zobell soln. - prepared 309/61 3/15/00

4/19/00 — Eh measurement of TcR solutions while N is continuing to bubble thru the soln and thus preventing the introduction of outside oxygen into the sample vessel.

Sample Label	mV	Eh	pH
TcR 2.5	410	611	2.15
TcR 4.0	332	N/A	4.06
TcR 5.5	266	N/A	8.01
TcR 7.0	~~316~~ RL 4/19/00 ~~216~~	316	N/A 7.90
TcR 8.5	4/19/00 168	369	8.69
Zobell soln.	241	442	NA

RL 4/19/00

TcS Experiment

Test Method for Tc solution reduction
Experiment TcS

Written by: J.D. Prikryl — Date Written: April 20, 2000
Revision No.: 0 — Date Revised: N/A

CONDITIONS:

1. Σ Tc = 30 ppb
2. 0.01 m NaCl matrix
3. pH range 2.5 to 8.5
4. Initial solution volume = 20 ml

OBJECTIVES:

To investigate the effects of reduction on the pH and Eh of Tc solutions.

METHOD:

Add sodium sulfate and sodium sulfite to Tc solutions.

EQUIPMENT:

Gyratory shaker
Orion pH/mV/ISE/°C meter
Combination pH electrode
Combination platinum redox/ORP and silver:silver chloride electrodes
ATC probe
Calibrated thermometer
Eppendorf micropipettors
Oxford macropipettors
Analytical balances (Mettler 4600 and 240AE)
Nitrogen gas (99.999%)

SUPPLIES:

50 ml polycarbonate centrifuge tubes (acid washed and dried)
Eppendorf and Oxford pipette tips
pH buffer solutions
Reagent grade NaOH and HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water
stir bars
Potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$ (lot no. _____)
potassium ferricyanide, $K_3Fe(CN)_6$ (lot no. _____)
potassium chloride, KCl (lot no. _____)
quinhydrone (lot no. _____)
sodium sulfate, Na_2SO_4 (lot no. _____)
sodium sulfite, Na_2SO_3 (lot no. _____)
glassware and plasticware as needed

PROCEDURE:

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. Use degassed ultrapure water to make the NaOH solutions. The NaOH solutions should be stored in tightly capped glass reagent bottles. Record lot numbers and preparation steps in scientific notebook. The following stock solutions are required:

1. 1.0 M HCl
2. 0.1 M HCl
3. 0.01 M HCl
4. 1.0 M NaOH
5. 0.1 M NaOH
6. 0.01 M NaOH

Note These solutions should be available from earlier sorption experiments.*

B. Experimental Solution Preparation

1. Label six (6) 50 ml polycarbonate centrifuge tubes (e.g., TcS-pHi – where i is the approximate pH of each solution, see Table 1).

2. Prepare 30 ppb Tc solution (total of 120 g)
   a. Tare a FEP bottle on the Mettler 4600 balance.
   b. Add 12 g of the 300 ppb Tc stock solution (spike 43A) to the tared FEP bottle.
   c. Add ultrapure water to the FEP bottle until the balance reads 120 g.

3. Add S-bearing reagents to Tc solution
   a. Add 0.151 g Na2SO3 and 0.085 g Na2SO4 to the FEP bottle. Mix thoroughly and make sure reagents are totally dissolved before proceeding to next step.

4. Add Tc solution to each experimental container
   a. Add ~20 g of the 30 ppb Tc solution to each TcS-pHi container

5. Adjust pH of experimental solutions
   a. Use an Eppendorf micropipettor and associated tip to add HCl or NaOH to each experimental container. The concentration and approximate volume of acid or base to be added to each container is given in Table 1.

6. Cap the tubes tightly and place on gyratory shaker.

C. Measure solution pH

1. Measure and record the pH each solution periodically (every day). Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.

2. When pH appears to be stable proceed to next step (Eh measurement).

D. Measure solution Eh

Measure ORP (oxidation-reduction potential) of experimental solutions by following the method outlined below.

1. Prepare redox reference quinhydrone solutions
   a. Mix 50 ml of pH 4 buffer solution with 0.5 g of quinhydrone in a plastic cup.
   b. Mix 50 ml of pH 7 buffer solution with 0.5 g of quinhydrone in a plastic cup.

   *Be sure that excess quinhydrone is used in each solution so that crystals are always present. These solutions are stable for only 8 hrs. Table 3 in ASTM Method D1498-93 lists the nominal millivolt readings for the reference solutions.

2. Prepare ZoBell's solution
   a. In a 1L volumetric flask add 1.4080 g potassium ferrocyanide, 1.0975 g potassium ferricyanide, and 7.4557 g potassium chloride.
   b. Dissolve in ultrapure water and dilute solution to 1L.
   c. Store the solution in a dark bottle, label, and refrigerate. The solution is stable for at least 90 days if kept chilled at 4 °C.
   d. Table 6.5-3 of the U.S. Geological Survey TWRI Book 9 lists the Eh of ZoBell's solution as a function of temperature.

3. Check response of ORP electrode to the standard redox solutions. Readings should be within 30 mV of the value expected for the standard solution.

   If response of ORP electrode is acceptable then proceed to the next step, otherwise contact principal investigator for instructions.

4. Measure ORP of experimental solutions
   a. Place a stir bar in each experimental tube to insure agitation of solution during measurement.
   b. Measure the ORP of the sample solution by immersing the electrode directly into the sample tube and record the millivolt potential. Also record temperature of experimental solutions using a calibrated thermometer.
   c. The Eh of each solution can be calculated using the following formula

      Eh = Eobs + Eref

      Where Eh is oxidation-reduction potential referred to the hydrogen scale, mV; Eobs is the observed oxidation-reduction potential of the platinum electrode, mV; and Eref is the oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

Table 1. Container labels, estimated solution pH, and volume of HCl or NaOH solutions needed for adjustment of pH in 0.01 NaCl solutions with 30 ppb Tc in a reducing environment. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated Solution pH	Volume of HCl Needed, ml	Molarity
TcS-pH2.5	2.5	0.55	1.0
TcS-pH4	4	0.21	1.0
TcS-pH5.5	5.5	0.19	1.0
TcS-pH7	7	0.095	1.0
TcS-pH8.5	8.5	0.05	0.1
TcS-pH9.5	9.5	--	--

Tc-S Experiment

13 Jun 00
PO

Copy of notebook made for QA archives.

6/13/00
pc

Preparation of 30ppb ^{99}Tc solution:

1. added to tared 200mL FEP PP bottle; 6/13/00
   Wt. of 300ppb ^{99}Tc soln. = 12.0064 g
   Wt. of nanopure H_2O + Tc soln. = 119.9415 g

2. Added S bearing reagents to 30ppb Tc soln:
   Wt. of Na_2SO_3 = 0.1515 g
   Wt. of Na_2SO_4 = 0.0851 g

3. Added above prepared soln. to labeled Pc tubes 20g using an eppendorf 10ml pipettor. Did not weigh containers or added reagents.

4. Added required quantities of HCl (309/55) to sample tubes, capped tightly, and placed on shaker.

6/13/00 mc-c

Reagents: Na_2SO_3
Fisher Scientific
Lot No. 905923

Na_2SO_4
Fisher Scientific
Lot No. 901213

HCl solutions prepared 309/55

Zobell's Soln. prepared 309/61

300 ppb ^{99}Tc solution
Spike 43-A 031/289

6/16/00
6/14/00 pc
pc

Tc-S Experiment

Calibrated Orion pH meter using buffers as per 309/30 but substituting pH buffer 4.00 (Fisher Chemical, Lot No. 994523-74) for the 3.00 buffer.

Slope = 99.2

Data:

Sample I.D	pH	mV	interpolated Eh @ 21.2°C
Tc-S pH 2.5	1.75	543	745
Tc-S pH 4.0	2.19	565	767
Tc-S pH 5.5	2.64	209	441
Tc-S pH 7.0	3.25	220	422
Tc-S pH 8.5	6.89	218	420
Tc-S pH 9.5	7.61	208	410

mc
6/30/00

6/21/00
JC

TcS-1 Experiment

Test Method for Tc solution reduction
Experiment TcS1

Written by: J.D. Prikryl
Revision No.: 0

Date Written: June 19, 2000
Date Revised: N/A

CONDITIONS:

1. Σ Tc = 30 ppb
2. 0.01 m NaCl matrix
3. pH range 2.5 to 4.0
4. Initial solution volume = 25 ml

OBJECTIVES:

To investigate the effects of reduction on the pH and Eh of Tc solutions.

METHOD:

Add sodium sulfite to Tc solutions.

EQUIPMENT:

Gyratory shaker
Orion pH/mV/ISE/°C meter
Combination pH electrode
Combination platinum redox/ORP and silver:silver chloride electrodes
ATC probe
Calibrated thermometer
Eppendorf micropipettors
Oxford macropipettors
Analytical balances (Mettler 4600 and 240AE)

SUPPLIES:

50 ml polycarbonate centrifuge tubes (acid washed and dried)
Eppendorf and Oxford pipette tips
pH buffer solutions
Reagent grade HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water
stir bars
Potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$ (lot no. _____)
potassium ferricyanide, $K_3Fe(CN)_6$ (lot no. _____)
potassium chloride, KCl (lot no. _____)
quinhydrone (lot no. _____)
sodium sulfite, Na_2SO_3 (lot no. _____)
glassware and plasticware as needed

6/21/00
JC

TcS-1 Experiment

PROCEDURE:

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. The following stock solutions are required:

1. 1.0 M HCl
2. 0.1 M HCl

Note These solutions should be available from earlier sorption experiments.*

B. Experimental Solution Preparation

1. Label six (4) 50 ml polycarbonate centrifuge tubes (e.g., TcS1-pHi – where i is the approximate pH of each solution, see Table 1).

2. Prepare 30 ppb Tc solution (total of 100 g)
   a. Tare a FEP bottle on the Mettler 4600 balance.
   b. Add 10 g of the 300 ppb Tc stock solution (spike 43A) to the tared FEP bottle.
   c. Add ultrapure water to the FEP bottle until the balance reads 100 g.

3. Add Tc solution to each experimental container
   a. Add ~25 g of the 30 ppb Tc solution to each TcS1-pHi container

4. Add sodium sulfite to each experimental container
   a. Add mass of sodium sulfite given in Table 1 to the sample containers

5. Adjust pH of experimental solutions
   a. Use an Eppendorf micropipettor and associated tip to add HCl to each experimental container. The concentration and approximate volume of acid or base to be added to each container is given in Table 1.

6. Cap the tubes tightly and place on gyratory shaker.

C. Measure solution pH

1. Measure and record the pH each solution periodically (every day). Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.

2. When pH appears to be stable proceed to next step (Eh measurement).

6/21/00
pr

TcS-1 Experiment

D. Measure solution Eh

Measure ORP (oxidation-reduction potential) of experimental solutions by following the method outlined below.

1. Prepare redox reference quinhydrone solutions
   a. Mix 50 ml of pH 4 buffer solution with 0.5 g of quinhydrone in a plastic cup.
   b. Mix 50 ml of pH 7 buffer solution with 0.5 g of quinhydrone in a plastic cup.

   *Be sure that excess quinhydrone is used in each solution so that crystals are always present. These solutions are stable for only 8 hrs. Table 3 in ASTM Method D1498-93 lists the nominal millivolt readings for the reference solutions.

2. Prepare ZoBell's solution
   a. In a 1L volumetric flask add 1.4080 g potassium ferrocyanide, 1.0975 g potassium ferricyanide, and 7.4557 g potassium chloride.
   b. Dissolve in ultrapure water and dilute solution to 1L.
   c. Store the solution in a dark bottle, label, and refrigerate. The solution is stable for at least 90 days if kept chilled at 4 °C.
   d. Table 6.5-3 of the U.S. Geological Survey TWRI Book 9 lists the Eh of ZoBell's solution as a function of temperature.

3. Check response of ORP electrode to the standard redox solutions. Readings should be within 30 mV of the value expected for the standard solution.

   If response of ORP electrode is acceptable then proceed to the next step, otherwise contact principal investigator for instructions.

4. Measure ORP of experimental solutions
   a. Place a stir bar in each experimental tube to insure agitation of solution during measurement.
   b. Measure the ORP of the sample solution by immersing the electrode directly into the sample tube and record the millivolt potential. Also record temperature of experimental solutions using a calibrated thermometer.
   c. The Eh of each solution can be calculated using the following formula

      Eh = Eobs + Eref

      Where Eh is oxidation-reduction potential referred to the hydrogen scale, mV; Eobs is the observed oxidation-reduction potential of the platinum electrode, mV; and Eref is the oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

Table 1. Container labels, estimated solution pH, mass of Na2SO3 to add, and volume of HCl solution needed for adjustment of pH and Eh in 0.01 NaCl solutions with 30 ppb Tc. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated Solution pH	Mass of Na2SO3 Needed, g	Volume of HCl Needed, ml	Molarity
TcS1-pH2.5	2.5	0.0032	0.2	1.0
TcS1-pH3.3	3.3	0.0032	0.1	1.0
TcS1-pH4.0	4.0	0.0032	0.04	1.0
TcS1-pH4.1	4.1	0.0016	0.02	1.0

6/21/00
pr

TcS-1 Experiment – Preparation of ^{99}Tc soln. (30 ppb)

Reagents: ^{99}Tc Spike solution 43A (300 ppb)
Previously prepared GC 031/289 on 6/23/99
5×10^{-3} uCi/g ; 2.97×10^{-7} M Tc in 0.1 M NaCl matrix

Sodium sulfite
Mfg. Fisher Chemical
Lot No. 905923

Hydrochloric ~~acid~~ 6/21/00 pr acid solns.
Previously prepared GC 309/55 on 2/25/00 by RC

Procedure:

1. added ~10 ml of ^{99}Tc Spike 43A using an eppendorf micro-pipettor to a tared 125 ml FEP bottle and weighed on the analytical balance.

2. added nanopure D.I. water to FEP bottle to bring weight up to ~100 grams.

Weight of added Tc Spike 43A = 10.0600 g
Weight of Tc Spike 43A + D.I. H2O = 100.0147 g

3. added ~25 grams of ^{99}Tc soln. (30 ppb) to 4 individual tared PC centrifuge tubes and weighed. Added HCl as per table 1:

Labeled PC Sample Tube	Weight of added Tc soln.	Quantity of added 1.0M HCl
TcS-1 pH 2.5	24.9276 g	200 uL
TcS-1 pH 3.3	25.0023 g	100 uL
TcS-1 pH 4.0	24.9938 g	40 uL
TcS-1 pH 4.1	24.9479 g	20 uL

added Na_2SO_3 as required in Table 1. pg 78

TcS-1 Experiment

6/21/00 RE

Procedure - cont'd.

4. Tightly capped the PC sample tubes and placed in tube rack. Placed rack on gyratory shaker.

6/30/00 RE

5. Measured pH of sample tube solutions:

Sample Tube I.D.	Sample pH	~~mV~~ Eh RE	Eh
TcS-1 2.5	2.224	229	433
TcS-1 3.3	2.648	213	417
TcS-1 4.0	3.996	179	383
TcS-1 4.1	3.251	223	427

2 pt. calibration of pH meter using 2.00 and 4.00 buffers.

pH buffer soln. 2.00
Fisher Scientific
Lot No. 996387-24
Exp. date 9/2001

pH buffer soln. 4.00
Fisher Scientific
Lot No. 994523-24
Exp. date 7/2001

slope = 97.6

7/3/00 RE

The Eh values of the TcS-1 experiment samples were higher than required to ~~confirm~~ indicate a reducing environment. RE 7/3/00

Additional Na_2SO_3 (Lot No. 905923, Fisher Chemical) was added to each sample tube in the quantity of approximately 0.03 grams. Exact weights were not measured. If reduction (lower Eh value) is accomplished, a new experiment will be run.

TcS-1 Experiment

7/4/00 RE

pH meter (Orion model 920A) was calibrated with two standards (as per 309/80). Slope = 97.7

Zobell's solution standard measured 239 mV @ 19.7°C.

Measured pH and Eh of modified TcS-1 samples:

Sample Tube I.D.	Sample pH	mV	Eh	Temp.
TcS pH 2.5	7.78	16.6	~~not calculated~~ RE 7/4/00	19.5°C
TcS pH 3.3	8.07	11.1		19.5°C
TcS pH 4.0	8.51	7.4		19.6°C
TcS pH 4.1	8.77	10.8		19.7°C

7/5/00 RE

Added concentrated HCl (Lot No. 418110, Fisher Chemical) to Samples TcS pH 2.5 + TcS pH 4.0 drop by drop while continuously measuring the pH. Reduced pH of each sample to ~3.0. Capped tubes and placed on shaker overnight to equilibrate.

7/7/00 RE

Measured pH + EH of TcS samples modified with additional HCl:

Sample I.D.	pH	mV	Eh	Temp.
TcS pH 2.5	2.554	186	390	19.8°C
TcS pH 4.0	2.800	165	369	19.8°C

RE 7/7/00

TcS-2 Experiment

Test Method for Tc solution reduction
Experiment TcS2

Written by: J.D. Prikryl
Revision No.: 0

Date Written: July 18, 2000
Date Revised: N/A

CONDITIONS:

1. Σ Tc = 30 ppb
2. 0.01 m NaCl matrix
3. pH range 2.5 to 4.0
4. Initial solution volume = 25 ml

OBJECTIVES:

To investigate the effects of reduction on the pH and Eh of Tc solutions.

METHOD:

Add sodium sulfite to Tc solutions.

EQUIPMENT:

Gyratory shaker
Orion pH/mV/ISE/°C meter
Combination pH electrode
Combination platinum redox/ORP and silver:silver chloride electrodes
ATC probe
Calibrated thermometer
Eppendorf micropipettors
Oxford macropipettors
Analytical balances (Mettler 4600 and 240AE)

SUPPLIES:

50 ml polycarbonate centrifuge tubes (acid washed and dried)
Eppendorf and Oxford pipette tips
pH buffer solutions
Reagent grade HCl
300 ppb Tc stock solution (spike 43A)
ultrapure water
stir bars
Potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$ (lot no. _____)
potassium ferricyanide, $K_3Fe(CN)_6$ (lot no. _____)
potassium chloride, KCl (lot no. _____)
quinhydrone (lot no. _____)
sodium sulfite, Na_2SO_3 (lot no. _____)
glassware and plasticware as needed

TcS-2 Experiment

PROCEDURE:

A. Stock Solution Preparation

Using reagent grade chemicals, prepare the following stock solutions for use in adjustment of pH of the experimental solutions. The following stock solutions are required:

1. 1.0 M HCl
2. 0.1 M HCl

Note These solutions should be available from earlier sorption experiments.*

B. Experimental Solution Preparation

1. Label six (4) 50 ml polycarbonate centrifuge tubes (e.g., TcS2-pHi – where i is the approximate pH of each solution, see Table 1).

2. Prepare 30 ppb Tc solution (total of 100 g)
   a. Tare a FEP bottle on the Mettler 4600 balance.
   b. Add 10 g of the 300 ppb Tc stock solution (spike 43A) to the tared FEP bottle.
   c. Add ultrapure water to the FEP bottle until the balance reads 100 g.

3. Add Tc solution to each experimental container
   a. Add ~25 g of the 30 ppb Tc solution to each TcS2-pHi container

4. Add sodium sulfite to each experimental container
   a. Add mass of sodium sulfite given in Table 1 to the sample containers

5. Adjust pH of experimental solutions
   a. Use an Eppendorf micropipettor and associated tip to add HCl to each experimental container. The concentration and approximate volume of acid or base to be added to each container is given in Table 1.

6. Cap the tubes tightly and place on gyratory shaker.

C. Measure solution pH

1. Measure and record the pH each solution periodically (every day). Calibrate the pH meter and electrode over the expected range of pH. Record pH meter and probe calibration in the scientific notebook.

2. When pH appears to be stable proceed to next step (Eh measurement).

TcS-2 Experiment

D. Measure solution Eh

Measure ORP (oxidation-reduction potential) of experimental solutions by following the method outlined below.

1. Prepare redox reference quinhydrone solutions
   a. Mix 50 ml of pH 4 buffer solution with 0.5 g of quinhydrone in a plastic cup.
   b. Mix 50 ml of pH 7 buffer solution with 0.5 g of quinhydrone in a plastic cup.

   *Be sure that excess quinhydrone is used in each solution so that crystals are always present. These solutions are stable for only 8 hrs. Table 3 in ASTM Method D1498-93 lists the nominal millivolt readings for the reference solutions.

2. Prepare ZoBell's solution
   a. In a 1L volumetric flask add 1.4080 g potassium ferrocyanide, 1.0975 g potassium ferricyanide, and 7.4557 g potassium chloride.
   b. Dissolve in ultrapure water and dilute solution to 1L.
   c. Store the solution in a dark bottle, label, and refrigerate. The solution is stable for at least 90 days if kept chilled at 4 °C.
   d. Table 6.5-3 of the U.S. Geological Survey TWRI Book 9 lists the Eh of ZoBell's solution as a function of temperature.

3. Check response of ORP electrode to the standard redox solutions. Readings should be within 30 mV of the value expected for the standard solution.

   If response of ORP electrode is acceptable then proceed to the next step, otherwise contact principal investigator for instructions.

4. Measure ORP of experimental solutions
   a. Place a stir bar in each experimental tube to insure agitation of solution during measurement.
   b. Measure the ORP of the sample solution by immersing the electrode directly into the sample tube and record the millivolt potential. Also record temperature of experimental solutions using a calibrated thermometer.
   c. The Eh of each solution can be calculated using the following formula

      Eh = Eobs + Eref

      Where Eh is oxidation-reduction potential referred to the hydrogen scale, mV; Eobs is the observed oxidation-reduction potential of the platinum electrode, mV; and Eref is the oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

Table 1. Container labels, estimated solution pH, mass of Na2SO3 to add, and volume of HCl solution needed for adjustment of pH and Eh in 0.01 NaCl solutions with 30 ppb Tc. Amount of reagent needed for pH adjustment was calculated using EQ3 v7.2b.

Sample Label	Estimated Solution pH	Mass of Na2SO3 Needed, g	Volume of HCl Needed, ml	Molarity
TcS2-pH2.5	2.5	0.063	1.2	1.0
TcS2-pH3.0	3.0	0.063	1.0	1.0
TcS2-pH3.5	3.5	0.063	0.9	1.0
TcS2-pH4.5	4.5	0.063	0.8	1.0

TcS-2 Experiment

7/20/00 pl

Preparation of 30ppb ^{99}Tc solution:
Weighed ~10 g Tc 300 ppb solution from Spike 43A.
Wt. of 300 ppb Tc soln. = 10.1560 g
Wt. of Tc soln. + D.I. H_2O = 100.7960 g

Pipetted ~25 g of prepared 30 ppb ^{99}Tc solution into tared PC centrifuge tubes:

Wt. of 30ppb Tc soln	Sample I.D.	Wt. of Na_2SO_3	added 1M HCl
24.8708 g	TcS2-pH2.5	0.0641 g	1.2 mL
25.0793 g	TcS2-pH3.0	0.0639 g	1.0 mL
25.0842 g	TcS2-pH3.5	0.0637 g	0.9 mL
25.0504 g	TcS2-pH4.5	0.0646 g	0.8 mL

Added required Na_2SO_3 and HCl pH adjuster from Table 1 pg. 84. Capped PC tubes and placed on shaker overnight to equilibrate.

7/21/00 pl

Measured pH of sample tube solutions and measured mV using Eh probe

Sample I.D.	pH	mV	Temp °C
TcS2-pH2.5	1.728	264	19.6
TcS2-pH3.0	1.990		
TcS2-pH3.5	2.117		
TcS2-pH4.5	2.236		

(mV measured on 7/24/00; temp measured on 7/21/00.)

7/26/00 pl

Measured pH and Eh of sample tube solns.
2 pt. calibration of pH meter and probe yielded slope = 98.4

Sample I.D.	pH	mV	Eh Interpolated	Temp °C
TcS2-pH2.5	1.736	285	490	19.4
TcS2-pH3.0	1.952	270	475	19.4
TcS2-pH3.5	1.866	266	470	19.6
TcS-2 pH4.5	1.867	266	470	19.6
Zobell's soln.	NA	237	446	19.7

7/26/00
pe

Preparation of Zobell's Solution used verifying the sensitivity and accuracy of the Corning Platinum Redox Combination probe. - Cat. No. 476516.

Procedure from U.S. Geological Survey TWRI Book 9 - Redox pg. 5

REDOX — 5

pe 7/26/00

CAUTION: The standard hydrogen reference electrode (SHE) can be dangerous and is not recommended for field use.

- Silver:silver-chloride or calomel reference electrodes are the redox electrodes in common use.
- The Orion™ combination electrodes are platinum redox and silver:silver-chloride reference electrodes in one body (the Orion™ brand is used for purposes of illustration only).

ZoBell's solution. ZoBell's is the standard solution for testing redox instruments. ZoBell's solution can be obtained from the QWSU in Ocala, Fla., or it can be prepared fresh (see below). Quinhydrone solution is sometimes used but is not recommended because it is significantly less stable above 30°C and its temperature dependence is not as well defined as that of ZoBell's.

ZoBell's solution consists of a 0.1 molal KCl solution containing equimolal amounts of $K_4Fe(CN)_6$ and $K_3Fe(CN)_6$. ZoBell's is reported stable for at least 90 days if kept chilled at 4°C. To prepare ZoBell's solution:

1. Weigh the chemicals (dry chemicals should be stored overnight in a desiccator before use).
   - 1.4080 g $K_4Fe(CN)_6 \cdot 3H_20$ (Potassium ferrocyanide)
   - 1.0975 g $K_3Fe(CN)_6$ (Potassium ferricyanide)
   - 7.4557 g KCl (Potassium chloride)
2. Dissolve these chemicals in deionized water and dilute solution to 1,000 mL.
3. Store the solution in a dark bottle, clearly labeled with its chemical contents, preparation date, and expiration date. Keep the solution chilled.

CAUTION: ZoBell's solution is toxic—handle with care.

Reagents: Used same chemicals with same Lot numbers as per 309/61.

Wt. of $K_4Fe(CN)_6 \cdot 3H_2O$ = 1.4024 g
Wt. of $K_3Fe(CN)_6$ = 1.0974 g
Wt. of KCl = 7.4560 g
Labeled as "Zobell's Solu." expiration date 10/26/00.

7/26/00
pe

SMF Well Cuttings sample preparation for chemical, XRD and analyses:

Samples were prepared by removing representative splits of well samples and powdered by placing in a tungsten-carbide vial and placing in the SPEX 8000 Mixer Mill for 10 minutes.

SMF Sample I.D.	Physical Description of Raw Sample	Method for obtaining Split
1010637	Homogenous colored coarse particles - beige, gray, white & rust. Some larger pieces of all colors 50 to 75 mm across.	Quartered entire sample.
1010638	Same as sample 1010637.	Quartered entire sample.
1010648	Same colors as sample 1010637. Particle sizes ranging from coarse sand size to ~~7/26/00~~ pe 10 to 40 mm, with many 1 to 1.5 cm size.	Separated some larger dark colored pieces 0.5 to 1.5 cm. Separated some larger dark grey pieces 2.0 to 2.5 cm. Quarted remaining sample.
	Dark colored pieces labeled as 1010648-A Grey colored pieces labeled as 1010648-B Remaining quartered sample as 1010648	
1010649	Homogenous sized particles like coarse sand. Evenly divided colors of beige, white and dark grey.	Quartered entire sample.

7/26/00 pe	SMF Sample I.D	Physical Description of Raw Sample	Method for obtaining split
	1010650	Particle size mostly course sand-like. Color range of beige, grey + chalk, mostly chalk colored. Homogenous. Larger size pieces range 30 to 40 mm.	Quartered entire sample.
	1010653	Sand size particles with some larger 30 to 70 mm sizes. Homogenous small grains mostly sand colored.	Quartered entire sample.
	1010654	Coarse grains, same as sample 1010653, some larger pieces 0.75 to 1.5 cm, gray. Grey colored pieces labeled as 1010654-A.	Separated larger, grey fraction. Quartered remaining sample.
	1010655	Homogenous fine particles, mostly beige colored with some chalk and grey.	Quartered entire sample.
7/27/00 pe	1010656	Coarse grained, mostly sand and chalk colored, less grey.	Separated grey pieces 1 to 2 cm. Separated white colored pieces 1 to 1.5 cm. Quartered remaining small grains.
		Grey colored pieces labeled as 1010656-A White pieces labeled as 1010656-B	

7/27/00 pe	SMF Sample I.D.	Physical Description of Raw Sample	Method for obtaining split
	1010657	Coarse beige particles with larger conglomerate pieces of same color .75 to 2.5 cm.	Quartered entire sample.
	1010661	Fine to coarse beige particles. Homogeneous in color.	Quartered entire sample.
	1010665	Fine to course beige and grey pieces. Fairly homogeneous. pe 7/27/00	Quartered entire sample.
	1010666	Same as sample 1010665	Quartered entire sample.
	1010667	Same as sample 1010665	Quartered entire sample.
	1010668	Fine to coarse homogenous grains, mostly beige with grey and some chalk colored pieces. Larger sizes are 0.10 to 0.75 cm.	Quartered entire sample.
	1010684	Fine to large particles, mostly sand colored with some grey pieces.	Quartered entire sample.
	1010685	Fine to course particles. ~~Beige~~ pe 7/27/00 Mostly sand colored, some beige, grey and chalk. Largest sizes 0.5 to 1.5 cm.	Quartered entire sample.

	SMF Sample I.D.	Physical Description of Raw Sample	Method for Obtaining Split
7/27/00 nr	1010690	Brown colored coarse to 1.5 cm pieces. Two large pieces 3 cm across. Large pieces same in color and appear to be conglomerate of finer particles.	Separated large piece and quartered. Quartered remaining smaller particles.
nr 7/27/00		Large piece labelled as 1010690-A	nr 7/27/00
	1010694	Brown, some chalk and ~~brown~~ grey particles interspersed. Homogeneous, coarse particles.	nr 7/27/00 Quartered entire sample.
7/28/00 nr	Samples from Borehole: NC-Washburn-1X		
	1010841	Fine brown clay colored particles mixed with small to large, 0.1 to 5.0 cm grey rock. Some large chalk colored rocks.	Separated grey rock. Quartered remaining sample.
		Grey rock sample ~~lost~~ nr 7/31/00 labeled as 1010841-A	
	1010842	Same as sample 1010841.	Quarted entire sample, did not include largest grey rock.
	1010852	Homogenous beige and chalk colored grains with same colored rock pieces 0.5 to 2.0 cm.	Quartered entire sample.
	1010853	Fine to coarse particles, brown, grey and chalk colored. Large pieces 1 to 2 cm.	Quartered entire sample.

	SMF Sample I.D.	Physical Description of Raw Samples	Method for Obtaining Split
7/28/00 nr	1010854	Coarse grains of brown and grey. Large pieces 0.5 to 1.5 cm. Few pieces of mottled rock.	Quartered entire sample.
	1010855	Grey rock pieces 0.5 to 2.25 cm. Small quantity of brown + grey coarse sand particles.	Quartered entire sample.
	1010856	Brown and grey fine to coarse particles. Larger pieces are grey and chalk colored, 0.5 to 1.5 cm.	Quartered entire sample.
	1010864	Multi-colored coarse particles and rock pieces up to 2.5 cm. Brown beige, chalk and rust colored.	Quartered entire sample.
	1010865	Same as sample 1010864	Quartered entire sample.
	~~1010866~~	nr 7/28/00	
	1010866	Same as sample 1010864	Quartered entire sample.
	1010867	Coarse beige, brown, grey, chalk and rust colored pieces up to 2.0 cm.	Quartered entire sample.
	1010868	Same as sample 1010867	Quartered entire sample.
7/31/00 nr	1010869	Similar to sample 1010867.	Quartered entire sample.

7/31/00
M

SMF Sample I.D.	Physical Description of Raw Sample	Method for obtaining Split
1010870	Coarse to 4.0 cm pieces of light brown clay-like piece with a few small pieces of grey rock.	Quartered entire sample.
1010871	Fine to coarse brown, grey, some chalk + rust colored pieces. Largest 1.5 cm.	Quartered entire sample.
1010872	Fine to coarse brown, grey, more chalk than previous sample. Chalk + grey colored rock 1.0 to 2.0 cm.	Quartered entire sample.
1010873	Fine to coarse mostly brown particles. Small pieces of rock chalk and brick-red color.	Quartered entire sample.
1010876	Fine to coarse particles. Rock sizes 0.5 to 1.5 cm. Similar in color as sample 1010873 but with more red rock.	Quartered entire sample.
1010877	Similar to sample 1010876.	Quartered entire sample.
1010878	Similar to sample 1010876	Quartered entire sample.

7/31/00
M

Samples from borehole NC-EWDP-32D

SMF Sample I.D.	Physical Description of Raw Samples	Method for obtaining Split
1010549	Light brown-grey dust to 0.5 cm pieces. Some grey rock pieces ~~0.05~~ to 1.0 cm. 7/31/00 0.05 PB 03/08/02	Quartered entire sample.
1010550	Similar to sample 1010549 but with several large 2.5 cm grey rock pieces.	Quartered entire sample.
1010566	Fine light brown particles with grey rock pieces 0.075 to 2.5 cm. One large grey rock 4.5 cm. Grey rock sample I.D. 1010566-A	Separated grey rock. Quartered remaining sample.
1010567	Similar to sample 1010566	Quartered entire sample
1010570	Fine to coarse brown and grey particles. Rock pieces .05 to 1.25 cm.	Quartered entire sample.
1010571	Coarse brown particles. Grey rock pieces coated with brown dust 0.05 to 2 cm. Grey rock pieces sampled as 1010571-A	Separated grey rock pieces. Quartered remaining sample.
1010578	Coarse brown and grey particles. Few large conglomerate of loosely bound grey and brown particles.	Quartered entire sample after crushing conglomerate pieces.

8/1/00 pe	SMF Sample I.D.	Physical Description of Raw Sample	Method for Obtaining Split
	1010579	Brown clay colored particles with some grey rock pieces 3.0 to 80.0 mm. One large quartz-like piece 3.0 cm.	Separated quartz-like rock. Quartered remaining sample.
		Quartz-like rock sampled as 1010579-A	
	1010583	Similar to sample 1010579 with more grey rock pieces. One large grey rock 4.0 cm	Quartered entire sample excluding large grey rock.
	1010584	Fine brown particles with many grey rock pieces. Several white rock pieces 0.50 to 1.5 cm.	Separated white rock pieces. Quartered remaining sample.
		White rock sampled as 1010584-A	
	1010603	Fine to coarse beige, grey and rust colored particles. Rock pieces of multi colors 0.50 to 1.0 cm.	Quartered entire sample.
	1010602	Similar to sample 1010603.	Quartered entire sample.
	1010663	Fine to coarse beige, grey, dark colored pieces. Rock pieces of same colors 0.25 to 1.5 cm.	Quartered entire sample.
	1010664	Fine to coarse. Similar to Sample 1010663	Quartered entire sample.

8/1/00 pe	SMF Sample I.D.	Physical Description of Raw Sample	Method for Obtaining Split
	1010669	Fine to coarse mostly brick red colored particles. Few pieces over ~~0.05~~ pe 8/1/00 ~~0.0~~ mc 8/1/00 0.50 cm.	Quartered entire sample.
	1010670	Fine to coarse beige, some grey and ~~chalk~~ mc 8/1/00 chalk colored particles. Rock pieces 0.25 to 1.0 cm.	Quartered entire sample.
	1010701	Mostly brown with ~~grey~~ pe 8/1/00 chalk and grey colored pieces. Some large conglomerate pieces upto 4.0 cm.	Separated large pieces. Quartered remaining sample. Crushed large pieces and added to sample.
	1010702	Similar to sample 1010701	Quartered entire sample.
	1010703	Mostly fine brown, grey, some beige colored particles.	Quartered entire sample.
8/2/00 pe	QA Prepared check standards:		
	1010500	WS10 60/100 mesh * UC * RC * RFe * HL prepared 10-5-94 by MA removed.	Approx. 7 grams
	1010501	Death Valley Jct. Clinoptilolite #25-35	
	1010900	NBS 70A Potassium Feldspar	

9/11/00 PRD

copy made for QA archives

9/15/00 PRD

As noted on pp. 87 - 95 of this notebook, selected cuttings samples from Nye County EWDP wells NC-Washburn-1X and NC-EWDP-02D were processed in preparation for chemical and mineralogical analyses. The samples will be analyzed using XRF and ICP-MS, XRD, and with thin sections (thin sections will be produced using non-crushed cuttings as described below).

As described on p. 87, each cuttings sample was subsampled so that a few grams could be crushed and powdered. The powders were placed in 15 mL polypropylene vials and appropriately labeled. Original SMF sample numbers were used for each sample, except when additional or more than one powder was made from a cuttings sample - an additional letter designator was used. Since all sample numbers began with 1010, only the final 3 digits from each sample number were used for labeling.

Additional samples were prepared for inclusion as blind samples or check standards. These samples were selected in an attempt to provide known standards and bracket the range of expected values from analyses. XRD blinds are listed on p. 95 as #'s 500, 501, and 900. Other sample blinds include

502 - also NBS 70a K feldspar (same as 900)
503 - NBS 99a Na feldspar
504 - NBS 278 obsidian rock
505 - RGM 1 rhyolite (USGS standard)

These samples are already powdered as purchased.

9/15/00 PRD

Samples for XRD and XRF/ICP-MS were split from the same ~~powder~~ 9/15/00 powders. Splits were labeled with an X or C depending on the expected analysis type (X for XRD and C for chemical).

XRD analyses were accomplished at Div 18 (Jim Spencer) in August 2000. XRF and ICP-MS analyses will be done at the Geoanalytical Lab at Wash. State University.

A subset of the cuttings to be analyzed chemically and by XRD was selected for thin section preparation. In this case the cuttings were not crushed. Thin section prep will be done at Mineral Optics Laboratory.

Specimen I.D.	
500-x	* - QUARTZ
501-x	* - CDV
549-x	
550-x	
566-x	
566-ax	
567-x	
570-x	
571-x	
571-ax	
578-x	
579-x	
579-ax	
583-x	
584-x	
584-ax	
602-x	
603-x	
637-x	
638-x	
648-ax	
648-bx	
648-x	
649-x	
650-x	
653-x	
654-x	
654-ax	
655-x	
656-x	
656-ax	
656-bx	
657-x	
661-x	
663-x	
664-x	
665-x	

Specimen I.D.	
666-x	
667-x	
668-x	
669-x	
670-x	
684-x	
685-x	
690-x	
690-ax	
694-x	
701-x	
702-x	
703-x	
841-x	
841-ax	
842-x	
852-x	
853-x	
854-x	
855-x	
856-x	
864-x	
865-x	
866-x	
867-x	
868-x	
869-x	
870-x	
871-x	
872-x	
873-x	
876-x	
877-x	
878-x	
900-x	* - NBS 70A

Sample list for XRD analyses

sent out 8/4/00

9/15/00 PB — Sample list for XRF / ICP-MS sent to WSU. as of 8/9/00.

GeoAnalytical Laboratory XRF or ICP-MS Submittal Form

Name: **Paul Bertetti**
Address: **CNWRA, Southwest Research Institute**
6220 Culebra Road
San Antonio, TX 78238-5166
Phone: **(210) 522-5228** Fax: **(210) 522-5184**
Analysis Requested: **both XRF and ICP-MS**

	Sample No.	Rock Province	Rock Type	State	County	Latitude	Longitude
1	501 *	All are from	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
2	502 *	Southwestern	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
3	503 *	Nevada	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
4	504 *	Volcanic Field	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
5	505 *	within Southern	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
6	549	Basin and	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
7	550	Range Prov.	alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
8	566		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
9	566A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
10	567		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
11	570		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
12	571		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
13	571A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
14	578		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
15	579		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
16	579A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
17	583		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
18	584		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
19	584A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
20	602		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
21	603		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
22	637		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
23	638		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
24	648		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
25	648A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
26	648B		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
27	649		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
28	650		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
29	653		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
30	654		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
31	654A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
32	655		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
33	656		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
34	656A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
35	656B		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
36	657		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
37	661		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
38	663		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
39	664		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"

location data req'd because WSU's analytical equipment was funded by NSF. (*) standards as noted on pp. 95 and 96. * included standards

9/15/00 PB — Sample list for WSU (cont'd.)

	Sample No.	Rock Province	Rock Type	State	County	Latitude	Longitude
40	665		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
41	666		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
42	667		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
43	668		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
44	669		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
45	670		tuff	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
46	684		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
47	685		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
48	690		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
49	690A		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
50	694		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
51	701		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
52	702		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
53	703		alluvium	NV	Nye	36° 39' 38.521"	116° 27' 56.834"
54	841		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
55	841A		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
56	842		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
57	852		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
58	853		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
59	854		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
60	855		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
61	856		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
62	864		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
63	865		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
64	866		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
65	867		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
66	868		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
67	869		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
68	870		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
69	871		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
70	872		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
71	873		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
72	876		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
73	877		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"
74	878		alluvium	NV	Nye	36° 39' 50.772"	116° 25' 26.835"

PB 9/15/00

9/15/00 KB

Sample list for thin section preparations

Mineral Optics Laboratory
20 "A" Street P.O. Box 828 Tel: 802-295-0373
Wilder, Vermont 05088 US FAX: 802-295-7540

Petrography Order Checklist

Bill To: Name
Dept
Company
Address
City State ZIP

Ship To: Name
Dept.
Company
Street Address
City State ZIP

Telephone
Date Received Order No.
Date Entered Date Mailed

Date _____ Customer P.O. No. _____
VISA / MASTERCARD
No. _____ Exp. Date

Sample No.	.03 mm Thin Sections: Quantity	27x46mm Top Polished	27x46mm Lap no cover	27x46mm Lap w/ cover	50x75 Lap no cover	50x75 Lap w/ cover	35D Lap no cover	35D Top Polished	___ mm Thick: 2 Side Polished	27 x 46 mm Top Polished	Impregnate: Clear Resin	Color Resin	Cut: Bulk Sample	Oriented	Wafer Remailbe	Stain	ASTM Lap Cons	ASTM Lap slab
500 (#)	1	X									X							
578	1	X									X				X			
584	1	X									X							
649	1	X									X				X			
650	1	X									X							
654 AT	1	X									X							
656 AT	1	X									X							
656 BT	1	X									X							
657	1	X									X				X	→ HOT MAFIC ?		
661	1	X									X				X			
664	1	X									X				X			
665	1	X									X				X			
666	1	X									X				X			
684	1	X									X							
690	1	X									X				X			
690 BT	1	X									X							
701	1	X									X							
855	1	X									X							
856	1	X									X				X			
861	1	X									X				X			
867	1	X									X							
868	1	X									X							
869	1	X									X							

500 (#) - quartz, not powdered.

stain requested for feldspars

9/15/00 KB

For completeness, sample weights of cuttings as originally shipped were recorded by Cherrington ~~prior~~ 9/15/00 prior to selecting samples for analyses. These are listed on the next few pages and coincide with the spreadsheet values following.

CSITS v.1

SMF Specimen Custody Receipt Copy

Ship To:
Paul Bertetti
CNWRA/SWRI
6220 Culebra Road
Building 57
San Antonio, TX 78238-5166

Date Received: _____
Shipment ID: 01000358 Shipping Date: 27-dec-1999
SMF Geotechnician Thomas M Yokum Date 12-23-99

Container ID: 01002655

Specimens in this container: 62

Specimen ID	Type	Top	Bottom	Parent Borehole:	
01010819	Cuttings	5.0	10.0	NC-Washburn-1X	130.99
01010820	Cuttings	15.0	20.0	NC-Washburn-1X	137.23
01010821	Cuttings	25.0	30.0	NC-Washburn-1X	155.23
01010822	Cuttings	35.0	40.0	NC-Washburn-1X	149.54
01010823	Cuttings	45.0	50.0	NC-Washburn-1X	160.90
01010824	Cuttings	55.0	60.0	NC-Washburn-1X	159.37
01010825	Cuttings	65.0	70.0	NC-Washburn-1X	136.44
01010826	Cuttings	75.0	80.0	NC-Washburn-1X	142.24
01010827	Cuttings	85.0	90.0	NC-Washburn-1X	183.27
01010828	Cuttings	95.0	100.0	NC-Washburn-1X	146.48
01010829	Cuttings	105.0	110.0	NC-Washburn-1X	157.68
01010830	Cuttings	115.0	120.0	NC-Washburn-1X	196.88
01010831	Cuttings	125.0	130.0	NC-Washburn-1X	151.93
01010832	Cuttings	135.0	140.0	NC-Washburn-1X	137.73
01010833	Cuttings	145.0	150.0	NC-Washburn-1X	184.58
01010834	Cuttings	155.0	160.0	NC-Washburn-1X	147.51
01010835	Cuttings	165.0	170.0	NC-Washburn-1X	144.45
01010836	Cuttings	175.0	180.0	NC-Washburn-1X	155.39
01010837	Cuttings	185.0	190.0	NC-Washburn-1X	161.15
01010838	Cuttings	196.0	200.0	NC-Washburn-1X	154.54
01010839	Cuttings	205.0	210.0	NC-Washburn-1X	~~155.45~~
01010840	Cuttings	215.0	220.0	NC-Washburn-1X	162.06
01010841	Cuttings	225.0	230.0	NC-Washburn-1X	179.10
01010842	Cuttings	235.0	240.0		
01010843	Cuttings	245.0	250.0		

2.19.53

I hereby acknowledge the receipt of the Specimens listed above.
I will return this form to the SMF within 10 business days of receipt.

Recipient _____ Date _____

nd return to:
Facility
Characterization Project

i17

Page 4 of 6: 2 Containers shipped.

9/15/00 PM

SMF Specimen Custody Receipt

CSITS v.1

Requestor:	Ship To:	
Paul Bertetti **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	**Paul Bertetti** **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	Date Received: _____ Shipment ID: 01000358 Shipping Date: 27-dec-1999 SMF Geotechnician Susan M Nelson Date 12-23-99

Container ID: 01002655

Specimens in this container: 62

Specimen ID	Type	Top	Bottom	Parent Borehole:	
01010844	Cuttings	255.0	260.0	NC-Washburn-1X	140.28
01010845	Cuttings	265.0	270.0	NC-Washburn-1X	173.88
01010846	Cuttings	275.0	280.0	NC-Washburn-1X	151.35
01010847	Cuttings	285.0	290.0	NC-Washburn-1X	154.97
01010848	Cuttings	295.0	300.0	NC-Washburn-1X	118.23
01010849	Cuttings	305.0	310.0	NC-Washburn-1X	151.99
01010850	Cuttings	315.0	320.0	NC-Washburn-1X	131.62
01010851	Cuttings	325.0	330.0	NC-Washburn-1X	142.80
01010852	Cuttings	335.0	340.0	NC-Washburn-1X	114.97
01010853	Cuttings	345.0	350.0	NC-Washburn-1X	142.16
01010854	Cuttings	355.0	360.0	NC-Washburn-1X	132.25
01010855	Cuttings	365.0	370.0	NC-Washburn-1X	127.55
01010856	Cuttings	375.0	380.0	NC-Washburn-1X	126.66
01010857	Cuttings	385.0	390.0	NC-Washburn-1X	174.62
01010858	Cuttings	395.0	400.0	NC-Washburn-1X	132.72
01010859	Cuttings	410.0	415.0	NC-Washburn-1X	148.71
01010860	Cuttings	420.0	425.0	NC-Washburn-1X	155.91
01010861	Cuttings	455.0	460.0	NC-Washburn-1X	152.33
01010862	Cuttings	465.0	470.0	NC-Washburn-1X	120.06
01010863	Cuttings	475.0	480.0	NC-Washburn-1X	159.91
01010864	Cuttings	485.0	490.0	NC-Washburn-1X	129.12
01010865	Cuttings	495.0	500.0	NC-Washburn-1X	167.92
01010866	Cuttings	505.0	510.0	NC-Washburn-1X	142.87
01010867	Cuttings	515.0	520.0	NC-Washburn-1X	127.22
01010868	Cuttings	525.0	530.0	NC-Washburn-1X	124.30

Please Sign this form and return to:
Sample Management Facility
Yucca Mountain Site Characterization Project
P.O. Box 617
Mercury, NV 89023-0617

I hereby acknowledge the receipt of the Specimens listed above. I will return this form to the SMF within 10 business days of receipt.

Recipient _____ Date _____

Page 5 of 6: 2 Containers shipped.

9/15/00 PM

SMF Specimen Custody Receipt

CSITS v.1

Requestor:	Ship To:	
Paul Bertetti **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	**Paul Bertetti** **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	Date Received: _____ Shipment ID: 01000358 Shipping Date: 27-dec-1999 SMF Geotechnician Susan M Nelson Date 12-23-99

Container ID: 01002655

Specimens in this container: 62

Specimen ID	Type	Top	Bottom	Parent Borehole:	
01010869	Cuttings	535.0	540.0	NC-Washburn-1X	131.38
01010870	Cuttings	545.0	550.0	NC-Washburn-1X	116.14
01010871	Cuttings	555.0	560.0	NC-Washburn-1X	131.74
01010872	Cuttings	570.0	575.0	NC-Washburn-1X	149.44
01010873	Cuttings	580.0	585.0	NC-Washburn-1X	170.46
01010874	Cuttings	590.0	595.0	NC-Washburn-1X	133.72
01010875	Cuttings	600.0	605.0	NC-Washburn-1X	183.99
01010876	Cuttings	610.0	615.0	NC-Washburn-1X	161.21
01010877	Cuttings	620.0	625.0	NC-Washburn-1X	146.25
01010878	Cuttings	630.0	635.0	NC-Washburn-1X	159.59
01010879	Cuttings	640.0	645.0	NC-Washburn-1X	158.41
01010880	Cuttings	650.0	655.0	NC-Washburn-1X	125.51

Please Sign this form and return to:
Sample Management Facility
Yucca Mountain Site Characterization Project
P.O. Box 617
Mercury, NV 89023-0617

I hereby acknowledge the receipt of the Specimens listed above. I will return this form to the SMF within 10 business days of receipt.

Recipient _____ Date _____

Page 6 of 6: 2 Containers shipped.

SMF Specimen Custody Receipt

CSITS v.1

Copy

Requestor:	Ship To:	
Paul Bertetti CNWRA/SWRI 6220 Culebra Road Building 57 San Antonio, TX 78238-5166	Paul Bertetti CNWRA/SWRI 6220 Culebra Road Building 57 San Antonio, TX 78238-5166	Date Received: 17 Dec 99 Shipment ID: 01000354 Shipping Date: 16-dec-1999 SMF Geotechnician Suzanne McNeil Date 12-15-99

Container ID: 01002648

Specimens in this container: 44

(9) Sample Wt. RC 3/17/00

Specimen ID	Type	Top	Bottom	Parent Borehole:
01010548	Cuttings	5.0	10.0	NC-EWDP-02D 133.07
01010549	Cuttings	15.0	20.0	NC-EWDP-02D 121.77
01010550	Cuttings	25.0	30.0	NC-EWDP-02D 142.10
01010551	Cuttings	35.0	40.0	NC-EWDP-02D 140.76
01010552	Cuttings	45.0	50.0	NC-EWDP-02D 144.81
01010553	Cuttings	55.0	60.0	NC-EWDP-02D 131.02
01010554	Cuttings	65.0	70.0	NC-EWDP-02D 139.74
01010555	Cuttings	75.0	80.0	NC-EWDP-02D 127.11
01010556	Cuttings	85.0	90.0	NC-EWDP-02D 147.93
01010558	Cuttings	95.0	100.0	NC-EWDP-02D 171.81
01010559	Cuttings	105.0	110.0	NC-EWDP-02D 124.87
01010560	Cuttings	115.0	120.0	NC-EWDP-02D 134.78
01010561	Cuttings	125.0	130.0	NC-EWDP-02D 126.52
01010562	Cuttings	135.0	140.0	NC-EWDP-02D 171.27
01010563	Cuttings	145.0	150.0	NC-EWDP-02D 132.17
01010564	Cuttings	155.0	160.0	NC-EWDP-02D 151.29
01010565	Cuttings	165.0	170.0	NC-EWDP-02D 156.91
01010566	Cuttings	175.0	180.0	NC-EWDP-02D 174.51
01010567	Cuttings	185.0	190.0	NC-EWDP-02D 136.69
01010568	Cuttings	195.0	200.0	NC-EWDP-02D 120.67
01010569	Cuttings	210.0	215.0	NC-EWDP-02D 122.78
01010570	Cuttings	220.0	225.0	NC-EWDP-02D 117.63
01010571	Cuttings	230.0	235.0	NC-EWDP-02D 73.26
01010572	Cuttings	240.0	245.0	NC-EWDP-02D 98.77
01010573	Cuttings	255.0	260.0	NC-EWDP-02D 81.14

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Yucca Mountain Site Characterization Project
P.O. Box 617
Mercury, NV 89023-0617

I hereby acknowledge the receipt of the Specimens listed above. I will return this form to the SMF within 10 business days of receipt.

Recipient Paul Bertetti Date 17 Dec 99

Page 1 of 2: 1 Containers shipped.

9/15/00 RC

Date 12-15-99

Specimens in this container: 44

(9) RC 3/17/00 Sample Wt.

Specimen ID	Type	Top	Bottom	Parent Borehole:
01010574	Cuttings	270.0	275.0	NC-EWDP-02D 117.05
01010575	Cuttings	280.0	285.0	NC-EWDP-02D 121.03
01010576	Cuttings	290.0	295.0	NC-EWDP-02D 99.38
01010577	Cuttings	300.0	305.0	NC-EWDP-02D 86.49
01010578	Cuttings	330.0	335.0	NC-EWDP-02D 91.87
01010579	Cuttings	345.0	350.0	NC-EWDP-02D 100.24
01010580	Cuttings	355.0	360.0	NC-EWDP-02D 133.81
01010581	Cuttings	365.0	370.0	NC-EWDP-02D 113.78
01010582	Cuttings	375.0	380.0	NC-EWDP-02D 53.99
01010583	Cuttings	385.0	390.0	NC-EWDP-02D 105.39
01010584	Cuttings	395.0	400.0	NC-EWDP-02D 114.98
01010585	Cuttings	405.0	410.0	NC-EWDP-02D 152.35
01010586	Cuttings	415.0	420.0	NC-EWDP-02D 122.20
01010587	Cuttings	430.0	435.0	NC-EWDP-02D 129.82
01010589	Cuttings	450.0	455.0	NC-EWDP-02D 108.32
01010590	Cuttings	460.0	465.0	NC-EWDP-02D 136.94
01010591	Cuttings	470.0	475.0	NC-EWDP-02D 115.75
01010592	Cuttings	480.0	485.0	NC-EWDP-02D 106.80
01010593	Cuttings	490.0	495.0	NC-EWDP-02D 57.51

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Recipient Paul Bertetti Date 17 Dec 99

Page 2 of 2: 1 Containers shipped.

9/15/00 PAB

SMF Specimen Custody Receipt

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Requestor:	Ship To:	
Paul Bertetti **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	**Paul Bertetti** **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	Date Received: _____ Shipment ID: 01000355 Shipping Date: 20-dec-1999 SMF Geotechnician Gina M Nelson Date 12/16/99

Container ID: 01002649 Specimens in this container: 50

Specimen ID	Type	Top	Bottom	Parent Borehole:	
01010595	Cuttings	500.0	505.0	NC-EWDP-02D	116.19
01010596	Cuttings	510.0	515.0	NC-EWDP-02D	110.58
01010597	Cuttings	520.0	525.0	NC-EWDP-02D	93.62
01010598	Cuttings	530.0	535.0	NC-EWDP-02D	113.21
01010599	Cuttings	540.0	545.0	NC-EWDP-02D	124.92
01010600	Cuttings	550.0	555.0	NC-EWDP-02D	123.23
01010601	Cuttings	560.0	565.0	NC-EWDP-02D	101.25
01010602	Cuttings	570.0	575.0	NC-EWDP-02D	106.83
01010603	Cuttings	580.0	585.0	NC-EWDP-02D	124.13
01010604	Cuttings	590.0	595.0	NC-EWDP-02D	124.71
01010605	Cuttings	600.0	605.0	NC-EWDP-02D	84.16
01010606	Cuttings	610.0	615.0	NC-EWDP-02D	99.82
01010607	Cuttings	620.0	625.0	NC-EWDP-02D	118.89
01010608	Cuttings	630.0	635.0	NC-EWDP-02D	115.74
01010609	Cuttings	640.0	645.0	NC-EWDP-02D	92.99
01010610	Cuttings	650.0	655.0	NC-EWDP-02D	122.63
01010611	Cuttings	660.0	665.0	NC-EWDP-02D	121.62
01010612	Cuttings	670.0	675.0	NC-EWDP-02D	121.34
01010613	Cuttings	680.0	685.0	NC-EWDP-02D	109.15
01010614	Cuttings	690.0	695.0	NC-EWDP-02D	112.83
01010615	Cuttings	700.0	705.0	NC-EWDP-02D	101.01
01010616	Cuttings	710.0	715.0	NC-EWDP-02D	90.24
01010617	Cuttings	720.0	725.0	NC-EWDP-02D	88.61
01010618	Cuttings	730.0	735.0	NC-EWDP-02D	97.84
01010619	Cuttings	740.0	745.0	NC-EWDP-02D	91.26

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Page 1 of 6: 2 Containers shipped.

9/15/00 PAB

SMF Specimen Custody Receipt

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Requestor:	Ship To:	
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Container ID: 01002649 Specimens in this container: 50

Specimen ID	Type	Top	Bottom	Parent Borehole:	
01010620	Cuttings	750.0	755.0	NC-EWDP-02D	99.88
01010621	Cuttings	760.0	765.0	NC-EWDP-02D	90.77
01010622	Cuttings	770.0	775.0	NC-EWDP-02D	92.68
01010623	Cuttings	780.0	785.0	NC-EWDP-02D	105.50
01010624	Cuttings	795.0	800.0	NC-EWDP-02D	40.07
01010625	Cuttings	805.0	810.0	NC-EWDP-02D	98.59
01010626	Cuttings	815.0	820.0	NC-EWDP-02D	84.67
01010627	Cuttings	825.0	830.0	NC-EWDP-02D	101.08
01010628	Cuttings	835.0	840.0	NC-EWDP-02D	105.28
01010629	Cuttings	845.0	850.0	NC-EWDP-02D	97.05
01010630	Cuttings	855.0	860.0	NC-EWDP-02D	116.52
01010631	Cuttings	865.0	870.0	NC-EWDP-02D	118.09
01010632	Cuttings	875.0	880.0	NC-EWDP-02D	126.99
01010633	Cuttings	885.0	890.0	NC-EWDP-02D	117.48
01010634	Cuttings	895.0	900.0	NC-EWDP-02D	117.59
01010635	Cuttings	905.0	910.0	NC-EWDP-02D	124.52
01010636	Cuttings	915.0	920.0	NC-EWDP-02D	110.88
01010637	Cuttings	925.0	930.0	NC-EWDP-02D	116.72
01010638	Cuttings	935.0	940.0	NC-EWDP-02D	114.40
01010639	Cuttings	945.0	950.0	NC-EWDP-02D	114.39
01010640	Cuttings	955.0	960.0	NC-EWDP-02D	117.25
01010641	Cuttings	965.0	970.0	NC-EWDP-02D	106.44
01010642	Cuttings	975.0	980.0	NC-EWDP-02D	117.25
01010643	Cuttings	985.0	990.0	NC-EWDP-02D	108.18
01010644	Cuttings	995.0	1000.0	NC-EWDP-02D	106.98

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Recipient _____ Date _____

Page 2 of 6: 2 Containers shipped.

9/15/00 RP

SMF Specimen Custody Receipt

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Paul Bertetti **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	**Paul Bertetti** **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	Date Received: _____ Shipment ID: 01000355 Shipping Date: 20-dec-1999 SMF Geotechnician Jenne McNelson Date 12/16/99

Container ID: 01002650

Specimens in this container: 62

Specimen ID	Type	Top	Bottom	Parent Borehole:
01010645	Cuttings	1005.0	1010.0	NC-EWDP-02D 93.67
01010646	Cuttings	1015.0	1020.0	NC-EWDP-02D 112.72
01010647	Cuttings	1025.0	1030.0	NC-EWDP-02D 115.30
01010648	Cuttings	1035.0	1040.0	NC-EWDP-02D 125.42
01010649	Cuttings	1045.0	1050.0	NC-EWDP-02D 107.79
01010650	Cuttings	1055.0	1060.0	NC-EWDP-02D 104.17
01010651	Cuttings	1065.0	1070.0	NC-EWDP-02D 102.31
01010652	Cuttings	1075.0	1080.0	NC-EWDP-02D 126.71
01010653	Cuttings	1085.0	1090.0	NC-EWDP-02D 107.54
01010654	Cuttings	1095.0	1100.0	NC-EWDP-02D 107.78
01010655	Cuttings	1105.0	1110.0	NC-EWDP-02D 106.44
01010656	Cuttings	1115.0	1120.0	NC-EWDP-02D 101.56
01010657	Cuttings	1125.0	1130.0	NC-EWDP-02D 84.86
01010658	Cuttings	1135.0	1140.0	NC-EWDP-02D 98.88
01010659	Cuttings	1145.0	1150.0	NC-EWDP-02D 79.63
01010660	Cuttings	1155.0	1160.0	NC-EWDP-02D 117.35
01010661	Cuttings	1165.0	1170.0	NC-EWDP-02D 105.96
01010662	Cuttings	1175.0	1180.0	NC-EWDP-02D 95.15
01010663	Cuttings	1185.0	1190.0	NC-EWDP-02D 106.04
01010664	Cuttings	1195.0	1200.0	NC-EWDP-02D 112.09
01010665	Cuttings	1205.0	1210.0	NC-EWDP-02D 121.07
01010666	Cuttings	1215.0	1220.0	NC-EWDP-02D 116.96
01010667	Cuttings	1225.0	1230.0	NC-EWDP-02D 113.05
01010668	Cuttings	1235.0	1240.0	NC-EWDP-02D 111.98
01010669	Cuttings	1245.0	1250.0	NC-EWDP-02D 101.42

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Page 4 of 6: 2 Containers shipped.

9/15/00 RP

SMF Specimen Custody Receipt

CSITS v.1

Requestor:	Ship To:	
Paul Bertetti **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	**Paul Bertetti** **CNWRA/SWRI** **6220 Culebra Road** **Building 57** **San Antonio, TX 78238-5166**	Date Received: _____ Shipment ID: 01000355 Shipping Date: 20-dec-1999 SMF Geotechnician Jenne McNelson Date 12/16/99

Container ID: 01002650

Specimens in this container: 62

Specimen ID	Type	Top	Bottom	Parent Borehole:
01010670	Cuttings	1255.0	1260.0	NC-EWDP-02D 108.74
01010671	Cuttings	1265.0	1270.0	NC-EWDP-02D 115.80
01010672	Cuttings	1275.0	1280.0	NC-EWDP-02D 117.28
01010673	Cuttings	1285.0	1290.0	NC-EWDP-02D 108.48
01010674	Cuttings	1295.0	1300.0	NC-EWDP-02D 115.02
01010675	Cuttings	1305.0	1310.0	NC-EWDP-02D 122.68
01010676	Cuttings	1315.0	1320.0	NC-EWDP-02D 119.75
01010678	Cuttings	1325.0	1330.0	NC-EWDP-02D 109.90
01010679	Cuttings	1335.0	1340.0	NC-EWDP-02D 115.60
01010680	Cuttings	1345.0	1350.0	NC-EWDP-02D 112.44
01010681	Cuttings	1355.0	1360.0	NC-EWDP-02D 105.30
01010682	Cuttings	1365.0	1370.0	NC-EWDP-02D 108.68
01010683	Cuttings	1375.0	1380.0	NC-EWDP-02D 123.79
01010684	Cuttings	1385.0	1390.0	NC-EWDP-02D 106.60
01010685	Cuttings	1395.0	1400.0	NC-EWDP-02D 100.94
01010686	Cuttings	1405.0	1410.0	NC-EWDP-02D 90.91
01010687	Cuttings	1415.0	1420.0	NC-EWDP-02D 109.99
01010688	Cuttings	1425.0	1430.0	NC-EWDP-02D 99.90
01010689	Cuttings	1435.0	1440.0	NC-EWDP-02D 122.24
01010690	Cuttings	1445.0	1450.0	NC-EWDP-02D 155.09
01010691	Cuttings	1455.0	1460.0	NC-EWDP-02D 128.63
01010692	Cuttings	1465.0	1470.0	NC-EWDP-02D 103.41
01010693	Cuttings	1475.0	1480.0	NC-EWDP-02D 121.58
01010694	Cuttings	1485.0	1490.0	NC-EWDP-02D 94.19
01010695	Cuttings	1495.0	1500.0	NC-EWDP-02D 100.45

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Page 5 of 6: 2 Containers shipped.

9/15/00
PB

SMF Specimen Custody Receipt

CSITS v.1

Date Received: _____

Shipment ID: 01000355 Shipping Date: 20-dec-1999

SMF Geotechnician Thomas M Yates Date 12/16/99

166

Requestor:

Paul Bertetti
CNWRA/SWRI
6220 Culebra Road
Building 57
San Antonio, TX 78238-5166

Ship To:

Paul Bertetti
CNWRA/SWRI
6220 Culebra Road
Building 57
San Antonio, TX 78238-5166

Container ID: 01002650

Specimens in this container: 62

Top	Bottom	Parent Borehole:	
1505.0	1510.0	NC-EWDP-02D	1122.67
1515.0	1520.0	NC-EWDP-02D	1117.70
1525.0	1530.0	NC-EWDP-02D	1107.39
1535.0	1540.0	NC-EWDP-02D	1097.02
1545.0	1550.0	NC-EWDP-02D	1086.20
1555.0	1560.0	NC-EWDP-02D	1076.46
1565.0	1570.0	NC-EWDP-02D	1066.92
1575.0	1580.0	NC-EWDP-02D	1056.35
1585.0	1590.0	NC-EWDP-02D	1046.75
1595.0	1600.0	NC-EWDP-02D	1036.36
1605.0	1610.0	NC-EWDP-02D	1024.12
1615.0	1618.4	NC-EWDP-02D	1013.95

Specimen ID	Type
01010696	Cuttings
01010697	Cuttings
01010698	Cuttings
01010699	Cuttings
01010700	Cuttings
01010701	Cuttings
01010702	Cuttings
01010703	Cuttings
01010704	Cuttings
01010705	Cuttings
01010706	Cuttings
01010707	Cuttings

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Page 6 of 6: 2 Containers shipped.

9/15/00
PB

Samples from NC-EWDP-02D, summary

	Specimen I.D.	Top Depth (ft)	Bottom Depth (ft)	Sample Wt. (grams)	① XRD+Chem	② Powder sub	Powder sub	③ Thin sub	④ Thin (stain)
Container ID 01002648									
	1010548	5.0	10.0	133.07					
	1010549	15.0	20.0	121.77	x				
	1010550	25.0	30.0	142.10	x				
	1010551	35.0	40.0	140.76					
	1010552	45.0	50.0	144.81					
	1010553	55.0	60.0	131.02					
	1010554	65.0	70.0	139.74					
	1010555	75.0	80.0	127.11					
	1010556	85.0	90.0	147.93					
	1010558	95.0	100.0	171.81					
	1010559	105.0	110.0	124.87					
	1010560	115.0	120.0	134.78					
	1010561	125.0	130.0	136.62					
	1010562	135.0	140.0	171.27					
	1010563	145.0	150.0	132.17					
	1010564	155.0	160.0	151.29					
	1010565	165.0	170.0	156.91					
	1010566	175.0	180.0	174.51	x	A			
	1010567	185.0	190.0	136.69	x				
	1010568	195.0	200.0	120.67					
	1010569	210.0	215.0	122.78					
	1010570	220.0	225.0	117.63	x				
	1010571	230.0	235.0	73.26	x	A			
	1010572	240.0	245.0	98.77					
	1010573	255.0	260.0	81.14					
	1010574	270.0	275.0	117.05					
	1010575	280.0	285.0	121.03					
	1010576	290.0	295.0	99.38					
	1010577	300.0	305.0	86.49					
	1010578	330.0	335.0	91.87	x				Ts
	1010579	345.0	350.0	100.24	x	A			
	1010580	355.0	360.0	133.81					
	1010581	365.0	370.0	113.78					
	1010582	375.0	380.0	53.99					
	1010583	385.0	390.0	105.39	x				
	1010584	395.0	400.0	114.98	x	A			T
	1010585	405.0	410.0	152.35					
	1010586	415.0	420.0	122.20					
	1010587	430.0	435.0	129.82					
	1010589	450.0	455.0	108.32					
	1010590	460.0	465.0	136.94					
	1010591	470.0	475.0	115.75					
	1010592	480.0	485.0	106.80					
	1010593	490.0	495.0	57.51					
Container ID 01002649									
	1010595	500.0	505.0	116.19					
	1010596	510.0	515.0	110.58					

Notes:

① x means cuttings sample selected for chem or xrd analysis and powdered

② subsample of cuttings - mineral or grain of rock, e.g. feldspar / rhyolite pebble, also powdered. → labeled A or B subsample

③ Thin section subsample

④ cuttings selected for thin section preparation.

9/15/00 PB

NC-EWDP-02D Summary - see notes p. 111

	1010597	520.0	525.0	93.62							
	1010598	530.0	535.0	113.21							
	1010599	540.0	545.0	124.92							
	1010600	550.0	555.0	123.23							
	1010601	560.0	565.0	101.25							
	1010602	570.0	575.0	106.83	x						
	1010603	580.0	585.0	124.13	x						
	1010604	590.0	595.0	124.71							
	1010605	600.0	605.0	84.16							
	1010606	610.0	615.0	99.82							
	1010607	620.0	625.0	116.89							
	1010608	630.0	635.0	115.74							
	1010609	640.0	645.0	92.99							
	1010610	650.0	655.0	122.63							
	1010611	660.0	665.0	121.62							
	1010612	670.0	675.0	121.34							
	1010613	680.0	685.0	109.15							
	1010614	690.0	695.0	112.83							
	1010615	700.0	705.0	131.01							
	1010616	710.0	715.0	90.24							
	1010617	720.0	725.0	88.61							
	1010618	730.0	735.0	97.84							
	1010619	740.0	745.0	91.24							
	1010620	750.0	755.0	99.88							
	1010621	760.0	765.0	90.77							
	1010622	770.0	775.0	92.68							
	1010623	780.0	785.0	105.50							
	1010624	795.0	800.0	40.07							
	1010625	805.0	810.0	98.59							
	1010626	815.0	820.0	84.67							
	1010627	825.0	830.0	101.08							
	1010628	835.0	840.0	105.28							
	1010629	845.0	850.0	97.05							
	1010630	855.0	860.0	116.52							
	1010631	865.0	870.0	118.04							
	1010632	875.0	880.0	126.99							
	1010633	885.0	890.0	117.48							
	1010634	895.0	900.0	117.59							
	1010635	905.0	910.0	134.52							
	1010636	915.0	920.0	110.88							
	1010637	925.0	930.0	116.72	x						
	1010638	935.0	940.0	114.40	x						
	1010639	945.0	950.0	114.39							
	1010640	955.0	960.0	117.25							
	1010641	965.0	970.0	106.44							
	1010642	975.0	980.0	117.25							
	1010643	985.0	990.0	108.18							
	1010644	995.0	1000.0	106.98							
Container ID 01002650											
	1010645	1005.0	1010.0	93.67							
	1010646	1015.0	1020.0	112.72							
	1010647	1025.0	1030.0	115.30							
	1010648	1035.0	1040.0	125.42	x	A	B				
	1010649	1045.0	1050.0	107.79	x						Ts
	1010650	1055.0	1060.0	104.17	x						T
	1010651	1065.0	1070.0	102.31							
	1010652	1075.0	1080.0	126.71							

9/15/00 PB

NC-EWDP-02D Summary - see notes p. 111

	1010653	1085.0	1090.0	107.54	x						
	1010654	1095.0	1100.0	107.78	x	A		AT			
	1010655	1105.0	1110.0	106.44	x						
	1010656	1115.0	1120.0	101.56	x	A	B	AT	BT		
	1010657	1125.0	1130.0	84.86	x						Ts
	1010658	1135.0	1140.0	98.88							
	1010659	1145.0	1150.0	79.63							
	1010660	1155.0	1160.0	117.35							
	1010661	1165.0	1170.0	105.96	x						Ts
	1010662	1175.0	1180.0	95.15							
	1010663	1185.0	1190.0	100.04	x						
	1010664	1195.0	1200.0	112.09	x						Ts
	1010665	1205.0	1210.0	121.07	x						Ts
	1010666	1215.0	1220.0	116.96	x						Ts
	1010667	1225.0	1230.0	113.06	x						
	1010668	1235.0	1240.0	111.98	x						
	1010669	1245.0	1250.0	101.42	x						
	1010670	1255.0	1260.0	108.74	x						
	1010671	1265.0	1270.0	115.80							
	1010672	1275.0	1280.0	117.20							
	1010673	1285.0	1290.0	108.48							
	1010674	1295.0	1300.0	115.02							
	1010675	1305.0	1310.0	122.68							
	1010676	1315.0	1320.0	119.75							
	1010678	1325.0	1330.0	109.90							
	1010679	1335.0	1340.0	115.60							
	1010680	1345.0	1350.0	112.44							
	1010681	1355.0	1360.0	105.30							
	1010682	1365.0	1370.0	108.68							
	1010683	1375.0	1380.0	123.79							
	1010684	1385.0	1390.0	106.60	x						T
	1010685	1395.0	1400.0	109.94	x						
	1010686	1405.0	1410.0	90.91							
	1010687	1415.0	1420.0	109.99							
	1010688	1425.0	1430.0	99.90							
	1010689	1435.0	1440.0	122.24							
	1010690	1445.0	1450.0	155.09	x	A		BT			Ts
	1010691	1455.0	1460.0	128.63							
	1010692	1465.0	1470.0	103.41							
	1010693	1475.0	1480.0	121.58							
	1010694	1485.0	1490.0	94.19	x						
	1010695	1495.0	1500.0	100.45							
	1010696	1505.0	1510.0	122.67							
	1010697	1515.0	1520.0	117.70							
	1010698	1525.0	1530.0	107.39							
	1010699	1535.0	1540.0	129.02							
	1010700	1545.0	1550.0	122.20							
	1010701	1555.0	1560.0	100.46	x						T
	1010702	1565.0	1570.0	142.92	x						
	1010703	1575.0	1580.0	116.31	x						
	1010704	1585.0	1590.0	123.75							
	1010705	1595.0	1600.0	140.36							
	1010706	1605.0	1610.0	114.12							
	1010707	1615.0	1620.0	133.95							
	Total Samples	156			38	8	2	3	1	12	

9/15/00 PB

Nc- Wash burn - 1x summary. See notes p. 111

Specimen I.D.	Top Depth (ft.)	Bottom Depth (ft.)	Sample Wt. (g)	XRD+Chem	Powder sub	Thin (stain)
1010819	5.0	10.0	130.99			
1010820	15.0	20.0	137.23			
1010821	25.0	30.0	155.23			
1010822	35.0	40.0	149.54			
1010823	45.0	50.0	159.48			
1010824	55.0	60.0	160.90			
1010825	65.0	70.0	154.37			
1010826	75.0	80.0	136.41			
1010827	85.0	90.0	143.24			
1010828	95.0	100.0	183.27			
1010829	105.0	110.0	146.48			
1010830	115.0	120.0	157.14			
1010831	125.0	130.0	146.88			
1010832	135.0	140.0	151.93			
1010833	145.0	150.0	137.73			
1010834	155.0	160.0	184.56			
1010835	165.0	170.0	147.51			
1010836	175.0	180.0	144.45			
1010837	185.0	190.0	155.39			
1010838	195.0	200.0	n/a			
1010839	205.0	210.0	161.15			
1010840	215.0	220.0	184.54			
1010841	225.0	230.0	219.53	x	A	
1010842	235.0	240.0	162.06	x		
1010843	245.0	250.0	179.10			
1010844	255.0	260.0	140.28			
1010845	265.0	270.0	173.88			
1010846	275.0	280.0	151.35			
1010847	285.0	290.0	154.47			
1010848	295.0	300.0	118.23			
1010849	305.0	310.0	151.99			
1010850	315.0	320.0	131.62			
1010851	325.0	330.0	142.80			
1010852	335.0	340.0	114.97	x		
1010853	345.0	350.0	142.16	x		
1010854	355.0	360.0	132.25	x		
1010855	365.0	370.0	127.55	x		T
1010856	375.0	380.0	126.66	x		Ts
1010857	385.0	390.0	174.62			
1010858	395.0	400.0	132.72			
1010859	410.0	415.0	148.71			
1010860	420.0	425.0	155.91			
1010861	455.0	460.0	152.53			
1010862	465.0	470.0	120.06			
1010863	475.0	480.0	159.91			
1010864	485.0	490.0	129.12	x		Ts
1010865	495.0	500.0	167.92	x		
1010866	505.0	510.0	143.87	x		
1010867	515.0	520.0	127.22	x		Ts
1010868	525.0	530.0	134.30	x		T
1010869	535.0	540.0	131.38	x		T
1010870	545.0	550.0	116.14	x		
1010871	555.0	560.0	131.74	x		
1010872	570.0	575.0	149.44	x		
1010873	580.0	585.0	170.40	x		
1010874	590.0	595.0	133.72			
1010875	600.0	605.0	163.99			
1010876	610.0	615.0	161.21	x		
1010877	620.0	625.0	146.25	x		
1010878	630.0	635.0	159.59	x		
1010879	640.0	645.0	158.41			
1010880	650.0	655.0	125.51			
Total Samples		62		20	1	6

9/15/00 PB

Thin section summary - see order form p. 100

Specimen I.D.						
500-x		T				W510
501-x						
502						
503						
504						
505						
550-x						
566-x						
566-ax						
567-x						
570-x						
571-x						
571-ax						
578-x		T	S			
579-x						
579-ax						
583-x						
584-x		T				
584-ax						
602-x						
603-x						
637-x						
638-x						
648-ax						
648-bx						
648-b						
649-x		T	S			
650-x		T				
653-x						
654-x						
654-ax		T				
655-x						
656-x						
656-ax		T				
656-bx		T				
657-x		T	S			
661-x		T	S			
663-x						
664-x		T	S			
665-x		T	S			
666-x		T	S			
667-x						
668-x						
669-x						
670-x						
684-x		T				
685-x						
690-x		T	S			
690-ax		T				
694-x						
701-x		T				

702-x						
703-x						
841-x						
841-ax						
842-x						
852-x						
853-x						
854-x						
855-x		T				
856-x		T	S			
864-x		T	S			
865-x						
866-x						
867-x		T	S			
868-x		T				
869-x		T				
870-x						
871-x						
872-x						
873-x						
876-x						
877-x						
878-x						
900-x						
75		23	11			nbs 70a

9/15/00
PB

Standards, check standards summary.

Other specimens				
Specimen I.D.	Description		XRD+Chem	Thin (stain)
500	W510 60/100*UC*Rc*RFe*HL		x	T
501	CDV #25535		x	
502	NBS 70a - Kspar		x	
503	NBS 99a - Na spar		x	
504	NBS 278 - obsidian		x	
505	RGM1 - USGS rhyolite		x	
900	NBS 70a - Kspar		x	

9/20/00
PB

received results from XRD analyses of well cuttings and preliminary results (XRF only) for chemical analyses of well cuttings.

WSU has provided procedures for their XRF and ICP-MS analyses. They are posted here for reference.

9/20/00 PB

icp_ms / wsu

TABLE 3

Information potentially subject to copyright protection was redacted from pages 124 through 130 of this scientific notebook. The redacted material is from the following reference:

Knaack, C. S. Cornelius, and P. Hooper. "Trace Element Analyses of Rocks and Minerals by ICP-MS." Washington State University Pullman, Washington: WSU GeoAnalytical Laboratory. December 1994.

lcp-ms/wsu (contd)

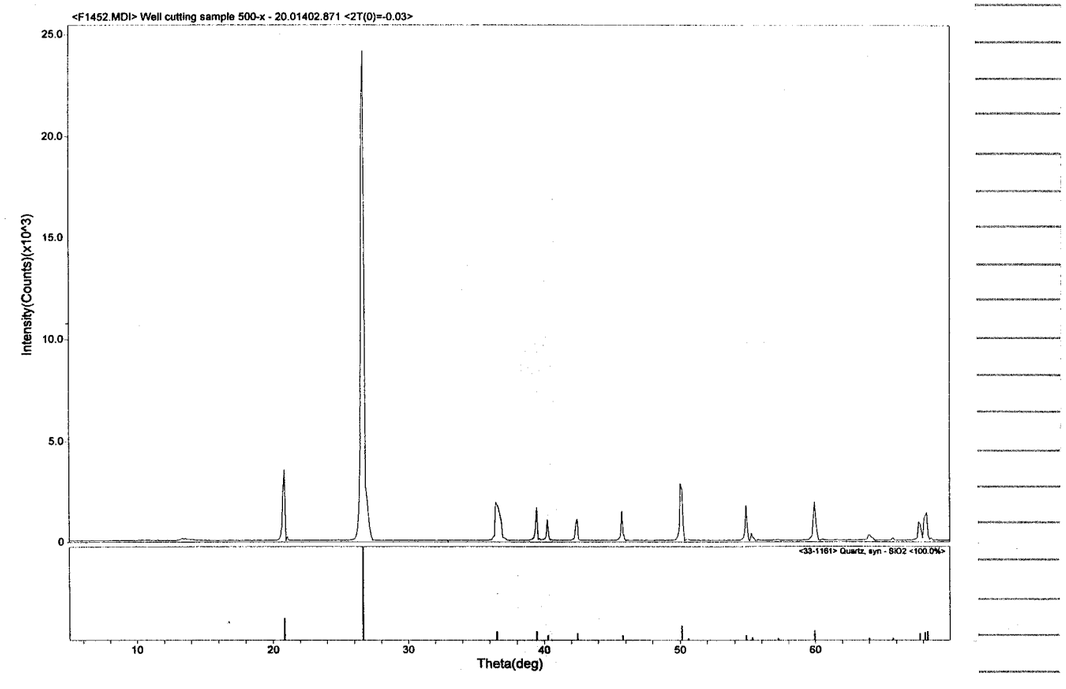
9/20/00
PD

9/20/00
PD

XRD data files are stored on a floppy diskette labeled
20.01402.871
well cutting
samples
- xrd data -

The diskette is stored in an envelope fixed to the back interior cover of this notebook

An inspection of the XRD patterns for samples 500, 501 and 900 indicates that the patterns are as expected for the samples provided. The pattern printouts are shown below and on the next page. Since the patterns are as expected, it is assumed that the XRD patterns for the cuttings are / have been collected appropriately.

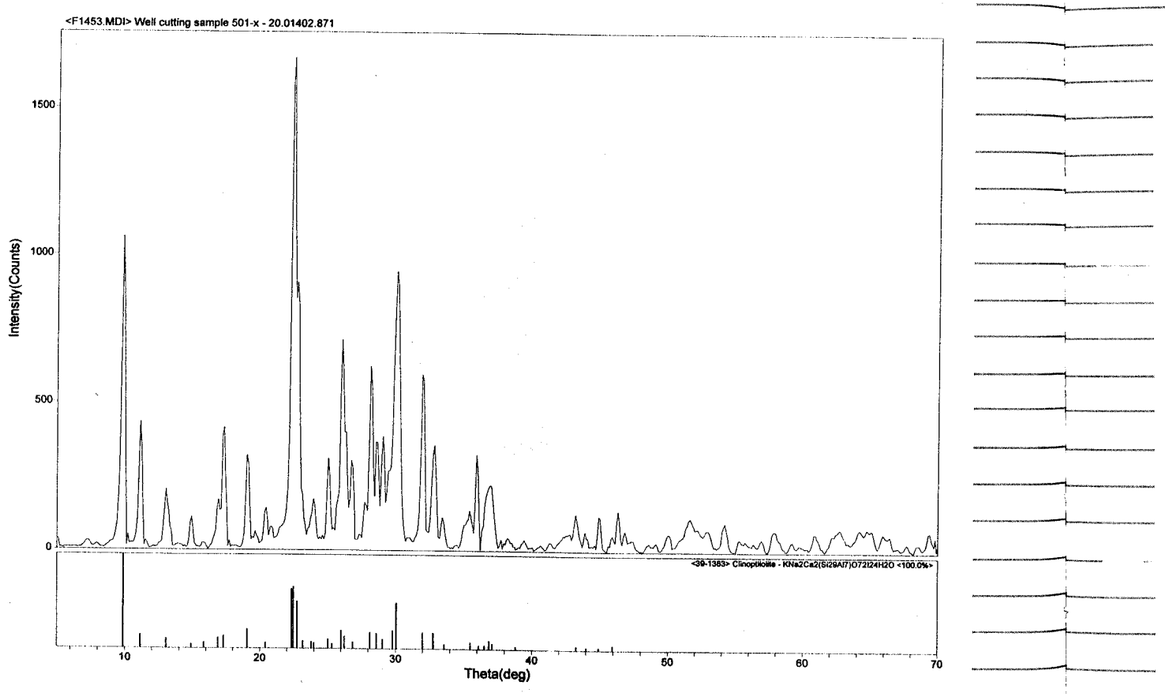


9/20/00
PB

note that the quartz and clinoptilolite patterns are readily identified by the search/id software used with the XRD.

#500 - quartz - powdered
#501 - clinoptilolite

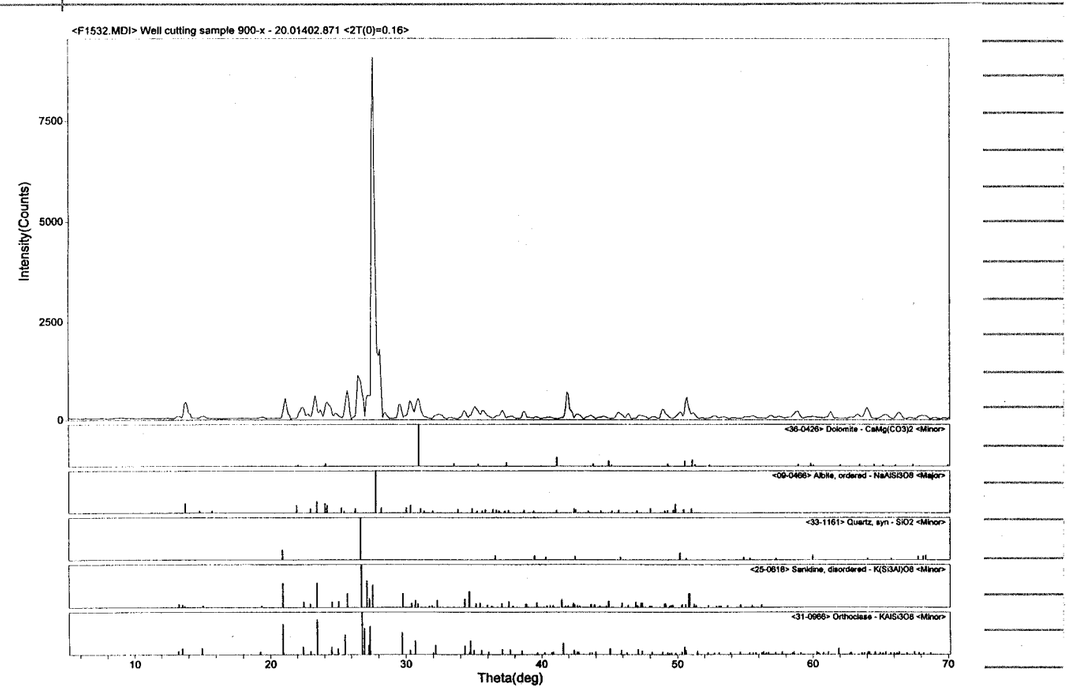
9/20/00
PB



9/20/00
PB

note the NBS 70a (potassium feldspar) pattern has many k-spar peaks (orthoclase/sanidine) but also has a significant albite peak. A review of the reference certificate shows that the material is perthitic with about 10-15% albite. The combined pattern is indicative of the complexities inherent in mixed feldspars and should be an example of the heterogeneity expected in the cuttings patterns.

9/20/00
PB



9/20/00 PS

WSU XRF and LOI results are listed on the following pages. 8 samples did not have enough powder to also be included in subsequent icp-ms analyses to be completed much later this year. The 8 were not specifically identified. All 74 have been analyzed using XRF and had LOI as well. Sample results are provided on the following pages. I have also included results of WSU analyses for its own internal standards (BCRP and GSP1$). Results of included standards (as blinds) are consistent with expectations. All analyses appear to be acceptable. Further interpretation and work will be conducted next calendar year.

9/21/00 PS

Late Entry: on 8/9/00, prior to sending powders to be analyzed, I collected a subset of powders for additional wet chemistry, if that was desired. The samples, weight of vials, and weight of powders collected are given below

→ Mettler AE 240

9/20/00 PS

Sample no.	wt vial empty (g)	wt vial + powder (g)	wt powder (g)
650	1.4311	2.4623	1.0312
602	1.4284	2.5449	1.1165
656	1.4045	2.4299	1.0254
663	1.4123	~~2.45 15~~ after 30 2.4514	1.0391
664	1.4031	2.5443	1.1412
668	1.4108	2.4848	1.0740
702	1.4138	2.5831	1.1693
853	1.4057	2.4550	1.0493
855	1.4016	2.4218	1.0202
865	1.4280	2.4764	1.0484
870	1.3946	2.7511	1.3565
876	1.4123	2.5453	1.1330

Analyses by XRF

WSU GeoAnalytical Laboratory 1

BCRP0700.xls

Date	BCRP 487 29-Aug-00 0700	BCRP 487 1-Sep-00 0700	BCRP 487 4-Sep-00 0700	BCRP 487 8-Sep-00 0700	BCRP 487 11-Sep-00 0700	BCRP 487 14-Sep-00 0700	BCRP 487 20-Sep-00 0700	BCRP 487 7 0700 stdev	BCRP 487 9-May-00 0300 average n=16	BCR1 accepted Gov, 94
Unnormalized Results (Weight %):										
SiO2	55.08	55.08	55.10	55.03	55.05	54.94	54.95	0.06	55.17	54.11
Al2O3	13.66	13.64	13.64	13.64	13.63	13.63	13.61	0.02	13.70	13.64
TiO2	2.290	2.297	2.294	2.299	2.291	2.299	2.301	0.004	2.298	2.240
FeO	12.48	12.45	12.51	12.45	12.49	12.44	12.45	0.03	12.48	12.07
MnO	0.186	0.186	0.186	0.187	0.187	0.187	0.187	0.001	0.186	0.180
CaO	7.02	7.02	7.01	7.02	7.02	7.02	7.02	0.00	7.03	6.95
MgO	3.54	3.52	3.56	3.51	3.55	3.52	3.56	0.02	3.56	3.48
K2O	1.74	1.74	1.74	1.74	1.74	1.74	1.74	0.00	1.74	1.69
Na2O	3.37	3.39	3.36	3.35	3.35	3.33	3.31	0.03	3.32	3.27
P2O5	0.384	0.380	0.378	0.379	0.378	0.380	0.378	0.002	0.380	0.360
Total	99.75	99.70	99.78	99.61	99.68	99.48	99.51	0.12	99.87	97.99
									from same quarry as BCR1	
Normalized Results (Weight %):										
SiO2	55.22	55.24	55.22	55.25	55.22	55.23	55.22	0.01	55.24	55.22
Al2O3	13.69	13.68	13.67	13.69	13.67	13.70	13.68	0.01	13.72	13.92
TiO2	2.296	2.304	2.299	2.308	2.298	2.311	2.312	0.007	2.301	2.29
FeO*	12.51	12.49	12.54	12.50	12.53	12.50	12.52	0.02	12.50	12.32
MnO	0.186	0.187	0.186	0.188	0.188	0.188	0.188	0.001	0.186	0.18
CaO	7.04	7.04	7.03	7.05	7.04	7.06	7.05	0.01	7.03	7.09
MgO	3.55	3.53	3.57	3.52	3.56	3.54	3.58	0.02	3.57	3.55
K2O	1.74	1.74	1.74	1.75	1.75	1.75	1.75	0.00	1.74	1.72
Na2O	3.38	3.40	3.37	3.36	3.36	3.35	3.33	0.02	3.33	3.34
P2O5	0.385	0.381	0.379	0.380	0.379	0.382	0.380	0.002	0.380	0.37
Trace Elements (ppm):										
Ni	7	10	10	8	8	8	10	1	10	13
Cr	28	28	27	31	32	29	31	2	29	16
Sc	29	34	34	38	32	26	29	4	34	33
V	399	398	397	399	388	392	398	4	391	407
Ba	705	712	705	709	700	714	717	6	708	681
Rb	46	46	45	47	46	48	47	1	46	47
Sr	325	326	322	324	325	324	326	1	324	330
Zr	178	179	180	179	179	178	179	1	179	190
Y	37	37	38	37	38	37	37	0	37	38
Nb	12.7	12.5	11.9	11.6	13.0	12.9	12.3	0.5	12.6	14.0
Ga	24	25	21	20	22	22	22	2	23	22
Cu	18	21	24	20	22	22	18	2	19	19
Zn	127	124	127	127	128	124	125	2	136	130
Pb	10	12	8	10	11	10	11	1	11	14
La	27	25	21	32	29	30	17	5	20	25
Ce	53	54	63	56	65	56	51	5	53	54
Th	6	6	6	5	6	7	6	1	6	6

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"+" denotes values >120% of our highest standard.

GSP10700.xls

Date	GSP1$ 1187 29-Aug-00 0700	GSP1$ 1187 1-Sep-00 0700	GSP1$ 1187 4-Sep-00 0700	GSP1$ 1187 8-Sep-00 0700	GSP1$ 1187 11-Sep-00 0700	GSP1$ 1187 14-Sep-00 0700	GSP1$ 1187 20-Sep-00 0700	GSP1$ 1187 4 0700 stdev	GSP1$ 1187 9-May-00 0300 average n=16	GSP1 Gov, 94 accepted
Unnormalized Results (Weight %):										
SiO2	68.39	68.37	68.43	68.29	68.28	68.18	68.24	0.09	68.72	67.22
Al2O3	15.40	15.38	15.41	15.39	15.38	15.35	15.38	0.02	15.46	15.10
TiO2	0.672	0.670	0.668	0.673	0.669	0.672	0.671	0.002	0.670	0.650
FeO	3.88	3.87	3.89	3.88	3.89	3.88	3.88	0.01	3.88	3.86
MnO	0.039	0.039	0.038	0.038	0.039	0.038	0.038	0.001	0.039	0.040
CaO	2.04	2.04	2.03	2.03	2.04	2.04	2.03	0.01	2.03	2.07
MgO	1.10	1.11	1.12	1.08	1.11	1.10	1.09	0.01	1.11	0.96
K2O	5.58	5.58	5.58	5.57	5.58	5.58	5.58	0.00	5.58	5.51
Na2O	2.84	2.86	2.86	2.81	2.83	2.82	2.82	0.02	2.83	2.80
P2O5	0.287	0.286	0.287	0.288	0.288	0.285	0.286	0.001	0.288	0.280
Total	100.23	100.22	100.31	100.05	100.10	99.94	100.02	0.13	100.61	98.49
Normalized Results (Weight %):										
SiO2	68.23	68.22	68.22	68.26	68.21	68.22	68.23	0.02	68.31	68.25
Al2O3	15.36	15.35	15.36	15.38	15.36	15.36	15.38	0.01	15.36	15.33
TiO2	0.670	0.669	0.666	0.673	0.668	0.672	0.671	0.002	0.666	0.660
FeO*	3.87	3.86	3.88	3.87	3.88	3.88	3.88	0.01	3.86	3.92
MnO	0.039	0.039	0.038	0.038	0.039	0.038	0.038	0.001	0.038	0.041
CaO	2.04	2.04	2.02	2.03	2.04	2.04	2.03	0.01	2.02	2.10
MgO	1.10	1.11	1.12	1.08	1.11	1.10	1.09	0.01	1.10	0.97
K2O	5.57	5.57	5.56	5.57	5.57	5.58	5.58	0.01	5.55	5.59
Na2O	2.83	2.86	2.85	2.81	2.83	2.82	2.82	0.02	2.81	2.84
P2O5	0.286	0.285	0.286	0.288	0.288	0.285	0.286	0.001	0.286	0.284
Trace Elements (ppm):										
Ni	13	16	13	12	15	15	14	1	14	9
Cr	8	9	11	10	12	11	9	1	11	13
Sc	8	1	2	8	1	9	5	4	6	6
V	56	52	53	54	52	48	62	4	53	53
Ba	1303	1283	1293	1292	1318	1298	1293	11	1299	1310
Rb	256	253	254	253	256	255	255	1	254	254
Sr	233	232	233	231	232	232	232	1	232	234
Zr	540	540	541	541	541	538	541	1	540	530
Y	26	26	26	25	25	26	26	0	26	26
Nb	25.4	25.9	26.3	25.6	26.0	25.6	26.0	0.3	26.0	28.0
Ga	23	22	21	20	24	19	21	2	22	23
Cu	37	33	31	34	35	34	34	2	34	33
Zn	102	104	101	104	106	106	105	2	105	104
Pb	53	54	53	53	57	56	55	2	54	55
La	182	189	196	181	192	186	187	5	183	184
Ce	389	381	398	389	403	383	395	8	395	399
Th	106	105	106	105	105	109	106	1	105	106

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"+" denotes values >120% of our highest standard.

WSU GeoAnalytical Laboratory 1

Analyses by XRF

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 501	BER 502	BER 503	BER 504	BER 505	BER 549	BER 550	BER 566	BER 566A	BER 567
Date	20-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00
LOI (%)	13.70	0.44	0.24	0.70	1.07	3.87	5.17	4.40	1.68	4.61
	501	502	503	504	505	549	550	566	566A	567
Unnormalized Results (Weight %):										
SiO2	76.36	67.74	65.44	73.20	73.93	73.44	70.51	72.98	72.98	72.84
Al2O3	12.96	18.21	20.86	14.54	14.15	12.84	13.32	14.28	14.32	14.48
TiO2	0.116	0.000	0.001	0.238	0.267	0.278	0.391	0.357	0.297	0.387
FeO	0.85	0.09	0.08	1.91	1.77	1.77	2.13	2.46	1.62	2.42
MnO	0.039	0.000	0.000	0.051	0.036	0.067	0.068	0.085	0.145	0.089
CaO	1.75	0.15	2.12	1.02	1.21	2.90	4.86	1.47	1.22	1.56
MgO	0.44	0.11	0.04	0.28	0.34	0.65	1.00	0.91	0.48	1.00
K2O	4.17	11.85	5.35	4.19	4.40	4.65	4.32	4.26	4.88	4.12
Na2O	3.85	2.49	6.19	4.82	4.18	3.28	3.25	3.06	3.70	3.10
P2O5	0.021	0.005	0.018	0.040	0.046	0.047	0.083	0.066	0.149	0.066
Total	100.56	100.65	100.09	100.29	100.32	99.92	99.93	99.93	99.79	100.06
Normalized Results (Weight %):										
SiO2	75.93	67.30	65.38	72.99	73.69	73.50	70.56	73.03	73.14	72.80
Al2O3	12.89	18.09	20.84	14.50	14.10	12.85	13.33	14.29	14.35	14.47
TiO2	0.115	0.000	0.001	0.237	0.266	0.278	0.391	0.357	0.298	0.387
FeO*	0.85	0.09	0.07	1.91	1.76	1.77	2.13	2.46	1.62	2.42
MnO	0.039	0.000	0.000	0.051	0.036	0.067	0.068	0.085	0.145	0.089
CaO	1.74	0.15	2.12	1.02	1.21	2.90	4.86	1.47	1.22	1.56
MgO	0.44	0.11	0.04	0.28	0.34	0.65	1.00	0.91	0.48	1.00
K2O	4.15	†11.77	5.34	4.18	4.39	4.65	4.32	4.26	4.89	4.12
Na2O	3.83	2.47	6.18	4.81	4.17	3.28	3.25	3.06	3.71	3.10
P2O5	0.021	0.005	0.018	0.040	0.046	0.047	0.083	0.066	0.149	0.066
Trace Elements (ppm):										
Ni	12	15	9	28	8	12	14	23	25	13
Cr	3	6	0	9	1	3	5	13	5	8
Sc	7	1	4	5	3	3	4	4	8	7
V	2	0	1	9	11	14	28	24	18	39
Ba	418	124	†2534	900	826	472	672	610	687	614
Rb	142	†530	110	131	153	153	128	145	175	141
Sr	214	66	443	65	106	207	318	218	161	226
Zr	97	5	15	276	212	242	259	271	252	291
Y	18	1	1	41	23	30	29	32	40	34
Nb	16.6	1.1	0.2	16.3	8.3	23.6	22.0	25.5	24.2	25.1
Ga	14	21	16	17	15	17	17	19	16	19
Cu	2	7	7	6	12	3	7	6	7	4
Zn	29	1	5	48	32	56	58	70	54	72
Pb	29	46	65	17	22	23	20	26	26	28
La	31	7	0	28	25	61	73	76	99	69
Ce	65	15	0	62	49	110	93	136	164	139
Th	20	0	1	13	15	22	19	24	30	22

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"†" denotes values >120% of our highest standard.

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 570	BER 571	BER 571A	BER 578	BER 579	BER 579A	BER 583	BER 584	BER 584A	BER 602
Date	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	12-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00
LOI (%)	3.79	3.49	2.14	4.40	4.83	2.16	4.38	3.60	14.47	2.49
	570	571	571A	578	579	579A	583	584	584A	602
Unnormalized Results (Weight %):										
SiO2	74.31	73.49	73.73	73.72	72.76	76.21	73.34	74.53	80.57	75.38
Al2O3	13.94	13.65	13.84	13.83	14.36	13.31	14.03	13.71	13.72	12.70
TiO2	0.255	0.379	0.332	0.315	0.364	0.170	0.313	0.243	0.126	0.348
FeO	1.56	2.42	1.68	1.82	2.31	0.82	1.94	1.46	0.52	2.47
MnO	0.066	0.085	0.090	0.079	0.084	0.031	0.070	0.071	0.008	0.077
CaO	1.37	1.39	0.93	1.63	1.66	0.53	1.86	1.37	0.72	1.10
MgO	0.78	0.80	0.54	0.85	0.99	0.36	0.87	0.65	0.74	0.60
K2O	4.34	4.34	5.10	4.29	4.20	5.29	4.42	4.80	2.65	4.35
Na2O	3.19	3.28	3.59	3.17	3.06	3.09	3.13	3.28	0.48	2.92
P2O5	0.077	0.086	0.055	0.071	0.079	0.061	0.101	0.108	0.044	0.098
Total	99.89	99.92	99.89	99.77	99.87	99.87	100.08	100.22	99.57	100.05
Normalized Results (Weight %):										
SiO2	74.39	73.55	73.81	73.89	72.86	76.31	73.28	74.37	80.92	75.35
Al2O3	13.96	13.66	13.86	13.86	14.38	13.33	14.02	13.68	13.78	12.69
TiO2	0.255	0.379	0.332	0.316	0.364	0.170	0.313	0.242	0.127	0.348
FeO*	1.56	2.42	1.68	1.82	2.31	0.82	1.94	1.45	0.52	2.47
MnO	0.066	0.085	0.090	0.079	0.084	0.031	0.070	0.071	0.008	0.077
CaO	1.37	1.39	0.93	1.63	1.66	0.53	1.86	1.37	0.72	1.10
MgO	0.78	0.80	0.54	0.85	0.99	0.36	0.87	0.65	0.74	0.60
K2O	4.34	4.34	5.11	4.30	4.21	5.30	4.42	4.79	2.66	4.35
Na2O	3.19	3.28	3.59	3.18	3.06	3.09	3.13	3.27	0.48	2.92
P2O5	0.077	0.086	0.055	0.071	0.079	0.061	0.101	0.108	0.044	0.098
Trace Elements (ppm):										
Ni	16	22	21	14	26	16	45	37	16	19
Cr	4	9	4	4	7	0	12	16	4	11
Sc	9	3	2	3	7	4	8	4	1	5
V	12	28	17	26	27	13	19	10	9	37
Ba	620	567	541	565	570	146	576	404	249	474
Rb	143	146	159	142	140	194	152	171	43	150
Sr	203	213	139	205	218	48	223	142	128	137
Zr	217	276	315	246	264	170	235	220	122	238
Y	32	32	32	32	32	30	32	34	11	31
Nb	22.5	25.7	26.8	25.0	25.6	23.4	24.6	25.2	20.3	22.5
Ga	16	17	19	17	17	17	18	17	16	15
Cu	4	2	4	1	7	2	5	0	4	7
Zn	53	70	64	59	66	39	56	54	24	58
Pb	22	28	24	23	28	29	22	25	20	23
La	72	68	96	79	79	43	75	75	43	52
Ce	119	129	145	108	131	84	106	109	73	121
Th	21	24	24	22	23	22	23	22	19	21

9/20/00 PM

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"†" denotes values >120% of our highest standard.

WSU GeoAnalytical Laboratory 1 *Analyses by XRF*

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 603	BER 637	BER 638	BER 648	BER 648A	BER 648B	BER 649	BER 650	BER 653	BER 654
Date	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00	13-Sep-00
LOI (%)	2.49	4.85	5.93	6.26	11.33	1.28	4.87	5.79	3.69	5.11
	603	637	638	648	648A	648B	649.00	650	653	654
Unnormalized Results (Weight %):										
SiO2	77.90	74.02	73.19	73.37	77.45	61.40	74.35	74.39	72.55	74.16
Al2O3	12.29	13.72	13.55	13.82	13.81	15.83	13.08	13.49	14.55	13.44
TiO2	0.187	0.239	0.239	0.303	0.263	1.269	0.312	0.313	0.306	0.294
FeO	1.18	1.31	1.43	1.84	0.50	5.79	1.71	1.79	1.55	1.80
MnO	0.046	0.071	0.083	0.070	0.012	0.136	0.089	0.060	0.055	0.066
CaO	0.74	2.10	3.27	2.68	1.48	4.46	2.11	1.98	1.75	1.92
MgO	0.36	0.90	0.84	1.10	1.23	1.88	0.67	0.77	0.71	0.78
K2O	4.71	4.98	4.46	4.32	3.03	3.41	4.45	4.38	5.01	4.57
Na2O	2.73	2.77	2.84	2.49	0.96	4.50	2.63	2.48	2.92	2.54
P2O5	0.055	0.074	0.062	0.087	0.047	0.502	0.070	0.065	0.068	0.070
Total	100.20	100.18	99.97	100.08	98.79	99.17	99.47	99.72	99.47	99.64
Normalized Results (Weight %):										
SiO2	77.75	73.89	73.21	73.31	78.40	61.91	74.75	74.60	72.94	74.43
Al2O3	12.27	13.70	13.55	13.81	13.98	15.96	13.15	13.53	14.63	13.49
TiO2	0.187	0.239	0.239	0.303	0.266	1.280	0.314	0.314	0.308	0.295
FeO*	1.18	1.30	1.43	1.84	0.51	5.84	1.72	1.79	1.56	1.81
MnO	0.046	0.071	0.083	0.070	0.012	0.137	0.089	0.060	0.055	0.066
CaO	0.74	2.10	3.27	2.68	1.50	4.50	2.12	1.99	1.76	1.93
MgO	0.36	0.90	0.84	1.10	1.25	1.90	0.67	0.77	0.71	0.78
K2O	4.70	4.97	4.46	4.32	3.07	3.44	4.47	4.39	5.04	4.59
Na2O	2.72	2.77	2.84	2.49	0.97	4.54	2.64	2.49	2.94	2.55
P2O5	0.055	0.074	0.062	0.087	0.048	0.506	0.070	0.065	0.068	0.070
Trace Elements (ppm):										
Ni	18	19	22	17	19	11	19	11	12	16
Cr	4	5	5	18	6	15	8	6	7	10
Sc	0	2	4	2	6	16	6	7	9	3
V	18	19	23	31	15	107	19	25	17	24
Ba	348	513	579	508	756	1593	640	568	775	660
Rb	162	172	160	171	109	78	144	150	134	151
Sr	90	182	276	240	340	545	326	434	325	260
Zr	182	208	196	203	240	319	259	259	297	243
Y	27	32	31	32	18	36	30	28	26	28
Nb	20.9	20.9	21.6	23.5	24.0	23.9	23.1	24.3	23.6	24.8
Ga	13	21	17	16	21	21	17	17	20	16
Cu	4	5	6	11	0	10	5	3	4	6
Zn	39	47	50	54	27	80	53	50	45	46
Pb	23	26	26	26	19	39	27	23	24	29
La	54	69	48	63	33	68	55	53	66	57
Ce	102	114	85	99	64	114	113	117	109	117
Th	19	19	20	22	22	16	20	21	21	24

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"†" denotes values >120% of our highest standard.

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Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 654A	BER 655	BER 656	BER 656A	BER 656B	BER 657	BER 661	BER 663	BER 664	BER 665
Date	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00	14-Sep-00
LOI (%)	1.00	2.92	3.93	1.22	7.28	6.37	13.33	13.91	6.68	13.12
	654A	655	656	656A	656B	657	661	663	664	665
Unnormalized Results (Weight %):										
SiO2	76.61	72.20	74.09	73.62	74.31	73.78	66.09	62.79	69.11	68.22
Al2O3	12.43	14.45	13.06	13.87	12.93	13.56	10.97	10.74	12.80	8.89
TiO2	0.155	0.290	0.264	0.233	0.394	0.291	0.278	0.269	0.347	0.234
FeO	0.86	1.63	1.63	1.25	2.15	1.67	1.99	2.73	3.33	1.84
MnO	0.054	0.046	0.079	0.076	0.058	0.057	0.081	0.090	0.069	0.101
CaO	0.52	1.88	1.93	0.85	2.19	2.31	8.95	9.64	3.77	9.00
MgO	0.28	0.51	0.61	0.34	1.12	1.03	5.34	6.05	1.76	5.69
K2O	5.16	5.13	5.48	5.35	4.78	5.21	4.40	5.62	6.33	4.26
Na2O	3.42	3.17	2.46	3.70	1.46	1.76	1.46	1.14	1.57	1.10
P2O5	0.052	0.069	0.064	0.067	0.100	0.075	0.101	0.077	0.085	0.061
Total	99.54	99.37	99.67	99.36	99.50	99.74	99.66	99.14	99.17	99.40
Normalized Results (Weight %):										
SiO2	76.96	72.66	74.34	74.10	74.69	73.97	66.32	63.33	69.69	68.64
Al2O3	12.49	14.54	13.10	13.96	13.00	13.59	11.01	10.83	12.91	8.94
TiO2	0.156	0.292	0.265	0.235	0.396	0.292	0.279	0.271	0.350	0.235
FeO*	0.86	1.64	1.64	1.26	2.16	1.67	2.00	2.75	3.36	1.85
MnO	0.054	0.046	0.079	0.076	0.058	0.057	0.081	0.091	0.070	0.102
CaO	0.52	1.89	1.94	0.86	2.20	2.32	8.98	9.72	3.80	9.05
MgO	0.28	0.51	0.61	0.34	1.13	1.03	5.36	6.10	1.77	5.72
K2O	5.18	5.16	5.50	5.38	4.80	5.22	4.42	5.67	6.38	4.29
Na2O	3.44	3.19	2.47	3.72	1.47	1.76	1.46	1.15	1.58	1.11
P2O5	0.052	0.069	0.064	0.067	0.101	0.075	0.101	0.078	0.086	0.061
Trace Elements (ppm):										
Ni	9	9	22	10	18	15	20	20	13	8
Cr	1	7	7	4	21	8	11	19	9	9
Sc	4	8	0	5	11	6	5	7	2	6
V	7	24	18	9	34	27	32	39	31	22
Ba	133	993	582	373	419	750	540	558	924	547
Rb	172	119	161	193	188	142	135	141	144	111
Sr	36	289	178	100	165	221	236	339	206	287
Zr	146	275	209	199	191	256	198	198	533	177
Y	25	20	27	29	28	29	22	21	29	24
Nb	21.5	15.5	20.1	23.2	23.7	22.2	15.6	14.5	21.3	14.6
Ga	14	18	15	19	17	17	12	12	17	12
Cu	4	6	4	2	10	3	9	20	8	8
Zn	34	40	49	48	59	47	50	61	71	45
Pb	28	20	27	30	19	28	23	22	32	20
La	42	60	62	57	43	61	51	34	60	45
Ce	86	95	103	122	84	129	108	100	133	75
Th	27	12	18	26	20	22	16	11	18	14

9/20/00 PM

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"R" denotes a duplicate bead made from the same rock powder.

WSU GeoAnalytical Laboratory 4 *Analyses by XRF*

9/20/00 PM

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 666	BER 667	BER 668	BER 669	BER 670	BER 684	BER 685	BER 690	BER 690A	BER 694
Date	14-Sep-00	14-Sep-00	14-Sep-00	15-Sep-00	15-Sep-00	15-Sep-00	15-Sep-00	15-Sep-00	18-Sep-00	18-Sep-00
LOI (%)	**8.61**	**9.83**	**5.47**	**6.63**	**5.97**	**6.71**	**5.05**	**11.70**	**12.81**	**8.89**
	666	667	668	669	670	684	685	690	690A	694
Unnormalized Results (Weight %):										
SiO2	68.75	67.94	73.32	68.94	69.06	72.06	73.32	67.76	66.53	69.71
Al2O3	12.37	11.01	12.67	12.85	13.20	12.80	12.71	11.39	11.42	12.62
TiO2	0.297	0.271	0.281	0.307	0.292	0.315	0.303	0.314	0.337	0.331
FeO	2.42	2.75	1.84	2.93	2.80	2.03	1.90	2.02	2.24	2.11
MnO	0.111	0.121	0.080	0.072	0.067	0.107	0.089	0.082	0.086	0.078
CaO	5.60	6.52	2.25	3.47	2.97	3.88	2.93	9.51	10.66	5.96
MgO	2.46	3.64	1.48	2.35	2.00	1.14	0.85	2.74	3.07	2.04
K2O	5.77	5.88	5.71	6.85	6.79	4.61	4.72	3.90	3.81	4.33
Na2O	1.71	1.21	1.85	1.48	1.77	2.18	2.58	1.80	1.65	2.19
P2O5	0.068	0.085	0.068	0.089	0.092	0.092	0.080	0.099	0.108	0.088
Total	99.56	99.43	99.55	99.34	99.04	99.21	99.48	99.61	99.92	99.46
Normalized Results (Weight %):										
SiO2	69.06	68.33	73.65	69.40	69.73	72.63	73.70	68.03	66.59	70.09
Al2O3	12.43	11.07	12.73	12.94	13.33	12.90	12.78	11.43	11.43	12.69
TiO2	0.298	0.273	0.282	0.309	0.295	0.318	0.305	0.315	0.337	0.333
FeO*	2.43	2.76	1.85	2.95	2.83	2.04	1.91	2.02	2.25	2.12
MnO	0.111	0.122	0.080	0.072	0.068	0.108	0.089	0.082	0.086	0.078
CaO	5.63	6.56	2.26	3.49	3.00	3.91	2.95	9.55	10.67	5.99
MgO	2.47	3.66	1.49	2.37	2.02	1.15	0.85	2.75	3.07	2.05
K2O	5.80	5.91	5.74	†6.90	†6.86	4.65	4.74	3.92	3.81	4.35
Na2O	1.72	1.22	1.86	1.49	1.79	2.20	2.59	1.81	1.65	2.20
P2O5	0.068	0.085	0.068	0.090	0.093	0.093	0.080	0.099	0.108	0.088
Trace Elements (ppm):										
Ni	14	17	14	17	13	26	19	11	18	16
Cr	12	6	9	6	7	14	9	11	16	14
Sc	6	5	7	15	5	3	9	3	0	11
V	30	30	24	15	18	27	12	30	43	41
Ba	887	854	978	1188	1109	650	629	1246	1061	774
Rb	137	131	143	138	150	143	148	129	127	140
Sr	224	185	183	203	199	248	216	298	310	270
Zr	357	295	243	420	294	250	257	219	216	242
Y	25	26	24	23	23	28	28	25	25	27
Nb	17.3	17.4	17.9	17.4	17.6	20.3	21.3	16.5	18.3	21.2
Ga	15	14	17	16	15	17	15	13	15	15
Cu	6	8	5	6	10	12	8	9	16	12
Zn	58	65	50	67	63	55	52	51	50	56
Pb	25	32	23	29	31	26	28	24	22	23
La	61	56	54	62	58	63	77	46	44	56
Ce	117	116	101	122	115	103	117	78	77	109
Th	17	15	18	16	16	24	21	15	16	19

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"R" denotes a duplicate bead made from the same rock powder.

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 701	BER 702	BER 703	BER 841	BER 841A	BER 842	BER 852	BER 853	BER 854	BER 855
Date	18-Sep-00	18-Sep-00	18-Sep-00	18-Sep-00	18-Sep-00	18-Sep-00	18-Sep-00	18-Sep-00	19-Sep-00	19-Sep-00
LOI (%)	**6.60**	**13.29**	**16.73**	**3.48**	**1.13**	**3.03**	**2.53**	**2.84**	**2.91**	**1.92**
	701	702	703	841	841A	842	852	853	854	855
Unnormalized Results (Weight %):										
SiO2	71.93	64.45	59.81	73.54	73.99	74.52	73.39	72.80	73.50	70.08
Al2O3	12.69	13.36	12.37	13.55	13.26	13.44	13.57	13.64	13.63	14.43
TiO2	0.311	0.442	0.465	0.333	0.282	0.274	0.366	0.381	0.342	0.510
FeO	2.10	2.97	3.17	2.04	1.47	1.67	2.08	2.24	1.91	2.51
MnO	0.076	0.095	0.083	0.081	0.076	0.066	0.085	0.081	0.067	0.091
CaO	3.10	8.86	14.67	1.31	0.89	1.11	1.22	1.27	1.27	1.80
MgO	1.83	3.68	4.27	0.78	0.42	0.59	0.62	0.61	0.69	0.90
K2O	5.36	3.92	3.46	4.37	4.84	4.56	4.48	4.51	4.51	4.71
Na2O	1.82	1.38	1.02	3.10	3.86	3.16	3.48	3.45	3.19	3.90
P2O5	0.080	0.153	0.162	0.073	0.087	0.064	0.093	0.116	0.101	0.189
Total	99.30	99.31	99.48	99.18	99.18	99.46	99.38	99.10	99.21	99.12
Normalized Results (Weight %):										
SiO2	72.44	64.90	60.12	74.15	74.60	74.93	73.85	73.46	74.08	70.70
Al2O3	12.78	13.45	12.43	13.66	13.37	13.51	13.65	13.76	13.74	14.56
TiO2	0.313	0.445	0.467	0.336	0.284	0.275	0.368	0.384	0.345	0.515
FeO*	2.12	2.99	3.19	2.06	1.48	1.68	2.09	2.26	1.93	2.53
MnO	0.077	0.096	0.083	0.082	0.077	0.066	0.086	0.082	0.068	0.092
CaO	3.12	8.92	14.75	1.32	0.90	1.12	1.23	1.28	1.28	1.82
MgO	1.84	3.71	4.29	0.79	0.42	0.59	0.62	0.62	0.70	0.91
K2O	5.40	3.95	3.48	4.41	4.88	4.58	4.51	4.55	4.55	4.75
Na2O	1.83	1.39	1.03	3.13	3.89	3.18	3.50	3.48	3.22	3.93
P2O5	0.081	0.154	0.163	0.074	0.088	0.064	0.094	0.117	0.102	0.191
Trace Elements (ppm):										
Ni	11	26	26	21	20	19	18	14	15	17
Cr	5	33	40	8	2	8	7	7	5	13
Sc	10	11	6	5	4	9	4	9	8	7
V	27	74	86	37	12	22	27	29	22	31
Ba	808	1416	1128	576	368	491	551	570	510	676
Rb	144	143	127	146	176	158	144	143	149	142
Sr	204	357	372	205	98	184	170	181	169	232
Zr	284	200	186	233	221	234	250	241	236	313
Y	26	27	26	30	35	33	33	31	31	35
Nb	20.9	18.6	17.0	25.7	26.5	26.1	25.4	24.2	25.6	24.2
Ga	15	18	14	18	17	17	18	18	16	16
Cu	6	21	33	7	3	4	5	6	5	5
Zn	53	97	100	77	55	85	64	65	55	66
Pb	25	23	21	25	26	27	26	25	22	25
La	51	40	52	61	40	71	64	75	65	77
Ce	110	85	77	109	92	121	115	132	110	164
Th	16	16	14	19	22	22	22	24	26	21

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"R" denotes a duplicate bead made from the same rock powder.

Run 0700, Paul Bertetti, CNWRA, Southwest Research Institute

	BER 856	BER 864	BER 865	BER 866	BER 867	BER 868	BER 869	BER 870	BER 871	BER 872
Date	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00
LOI (%)	**4.34**	**2.40**	**3.07**	**2.56**	**2.69**	**3.46**	**3.01**	**9.17**	**2.63**	**3.13**
	856	864	865	866	867	868	869	870	871	872
Unnormalized Results (Weight %):										
SiO2	73.85	75.28	74.51	74.15	75.82	74.78	74.80	71.88	76.16	75.49
Al2O3	13.65	12.83	13.26	13.48	12.70	13.71	13.25	15.60	12.72	12.67
TiO2	0.314	0.253	0.299	0.285	0.244	0.266	0.301	0.434	0.234	0.297
FeO	1.73	1.55	1.84	1.72	1.54	1.56	1.98	2.79	1.35	1.85
MnO	0.070	0.062	0.066	0.059	0.047	0.060	0.070	0.070	0.047	0.076
CaO	0.95	0.82	1.02	1.05	0.80	1.12	0.84	1.48	0.81	0.89
MgO	0.51	0.41	0.55	0.45	0.36	0.57	0.42	1.75	0.45	0.53
K2O	4.55	4.59	4.43	4.69	4.67	4.55	4.74	3.74	4.74	4.43
Na2O	3.23	3.12	3.07	3.24	2.93	3.05	3.04	1.96	2.90	2.85
P2O5	0.099	0.069	0.073	0.082	0.049	0.079	0.062	0.048	0.061	0.063
Total	98.95	98.99	99.11	99.21	99.16	99.75	99.50	99.76	99.48	99.15
Normalized Results (Weight %):										
SiO2	74.63	76.05	75.18	74.74	76.46	74.97	75.18	72.06	76.56	76.14
Al2O3	13.79	12.96	13.38	13.59	12.81	13.74	13.32	15.64	12.79	12.78
TiO2	0.317	0.256	0.302	0.287	0.246	0.267	0.303	0.435	0.235	0.300
FeO*	1.74	1.57	1.85	1.74	1.55	1.57	1.99	2.80	1.36	1.87
MnO	0.071	0.063	0.067	0.059	0.047	0.060	0.070	0.070	0.047	0.077
CaO	0.96	0.83	1.03	1.06	0.81	1.12	0.84	1.48	0.81	0.90
MgO	0.52	0.41	0.55	0.45	0.36	0.57	0.42	1.75	0.45	0.53
K2O	4.60	4.64	4.47	4.73	4.71	4.56	4.76	3.75	4.76	4.47
Na2O	3.26	3.15	3.10	3.27	2.95	3.06	3.06	1.96	2.92	2.87
P2O5	0.100	0.070	0.074	0.083	0.049	0.079	0.062	0.048	0.061	0.064
Trace Elements (ppm):										
Ni	13	16	16	14	16	11	19	22	13	16
Cr	2	2	9	8	5	6	6	26	9	5
Sc	9	2	4	11	2	5	6	9	10	6
V	19	19	13	24	17	19	33	41	17	25
Ba	424	374	498	481	427	529	521	472	417	497
Rb	147	158	144	156	160	153	151	174	166	149
Sr	124	106	145	157	123	171	126	163	110	134
Zr	241	196	218	218	211	211	261	262	200	235
Y	32	31	31	31	32	34	32	32	31	28
Nb	25.4	24.3	24.6	22.8	25.0	23.5	24.5	25.9	23.2	24.2
Ga	16	16	18	18	18	16	16	19	16	15
Cu	2	6	4	1	3	3	12	8	4	2
Zn	58	47	56	52	53	53	105	62	44	59
Pb	27	27	25	24	23	23	24	27	22	23
La	63	64	72	78	57	59	70	70	70	63
Ce	117	101	130	114	105	97	121	111	125	103
Th	27	23	21	23	24	22	24	23	25	21

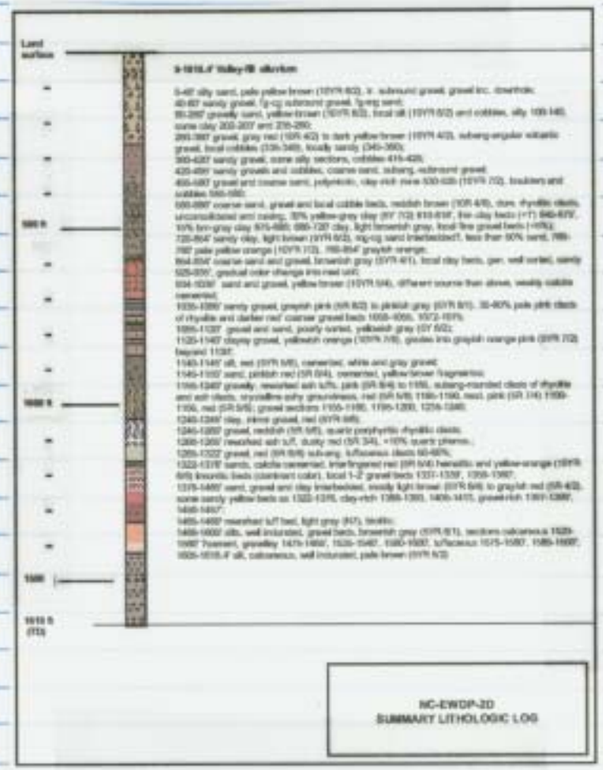
Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"R" denotes a duplicate bead made from the same rock powder.

	BER 873	BER 876	BER 877	BER 878	BER 501	BER 501
Date	19-Sep-00	19-Sep-00	19-Sep-00	19-Sep-00	12-Sep-00	20-Sep-00 repolished
LOI (%)	**3.46**	**4.19**	**3.15**	**3.28**		
	873	876	877	878		
Unnormalized Results (Weight %):						
SiO2	75.02	73.98	75.82	74.50	76.50	76.36
Al2O3	13.14	12.99	12.57	12.70	13.10	12.96
TiO2	0.319	0.299	0.255	0.349	0.115	0.116
FeO	1.91	1.67	1.48	2.12	0.85	0.85
MnO	0.066	0.064	0.057	0.078	0.038	0.039
CaO	1.03	2.23	1.18	1.54	1.75	1.75
MgO	0.58	0.70	0.51	0.60	0.50	0.44
K2O	4.50	4.40	4.51	4.50	4.16	4.17
Na2O	2.79	2.88	2.83	2.75	3.87	3.85
P2O5	0.071	0.121	0.067	0.086	0.020	0.021
Total	99.42	99.33	99.28	99.22	100.91	100.56
Normalized Results (Weight %):						
SiO2	75.46	74.48	76.37	75.08	75.81	75.93
Al2O3	13.22	13.08	12.66	12.80	12.98	12.89
TiO2	0.321	0.301	0.257	0.352	0.114	0.115
FeO*	1.92	1.68	1.49	2.13	0.85	0.85
MnO	0.066	0.064	0.057	0.079	0.038	0.039
CaO	1.04	2.24	1.19	1.55	1.73	1.74
MgO	0.58	0.70	0.51	0.60	0.50	0.44
K2O	4.53	4.43	4.54	4.54	4.12	4.15
Na2O	2.81	2.90	2.85	2.77	3.84	3.83
P2O5	0.071	0.122	0.067	0.087	0.020	0.021
Trace Elements (ppm):						
Ni	16	13	16	11	15	12
Cr	9	4	7	8	3	3
Sc	3	12	4	3	2	7
V	22	28	19	36	0	2
Ba	547	523	452	621	420	418
Rb	148	157	158	146	141	142
Sr	161	162	140	175	215	214
Zr	235	224	193	250	96	97
Y	29	31	29	30	18	18
Nb	22.4	22.4	23.0	22.4	16.2	16.6
Ga	17	16	17	14	15	14
Cu	2	1	3	3	4	2
Zn	54	45	44	53	27	29
Pb	25	26	25	31	25	29
La	71	78	59	64	30	31
Ce	127	102	98	104	61	65
Th	24	20	22	22	21	20

Major elements are normalized on a volatile-free basis, with total Fe expressed as FeO.
"R" denotes a duplicate bead made from the same rock powder.

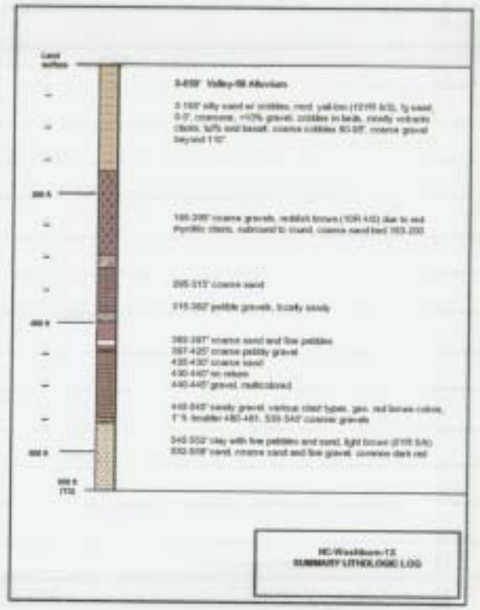
9/20/00 PG

Summary lithologic logs for NC-EWDP-020 and NC-Washburn-1X are posted for information. These are simply copies of those posted on Nye County's web site at www.nyecounty.com



9/20/00 PG

9/30/00 PG



01/10/01 PG

Received e-mail on 12/29/00 from WSU with excel file containing results of ICP-MS analyses for 66 of 74 well cuttings samples. Recall that 8 of the samples did not have enough material for both XRF and ICP-MS (this did not impact the included standards). Hard copies of the data sheets have been requested. I include a printout of the excel file on the next 4 pages.

A comparison of the results with analyses of known and included standards indicates that results are acceptable and are consistent with expectations and previous analyses of similar materials using this method at this (WSU) laboratory. For instance, compare ICPMS results and recommended values from this analysis (sample 505) to previous studies (see p. 126 - table 4).

01/10/01 PG

01/10/01 PB

Results of ICP-MS analyses of well cuttings

Paul Bertetti- CNWRA, Southwest Research Institute 1/10/01

Sample ID	La ppm	Ce ppm	Pr ppm	Nd ppm	Sm ppm	Eu ppm	Gd ppm	Tb ppm	Dy ppm	Ho ppm	Er ppm	Tm ppm	Yb ppm	Lu ppm
BER 501	36.00	62.82	6.35	21.83	4.42	0.52	3.59	0.58	3.48	0.70	1.97	0.30	1.95	0.31
BER 502	0.96	1.52	0.19	0.81	0.26	0.63	0.29	0.05	0.33	0.07	0.21	0.03	0.19	0.03
BER 503	2.96	4.59	0.56	2.17	0.63	0.88	0.59	0.08	0.40	0.07	0.16	0.02	0.13	0.02
BER 504	30.98	58.82	6.58	25.39	6.05	0.84	5.93	1.05	6.95	1.50	4.33	0.68	4.45	0.72
BER 505	23.90	44.27	4.85	18.33	4.19	0.65	3.86	0.65	4.14	0.87	2.44	0.38	2.46	0.41
BER 549	64.91	106.80	11.69	39.58	7.56	0.84	5.86	0.93	5.53	1.08	2.97	0.44	2.85	0.44
BER 550	66.25	109.58	11.95	40.69	7.60	1.01	5.92	0.93	5.38	1.09	2.96	0.44	2.82	0.45
BER 566	77.23	133.47	13.69	46.11	8.48	1.00	6.46	1.02	5.93	1.17	3.20	0.49	3.07	0.49
BER 566A	103.27	165.93	18.32	63.47	11.40	1.35	8.64	1.27	7.28	1.43	3.80	0.56	3.54	0.55
BER 567	80.88	136.33	14.40	48.96	9.00	1.11	6.78	1.07	6.21	1.23	3.31	0.49	3.18	0.49
BER 570	68.69	110.49	12.32	42.27	8.01	1.00	6.30	0.99	5.73	1.14	3.12	0.46	2.98	0.47
BER 571	74.33	126.60	13.53	46.06	8.58	1.00	6.58	1.05	5.99	1.18	3.27	0.49	3.08	0.51
BER571a	93.04	161.44	16.20	54.91	9.28	1.33	6.81	1.02	6.00	1.19	3.31	0.51	3.20	0.51
BER 579	70.08	123.86	12.85	43.47	8.32	1.00	6.29	1.01	5.83	1.18	3.20	0.49	3.11	0.48
BER 583	65.98	110.16	12.18	42.10	8.18	1.04	6.50	1.01	5.96	1.17	3.20	0.48	3.02	0.47
BER 584	66.17	108.33	12.66	43.62	8.69	0.94	7.03	1.12	6.49	1.29	3.52	0.53	3.23	0.50
BER 602	63.93	107.97	11.75	40.00	7.70	0.98	6.12	0.99	5.90	1.17	3.14	0.49	3.06	0.48
BER 603	54.49	87.68	10.06	34.09	6.71	0.77	5.33	0.85	5.02	1.02	2.80	0.43	2.67	0.42
BER 637	62.07	106.20	11.51	39.82	7.80	1.02	6.32	0.99	5.86	1.18	3.22	0.49	3.05	0.48
BER 648	60.11	100.66	10.99	38.04	7.46	0.94	5.99	0.95	5.57	1.15	3.11	0.48	3.00	0.47
BER 648b	76.91	129.59	14.36	52.07	10.08	2.44	8.14	1.25	7.06	1.40	3.65	0.55	3.31	0.53
BER 649	65.42	113.57	11.87	40.45	7.65	0.99	5.91	0.90	5.38	1.08	2.95	0.45	2.80	0.44
BER 650	62.77	110.89	11.41	38.94	7.44	0.93	5.81	0.93	5.38	1.06	2.96	0.45	2.84	0.45
BER 653	65.23	110.84	11.50	38.26	7.00	1.11	5.32	0.82	4.67	0.90	2.44	0.38	2.29	0.39
BER 654	63.06	108.85	11.17	38.05	7.15	0.98	5.56	0.88	5.14	1.05	2.96	0.44	2.79	0.44
BER 655	60.74	90.99	10.21	33.57	5.67	1.08	4.18	0.63	3.59	0.70	1.97	0.30	1.87	0.29
BER 656	59.34	99.91	10.57	35.93	6.66	0.95	5.30	0.83	4.90	0.98	2.72	0.40	2.52	0.40
BER 657	69.00	119.08	12.12	40.89	7.31	1.03	5.55	0.86	5.04	1.01	2.80	0.42	2.64	0.43
BER 661	51.24	88.81	9.05	31.03	5.80	0.91	4.49	0.71	4.04	0.82	2.18	0.34	2.14	0.34
BER 663	44.01	80.88	7.78	27.01	5.04	0.84	4.08	0.64	3.67	0.75	2.05	0.31	1.98	0.31
BER 664	68.10	117.29	11.83	40.16	7.20	1.19	5.60	0.87	5.09	1.03	2.94	0.45	2.91	0.47
BER 665	39.47	69.65	7.18	25.01	4.86	0.73	3.86	0.65	3.94	0.82	2.40	0.39	2.60	0.43
BER 666	58.66	103.86	10.31	35.24	6.47	1.06	4.97	0.80	4.70	0.96	2.67	0.42	2.61	0.41
BER 667	55.84	103.75	9.91	34.65	6.45	1.07	5.13	0.82	4.87	0.98	2.63	0.40	2.52	0.40
BER 668	54.07	94.07	9.50	32.22	6.07	1.02	4.64	0.73	4.33	0.88	2.29	0.36	2.31	0.36
BER 669	64.32	111.48	10.87	36.76	6.44	1.13	4.87	0.75	4.30	0.89	2.44	0.38	2.38	0.39
BER 670	62.63	106.09	10.70	36.49	6.59	1.13	5.01	0.76	4.36	0.89	2.39	0.36	2.33	0.37

WSU GeoAnalytical Laboratory Page 1 *Analyses by ICP-MS*

01/10/01 PB

Paul Bertetti- CNWRA, Southwest Research Institute 1/10/01

Sample ID	Ba ppm	Th ppm	Nb ppm	Y ppm	Hf ppm	Ta ppm	U ppm	Pb ppm	Rb ppm	Cs ppm	Sr ppm	Sc ppm	Zr ppm
BER 501	418	21.16	16.57	19.75	3.54	1.65	2.89	26.61	134.7	3.84	210	2.9	97
BER 502	123	0.39	0.97	1.99	0.08	0.16	0.13	48.15	521.1	9.31	62	0.1	2
BER 503	2551	0.57	0.41	2.09	0.35	0.03	0.21	66.79	108.5	0.50	434	0.1	8
BER 504	910	12.72	16.35	42.34	7.82	1.39	4.44	16.46	126.9	5.16	62	6.0	276
BER 505	831	15.17	8.90	24.66	6.00	0.99	8.04	24.17	150.9	10.01	102	5.2	215
BER 549	480	22.92	24.35	31.14	6.74	2.44	4.22	24.78	152.7	3.57	203	4.2	244
BER 550	689	20.09	23.52	31.41	7.07	1.55	4.08	23.14	132.3	3.60	317	5.3	268
BER 566	623	25.86	26.05	33.43	7.63	1.79	3.38	27.77	143.6	4.57	213	4.7	267
BER 566A	697	30.30	24.93	42.10	7.38	3.14	5.13	31.83	172.7	3.97	155	4.7	260
BER 567	630	25.20	26.83	34.37	7.83	2.27	3.42	28.71	140.9	4.49	220	5.3	283
BER 570	630	23.52	24.02	34.03	6.53	1.61	3.43	26.16	143.7	4.03	203	3.8	228
BER 571	581	24.09	26.69	33.47	7.82	1.76	3.32	27.94	140.1	4.95	207	4.7	278
BER571a	556	24.03	28.36	33.58	8.62	2.76	4.30	26.50	155.0	3.71	137	4.3	317
BER 579	581	23.96	27.85	33.10	7.64	2.82	4.19	27.44	138.1	5.27	213	4.8	272
BER 583	579	23.79	25.12	33.52	6.97	1.75	3.65	25.58	148.2	5.89	216	4.5	237
BER 584	416	24.39	25.94	36.98	6.89	1.73	3.85	26.99	166.9	5.62	137	3.6	222
BER 602	474	22.11	23.31	33.07	7.06	1.53	3.73	27.52	146.3	5.10	134	4.4	238
BER 603	368	22.01	22.08	28.78	5.69	3.13	3.58	23.91	155.8	4.20	88	2.9	183
BER 637	521	23.32	22.50	33.77	6.43	2.57	4.14	28.59	167.0	6.83	175	3.8	209
BER 648	516	21.85	24.00	32.82	6.29	1.87	4.76	26.29	167.4	8.15	235	4.8	206
BER 648b	1600	15.81	26.17	37.85	7.82	2.60	2.58	40.98	78.8	1.78	541	13.1	323
BER 649	667	21.88	25.96	30.50	7.53	2.31	3.43	30.52	141.8	7.57	318	3.7	273
BER 650	581	22.98	25.92	30.86	7.22	2.24	3.48	25.67	146.2	8.35	426	3.7	251
BER 653	781	20.05	24.29	26.01	7.83	2.47	2.38	25.08	130.7	5.41	316	3.2	293
BER 654	673	23.03	25.07	30.18	6.83	2.18	3.72	30.52	144.9	6.46	252	3.7	237
BER 655	1004	13.55	16.68	20.59	6.87	2.08	2.25	20.26	115.3	4.29	280	3.1	251
BER 656	588	19.91	21.41	28.31	5.85	2.18	3.53	27.56	154.3	5.28	172	3.7	201
BER 657	761	22.52	23.62	28.79	6.90	2.22	3.34	27.25	136.9	4.74	215	3.6	246
BER 661	558	16.52	16.56	23.75	5.25	1.31	4.08	21.44	133.7	10.03	235	5.0	192
BER 663	570	13.17	14.35	21.08	5.42	1.50	3.23	23.44	139.1	8.18	330	4.7	199
BER 664	963	18.77	22.22	29.66	12.38	1.31	3.30	33.82	142.0	4.83	202	4.4	512
BER 665	567	13.06	15.49	24.89	4.75	1.31	2.59	18.91	109.5	6.60	279	3.8	177
BER 666	905	16.05	18.74	26.87	8.53	1.92	2.88	28.46	135.9	4.95	221	4.3	340
BER 667	886	16.38	18.57	27.19	7.49	1.91	3.42	30.31	128.6	5.71	183	4.2	291
BER 668	970	17.28	19.14	24.18	6.32	2.01	2.85	25.54	138.3	4.82	178	3.6	234
BER 669	1201	16.46	18.61	24.55	10.45	1.93	2.91	32.60	135.9	4.05	201	4.0	422
BER 670	1128	17.26	18.96	24.92	7.39	1.22	3.12	31.70	146.0	4.23	194	3.7	287

WSU GeoAnalytical Laboratory Page 3 *Analyses by ICP-MS*

Note: I have placed pp. lacrd 3 and 2+4 together to colocate similar samples together. 1/10/01 PB

01/10/01 PB

ICP-MS results of analyses of ENDP authings (WSU)

Paul Bertetti- CNWRA, Southwest Research Institute 1/10/01

Sample ID	La ppm	Ce ppm	Pr ppm	Nd ppm	Sm ppm	Eu ppm	Gd ppm	Tb ppm	Dy ppm	Ho ppm	Er ppm	Tm ppm	Yb ppm	Lu ppm
BER 684	61.68	105.22	10.91	37.38	7.14	1.05	5.61	0.89	5.05	1.02	2.83	0.42	2.65	0.42
BER 685	63.24	107.14	11.29	38.18	7.31	1.02	5.63	0.93	5.30	1.07	2.86	0.45	2.80	0.45
BER 690	50.42	85.89	9.19	32.28	6.23	0.99	5.00	0.80	4.56	0.94	2.50	0.36	2.35	0.37
BER 690A	46.79	81.42	8.73	30.87	6.09	1.02	4.89	0.78	4.47	0.92	2.48	0.37	2.31	0.36
BER 694	58.28	100.08	10.48	36.40	7.11	1.07	5.51	0.86	5.03	1.00	2.67	0.41	2.56	0.40
BER 701	59.83	104.17	10.40	35.66	6.45	1.02	5.08	0.79	4.57	0.91	2.50	0.38	2.42	0.39
BER 702	51.27	90.40	9.57	34.62	6.83	1.19	5.60	0.89	5.13	1.04	2.81	0.42	2.59	0.41
BER 703	42.28	75.10	8.05	29.45	6.00	1.11	4.83	0.78	4.53	0.91	2.44	0.36	2.30	0.36
BER 841	62.88	109.92	11.43	39.54	7.57	0.95	6.01	0.97	5.52	1.11	2.99	0.46	2.90	0.45
BER 841a	50.73	89.55	10.47	37.95	7.97	0.84	6.29	1.07	6.33	1.25	3.38	0.51	3.17	0.51
BER 842	62.63	107.96	11.80	40.61	7.93	0.89	6.00	1.01	5.97	1.18	3.28	0.49	3.09	0.49
BER 852	67.03	115.35	12.27	42.13	8.15	1.04	6.38	1.03	6.02	1.19	3.21	0.48	3.04	0.48
BER 853	64.78	109.65	11.85	40.56	7.90	1.05	6.07	0.98	5.68	1.14	3.10	0.47	3.42	0.46
BER 854	70.27	112.79	12.27	41.70	7.92	1.05	6.10	1.00	5.91	1.17	3.25	0.49	3.16	0.49
BER 855	87.16	147.48	16.21	57.06	10.32	1.62	7.65	1.17	6.74	1.30	3.53	0.53	3.31	0.52
BER 856	68.35	112.55	12.07	40.86	7.79	0.92	6.03	0.99	5.81	1.16	3.22	0.49	3.11	0.48
BER 864	59.46	96.53	11.22	39.08	7.69	0.87	6.02	1.00	5.82	1.14	3.15	0.46	2.97	0.46
BER 865	66.00	108.01	12.09	41.51	7.91	1.01	6.05	0.98	5.66	1.11	3.09	0.46	2.92	0.46
BER 866	67.02	107.72	12.46	42.96	8.26	1.07	6.40	1.01	5.77	1.15	3.11	0.46	2.93	0.45
BER 867	61.63	98.80	11.50	39.50	7.65	0.87	5.82	0.97	5.67	1.14	3.19	0.48	3.06	0.48
BER 868	60.96	98.34	11.65	40.76	8.06	1.05	6.33	1.05	6.12	1.21	3.32	0.49	3.09	0.49
BER 869	75.46	126.73	13.68	46.67	8.65	1.14	6.52	1.04	5.97	1.17	3.18	0.47	3.02	0.48
BER 870	61.06	108.97	11.47	39.89	7.70	1.05	5.94	0.95	5.58	1.10	3.07	0.45	2.99	0.48
BER 871	63.10	97.05	11.65	40.15	7.61	0.92	5.90	0.94	5.55	1.10	3.05	0.44	2.90	0.45
BER 872	63.57	106.16	11.62	39.91	7.63	0.94	5.89	0.95	5.43	1.07	2.98	0.44	2.77	0.44
BER 873	67.15	109.24	12.26	42.03	7.82	1.07	5.91	0.93	5.41	1.06	2.93	0.44	2.75	0.43
BER 876	62.09	98.62	11.46	39.86	7.89	1.06	6.17	0.98	5.63	1.12	3.00	0.45	2.86	0.45
BER 877	55.15	91.16	10.20	35.13	6.86	0.83	5.40	0.88	5.17	1.02	2.84	0.43	2.73	0.42
BER 878	60.53	106.56	11.16	38.53	7.31	1.05	5.77	0.90	5.32	1.07	2.92	0.45	2.80	0.44
BER 866 ®fb	65.41	105.76	12.28	42.18	8.12	1.01	6.30	0.98	5.70	1.12	3.02	0.46	2.86	0.44

Note: analyses followed by ®fb are repeat fusions and analyses.

WSU GeoAnalytical Laboratory Page 2 *Analyses by ICP-MS*

01/10/01 PB

ICP-MS results of ENDP authings (WSU)

Paul Bertetti- CNWRA, Southwest Research Institute 1/10/01

Sample ID	Ba ppm	Th ppm	Nb ppm	Y ppm	Hf ppm	Ta ppm	U ppm	Pb ppm	Rb ppm	Cs ppm	Sr ppm	Sc ppm	Zr ppm
BER 684	664	22.56	21.55	29.37	6.99	2.23	4.77	29.24	138.9	5.63	242	4.3	249
BER 685	627	22.92	22.97	30.31	7.06	2.10	4.10	30.06	143.8	5.22	210	3.9	251
BER 690	1230	17.14	17.67	26.55	5.59	1.45	3.71	23.05	127.1	6.68	294	4.9	197
BER 690A	1036	16.41	17.20	26.01	6.09	1.47	3.57	22.15	124.9	7.30	302	5.1	215
BER 694	763	19.13	20.57	28.86	6.62	1.91	3.80	24.58	135.3	6.74	267	4.6	239
BER 701	806	17.71	19.83	26.31	7.29	2.12	3.52	25.09	141.4	5.46	201	4.4	276
BER 702	1406	19.06	17.96	29.45	5.67	1.24	4.92	27.93	143.0	9.54	354	8.2	198
BER 703	1113	15.48	16.15	26.52	5.28	1.29	4.57	23.03	129.2	9.66	372	8.8	193
BER 841	572	24.50	25.19	31.74	6.93	2.31	3.74	28.43	142.4	4.65	200	4.8	241
BER 841a	359	24.99	26.79	36.27	6.90	2.66	4.60	26.97	169.9	3.52	95	3.9	225
BER 842	505	23.18	25.62	32.43	6.83	2.14	3.77	27.75	154.2	4.54	180	3.5	229
BER 852	573	22.84	24.40	32.71	7.09	2.24	3.65	26.07	138.0	6.42	166	3.8	238
BER 853	586	23.07	24.65	31.46	6.99	2.28	3.53	26.90	139.7	6.15	178	4.0	235
BER 854	528	24.25	25.12	32.88	6.87	2.22	3.64	25.30	146.0	5.94	166	4.3	234
BER 855	687	21.52	24.32	36.45	8.12	2.19	3.49	25.67	137.3	4.00	227	5.9	299
BER 856	436	26.38	25.75	32.68	7.24	2.57	3.90	28.92	144.7	5.25	123	4.3	246
BER 864	385	23.26	23.91	32.64	6.12	2.91	3.89	26.63	152.9	5.50	103	3.5	195
BER 865	509	22.53	23.91	31.76	6.10	2.67	3.74	25.45	140.7	6.76	142	3.8	204
BER 866	502	23.33	23.08	32.05	6.25	2.45	3.66	25.22	152.7	6.22	155	3.9	207
BER 867	426	24.52	24.22	32.30	6.31	2.68	3.92	24.79	155.8	7.21	121	3.3	200
BER 868	538	22.88	23.20	34.92	6.20	2.43	3.67	26.13	152.3	7.40	168	3.8	200
BER 869	530	23.94	23.90	33.07	7.06	2.39	3.67	26.24	147.6	6.58	123	3.9	251
BER 870	473	24.16	25.73	32.20	6.95	1.90	3.21	29.61	171.9	16.42	158	8.0	230
BER 871	410	23.98	23.49	31.10	5.90	2.83	3.77	24.58	160.0	7.13	106	3.6	191
BER 872	506	23.43	24.46	30.30	6.66	2.67	3.62	26.30	145.9	7.54	132	3.9	226
BER 873	548	23.22	22.58	30.13	6.40	2.41	3.75	25.76	144.9	7.96	158	4.0	222
BER 876	525	22.35	22.39	32.46	6.09	1.52	3.79	26.51	152.2	7.20	157	4.6	206
BER 877	462	23.23	22.99	29.75	5.94	2.60	4.32	26.01	156.1	7.07	139	4.1	189
BER 878	625	21.89	22.42	29.90	6.72	1.47	3.32	28.64	141.3	6.85	170	3.9	229
BER 866 ®fb	497	23.72	23.31	32.16	6.29	2.48	3.73	25.52	152.4	6.35	154	4.2	209

WSU GeoAnalytical Laboratory Page 4 *Analyses by ICP-MS*

01/10/01 PB

A check of the sample list shows the following 8 samples did not have enough powder to be analyzed by both XRF and ICP-MS. As expected the majority of these were subsamples selected by Cherrington from a larger well cuttings samples. These subsamples were selected by Cherrington based on observed differences between the subsample (often a large pebble or grain) and the rest of the cuttings. Sample descriptions have been previously given (pp. 87-95).

Samples without ICP-MS data:

- 578
- 579 A
- 594 A
- 638
- 648 A
- 654 A
- 656 A
- 656 B — 8 total

1/16/2001 BAW

Restarting Np sorption experiments for calcite. Old solutions from notebooks 309 page 2 and 525 page 23 were used. New solutions were also prepared. Two solutions of 125g $CaCO_3$ in 0.02M $NaHCO_3$ (3.5 Liters) were prepared. These will be prepared in the way that the old standards were prepared.

reagents: Fisher C64-500 $CaCO_3$ lot# 986396
Fisher S233-500 $NaHCO_3$ lot# 006275
nanopure H_2O

Two 3.5 L solutions were prepared.
5.88g of $NaHCO_3$ required for each 3.5 L solution.
The $NaHCO_3$ was weighed in a 600mL beaker. All weights were measured with the Mettler PM 4600 balance.

1/16/2001 cont BAW

wt of beaker (g)	wt of $NaHCO_3$ and beaker (g)	wt of $NaHCO_3$ (g)	Solution id
228.79	~~234.67~~ ~~234.72~~ BAW 1/16/2001	5.88	S1
220.84	226.72	5.88	S2

Then approximately 300mL of H_2O was added to each beaker. The solutions were mixed (agitated - not combined). Each solution was added to a 2 gal PP bottle that was already weighed. H_2O was added to each bottle until a final weight of 3500g was reached

wt of bottle (g)	wt of solution and bottle (g)	wt of solution (g)	Solution id
249.30 ~~258.09~~ BAW	3749.3	3500	S1
248.02 ~~255.54~~ 1/16/2001	3748.0	3500	S2

To each of these solutions S1 and S2, 125g $CaCO_3$ was added.

wt of glass beaker (g)	wt of $CaCO_3$ and beaker (g)	wt of $CaCO_3$ (g)	Solution id
102.76	227.76	125.00	S1
103.67	228.67	125.00	S2

Solid and solution was thoroughly mixed and placed on gyratory shaker for 20 mins at ~ 80 rpm.

1/17/2001 Equilibrate solutions with atmospheric CO_2
The four 3.5L solutions (S1 and S2 from 1/16/2001 and 1-A and 1-B from 3/18/99 - 309/2) were opened and placed in the gyratory shaker for 10 minutes at ~80 rpm. These four solutions were capped and vigorously shaken by hand

1/17/2001 cont BW

The caps were removed and the solutions were placed on the gyratory shaker for 20 minutes at ~80 rpm.

$CaCO_3$ solns 1, 2, and A to F (GC525/23) were uncapped and gently swirled by hand for about 10 seconds. The solutions were capped and vigorously shaken by hand. The caps were removed and loosely placed on top for about 20 minutes. The caps were then tightly sealed.

1/18/2001 BW

Equilibrating solutions with atmospheric CO_2

Four 3.5 L solutions (S1 and S2 from 309/147 and 1-A and 1-B from 309/2) were openned and placed in the gyratory shaker for 10 minutes at ~80 rpm. These four solutions were capped and vigorously shaken by hand. The caps were removed and the solutions were placed on the gyratory shaker for 20 minutes at ~80 rpm.

$CaCO_3$ solns 1, 2, and A to F (GC525/23) were uncapped and gently swirled by hand for about 10 seconds. The solutions were capped and vigorously shaken by hand. The caps were removed and loosely placed on top for about 20 minutes. The caps were then tightly sealed.

Analysis of $CaCO_3$ samples (GC525/23 and 309/2 and 309/147) for Ca concentration using the Perkin Elmer 3100 Atomic Absorption Spectrometer and pH using the Orion model 920A pH meter and Orion 8103 Ross combination electrode w/ ATC probe.

1/19/2001 BAW

Prepared Ca^{+2} standards for AA calibration

Reagents: Fisher SC191-500 Ca^{+2} reference std at 1000 ppm ±1%, lot 986835-24
Fisher P217-500 KCl, lot 901422

1/18/2001 BAW

Soln 149-A Target concentration 0.1% (w/v) KCl in nanopure water.
The below mass of KCl was dissolved with nanopure water in a 500 mL Volumetric flask.

wt of boat (g)	wt of boat + KCl (g)	wt of KCl (g)
2.3586	2.8616	0.5020

The following 5 Standards were prepared as follows: Pipetted with class A volumetric pipette into volumetric flask, Filled to mark with 0.1% KCl (Soln 149-A)

$$5\text{ ppm} = \frac{(1000\text{ ppm Ref Std})(1\text{ mL})}{200\text{ mL}}$$

$$4\text{ ppm} = \frac{(5\text{ ppm } Ca^{+2})(40\text{ mL})}{50\text{ mL}}$$

$$2\text{ ppm} = \frac{(5\text{ ppm } Ca^{+2})(40\text{ mL})}{100\text{ mL}}$$

$$0.5\text{ ppm} = \frac{(5\text{ ppm } Ca^{+2})(10\text{ mL})}{100\text{ mL}}$$

$$0.2\text{ ppm} = \frac{(5\text{ ppm } Ca^{+2})(2\text{ mL})}{50\text{ mL}}$$

Prepared KCl solutions for AA analysis of $CaCO_3$ samples

Reagent P217-500 KCl, lot 901422

Soln KA Target concentration 10% (w/v) KCl
The below mass of KCl was used. The 10 mL volumetric flask was filled to mark with nanopure water

wt of boat (g)	wt of boat + KCl (g)	wt of KCl (g)
13.8234	14.8248	1.0014

1/19/2001
cont BAW

Solution KB Target concentration 1% (w/v) KCl

1mL of Solution KA (309/149) was diluted to mark in a 10mL volumetric flask with nanopure water

Solution KC Target concentration 0.1% (w/v) KCl
2 ~~50~~mL (class A vol. pipet) of soln ~~KB (309/150)~~ BW 1/19/2001 was diluted to mark into a 200 mL volumetric flask with nanopure water.

Preparation of $CaCO_3$ samples for AA analysis

Sample aliquots were obtained by using a syringe with a Dyna Gard model D62M-330-100 Syringe Filter (lot 75-66A)

Samples analyzed were:
S1 and S2 (309/147)
1A and 1B (309/2)
1, 2, A, B, C, D, E, + F (525/23)

BW 1/19/2001
Undiluted samples were ~~to~~ prepared by filling to mark a 10 mL volumetric flask with sample and then adding 0.1mL (eppendorf 50-100 ul) of soln KA (309/149)

Ten fold dilution samples (/DF10) were prepared by adding 1mL of sample (eppendorf 1000 ul) and 1mL of solution KB ~~(150)~~ BW 1/19/2001 (309/150) and diluting to mark in a 10mL volumetric flask with nanopure water.

Blank for AA is KC soln (309/150)

BW 1/19/2001

1/19/2001
cont BAW

Standard absorbance

Std conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
0.2	0.019	0.019	0.018	0.019	0.018
0.5	0.040	0.039	0.038	0.040	0.038
2.0	0.141	0.141	0.141	0.142	0.141
4.0	0.270	0.269	0.268	0.269	0.267
5.0	0.335	0.335	0.336	0.332	0.333

Samples (Absorbance)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
~~S1~~	~~0.065~~	~~0.067~~	~~0.067~~	~~0.067~~	~~0.067~~
S2	0.067	0.067	0.068	0.068	0.067
1A	0.027	0.027	0.028	0.027	0.026
1B	0.026	0.026	0.025	0.026	0.026
1 DF10	0.157	0.158	0.157	0.158	0.157
2 DF10	0.155	0.155	0.156	0.154	0.154
A DF10	0.159	0.158	0.159	0.159	0.156
B DF10	0.160	0.159	0.159	0.159	0.160
C DF10	0.155	0.156	0.155	0.156	0.157
D DF10	0.157	0.158	0.157	0.155	0.156
E DF10	0.155	0.154	0.154	0.153	0.154
F DF10	0.156	0.157	0.156	0.156	0.158
S1	0.065	0.067	0.067	0.067	0.067
CCV 4ppm	0.264	0.265	0.265	0.265	0.265

~~BW~~ BW 1/19/2001

pH analysis of $CaCO_3$ samples

Orion model 920A pH meter (S/N 002230) and Orion 8103 Ross combination electrode w/ ATC probe

1/19/2001 cont BW

Sample aliquots were obtained by using a syringe BW corrected 1/19/2001 with a Dynaguard model DG2M-330-100 syringe filter (lot 75-66A)

(calibrated with pH 7 and pH 10 buffer
slope = 104.0%

SAMPLES

ID	pH
51	8.42
52	8.48
1A	9.36
1B	9.36
1	8.06
2	8.07
A	8.04
B	8.12
C	8.12
D	*
E	
F	

*Analyzed pH buffer 7.03 with reading of 7.22
recalibrated with pH 7 and pH 10 buffer
slope = 103.5

1/22/2001 BW

Equilibrating $CaCO_3$ solns w/ atmospheric CO_2
Four 3.5L solutions (51 and 52 from 309/147 and 1A and 1B from 309/2) were opened and placed in the gyratory shaker for 10 minutes at 80rpm. These four solutions were capped and vigorously shaken by hand. The caps were removed and the solutions were placed in the gyratory shaker for 20 minutes at 80rpm. This was done once a day for 5 days a week until 3/9/2001

1/22/2001 cont BW

pH analysis of $CaCO_3$ Solutions
Solutions analyzed were:
51 + 52 (309/147)
1A + 1B (309/2)
1, 2, A, B, C, D, E, F (525/23)
Samples aliquots were obtained by using a syringe with a Dynaguard model DG2M330-100 syringe filter (lot 75-66A)

Orion model 920A pH meter (S/N 031322) and Orion model 8103 Ross combination electrode w/ ATC probe, calibrated with pH 7 (1/22/2001) and pH 10 (1/22/2001)
slope = 100.8%

SAMPLES

ID	pH
51	8.68
52	8.75
1A	9.37
1B	9.34
1	7.24 / 7.27 (remeasured)
2	7.90
A	7.94
B	7.96
C	7.93
D	8.04
E	8.04
F	8.13
pH 7 buffer	7.14
51 reanalysis	8.74

1/22/2001 BW

1/23/2001 BW

0.1M HCl for conditioning pH electrodes
Conc HCl = 12M
Added 2mL of conc HCl (Fisher P508-212, lot #418110) to ~238mL of nanopure H_2O.

pH analysis of $CaCO_3$ solns filtered & collected on 1/22/2001 (see 309/153) - (reanalysis)

Orion model 920A pH meter (S/N 039522) and Orion model 8102 Ross Combination electrode with ATC probe calibrated with pH 7 buffer (1/22/2001) and pH 10 buffer (1/22/2001). Slope = 98.8

SAMPLES

ID	pH
S1	8.76
S2	8.76
1A	9.37
1B	9.37
1	7.08
2	7.30
pH buffer 9 (1/23/2001)	8.97
A	7.40
B	7.54
C	7.60
D	7.59
E	7.72
F	7.70
S1 reanalysis	8.81
pH buffer 9 (1/23/2001)	8.97

1/23/2001 BW

2/1/2001 BW

Reagents for T.O.C. Analyzer

Target concentration 21% Phosphoric Acid - SOLN A
Added 37mL (100mL graduated cylinder) of 85% phosphoric acid* to 188mL (250mL grad cylinder) of nanopure water

Sodium ~~Sodium~~ Persulfate Solution - SOLN B
Added below mass of Sodium Persulfate # into 213mL of nanopure water (250mL grad cylinder) and 9mL (10mL grad cylinder) of 85% phosphoric acid.

wt of beaker (g)	wt of beaker + $Na_2S_2O_8$ (g)	wt of $Na_2S_2O_8$ (g)
230.29	255.29	25.00

85% Phosphoric acid* - Fisher A242-4 cert ACS, lot #001815, open 2/1/2001.

Sodium Persulfate # Acros 20202-5000, lot # B0104212, open 2/1/2001.

01 Feb 01 PD

Made initial standards for test and installation of Telemar. Dohrmann Phoenix 8000 Total organic carbon analyzer. Standards are for a test of inorganic carbon measurement (IC) and will be made from Na_2CO_3 and deionized water (nanopure water).

We will test the 0.1 to 20 ppm method range for IC. I will make 20, 10, and 5 ppm standards by weighing a quantity of Na_2CO_3 into water to make the 20 ppm standard (ppm as C) and diluting the 20 ppm standard with nanopure water to make the 10 and 5 ppm standards.

01 Feb 01

Because this is an evaluation of instrument performance upon installation, Aes certified Na_2CO_3 is all that is required for use in making the cal. standards.

Na_2CO_3 has a formula wt. of 105.99 g/mole
I will use Fisher lot # 006077
C has mol. wt of 12.011 g/mole

1 mole C in 1 mole Na_2CO_3

20 ppm of C $\Rightarrow$ 20×10^{-6} g C / g solution

$$\frac{20 \times 10^{-6}\ g\ C}{g\ soln} \times \frac{105.99\ g\ Na_2CO_3}{12.011\ g\ C} = \frac{1.76488 \times 10^{-4}\ g\ Na_2CO_3}{g\ solution}$$

$\therefore$ I will need 1.76488×10^{-4} g per g solution to make a 20 ppm std.

To make one liter: $\frac{1.76488\ g\ Na_2CO_3}{g} \times \frac{1000\ g}{L} = 0.176488\ g\ Na_2CO_3$

Tared a small plastic weighing dish on the mettler AE240 and weighed

0.1756 g Na_2CO_3

mixed the Na_2CO_3 in a 250-mL glass beaker using nanopure H_2O (nH_2O) and a stir bar, taking care to rinse all of the sodium carbonate into the beaker. After the Na_2CO_3 was dissolved in ~100 mL of nH_2O, I transferred, quantitatively, the solution to a clean 1 L volumetric flask. I added nH_2O to make up to the mark and mixed the solution.

01 Feb 01

The solution flask was labeled (20) for 20 ppm Std. ~150 mL was transferred to a clean 250-mL glass beaker. 100 mL(A) and 200 mL vol flasks were pp 2/1/01 cleaned and rinsed, and a 50 and 100 mL(B) flasks were cleaned and rinsed using the 20 ppm solution. Additional 20 ppm solution was transferred to the 250-mL beaker and was then used to fill the 50 and 100 mL vol. flasks that had been pre-rinsed. These were made up to the mark.

Solution from the 50 mL flask was then quantitatively transferred to the clean 100 mL(A) flask while solution from the 100 mL(B) flask was transferred to the 200 mL flask. When made up to the mark w/ nH_2O, the ~~100~~ mL (A) flask contained 5 ppm C and the pp 2/1/01 200 mL flask contained 10 ppm C

Note 100(A) is actually a 200 mL flask

$\Rightarrow$ 200 mL (A) labeled 5 ppm

50 mL of 20 ppm C diluted to 200 mL

200 mL (B) labeled 10 ppm
100 mL of 20 ppm C diluted to 200 mL

Tests in initial runs of the Toc were satisfactory.

pp 2/1/01

02 Feb 01 PB

Because the pH values of calcite/water solutions (see p. 154) have not stabilized, a gas bubbler will be added to some bottles to enhance gas exchange. pH will then be checked at intervals to evaluate if gas exchange is causing the pH variations.

Bubblers are added to solutions 1, 2, and B.

05 Feb 2001 BW

Reanalyzed IC standards (309/156-157) on Tekmar-Dohrmann phoenix 8000 TOC analyzer.

Prepared TOC standards for analysis on Tekmar Dohrmann

reagents - Aqueous carbon (acidified) potassium acid phthalate - Tekmar Dohrmann P/N 511-946 lot # 34690-60 open 2/5/2001, exp ~~6/3~~ BW 06/2003 @ 1000 ppm C.
2/5/2001
- nanopure water

20 ppm std - Added 1 mL (vol pipet) of 1000 ppm C, ~~and~~ 2/11/01 BW to a final volume of 50 mL (vol flask) with nanopure water.

10 ppm std Added 10 mL (vol pipet) of 20 ppm C to a final volume of 20 mL (10 mL vol pipet) with nanopure water

5 ppm std Added 5 mL (vol pipet) of 20 ppm C to a final volume of 20 mL (10 mL/5 mL vol pipet) with nanopure water

15 ppm check std. Added 15 mL (10 mL/5 mL vol pipet) to a final volume of 20 mL (5 mL vol pipet) with nanopure water.

05 Feb 2001 BW

07 Feb 2001 BAW

Prepared standards for TOC analysis

reagents - Aqueous carbon (acidified) potassium acid phthalate Tekmar Dohrmann P/N 511-946 lot # 34690-60 opened 2/5/2001, exp 06/2003 @ 1000 ppm C
- nanopure water

Soln A - Target concentration 20 ppm C
~~5 mL of~~ BW 2/7/2001
5 mL (vol pipet) of 1000 ppm C (reagent) into a 250 mL volumetric flask and diluted to mark with nanopure water

Serial dilution of Soln A for 2 more standards.

Soln B - Target concentration 10 ppm C
100 mL (vol pipet) of nanopure water into a 200 mL volumetric flask and diluted to mark with 20 ppm C (Soln A 309/159).

Soln C - Target concentration 5 ppm C
50 mL (vol pipet) of nanopure water into a 100 mL volumetric flask and diluted to mark with 10 ppm C (Soln B 309/159)

07 Feb 2001 BW

16 Feb 2001
BAW

Data from initial check of TOC Analyzer

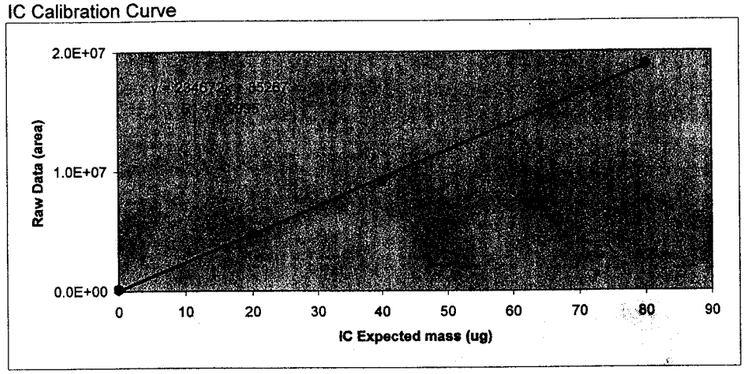
Inorganic Carbon (IC) Analysis
Initial Check for TOC Analyzer

PRN and RAW files located at D/Phoenix/Autosample/IC_01Feb01A
RPT files located at D/Phoenix/Reports/IC_01Feb01A
Excel file located at D/Phoenix/Excel data/IC_01Feb01A

IC Standard Data

TOC Std (ppm)	TOC expected mass (ug)	Raw Data Rep 1
0	0	136159
5	20	4761114
10	40	9171721
20	80	18926092

IC Calibration Curve



IC Blank Data

Blank Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Average*
IC Range 2	163157	24655	19594	22926	17143	17789	19286

*The average of the last 3 reps is used in the expected mass calculation for samples

IC Sample Data

Sample ID	Raw Data Rep 1	Rep 2	Rep 3	Average	Average ug TOC	Average ppm TOC	CV %
S1 @ 0 ppm	144969	143701	147592	145421	0.469394	0.117349	*
S2 @ 5 ppm	5332657	5363004	5359986	5351882	22.65552	5.663879	13.27758176
S3 @ 10 ppm	10136520	10259279	10323967	10239922	43.48476	10.87119	8.711893622

Note: These samples were standards so a manual calculation using the y-intercept was performed.
The original data classification was "sample" so the y-int was not used in the calculation

BW 2/16/01

16 Feb 2001
BW

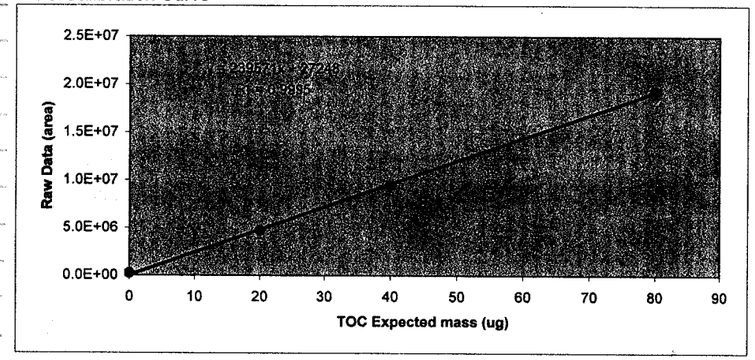
Total Organic Carbon (TOC) Analysis
Initial Check for TOC Analyzer

PRN and RAW files located at D/Phoenix/Autosample/TOC_07Feb01A
RPT files located at D/Phoenix/Reports/TOC_07Feb01A
Excel file located at D/Phoenix/Excel data/TOC_07Feb01A

TOC Standard Data

TOC Std (ppm)	TOC expected mass (ug)	Raw Data Rep 1
0	0	213840
5	20	4687581
10	40	9433512
20	80	19313954

TOC Calibration Curve



TOC Blank Data

Blank Type	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Average*
TC Range 2	377523	143159	107252	142703	144832	115412	134316

*The average of the last 3 reps is used in the expected mass calculation for samples

TOC Sample Data

Sample ID	Raw Data Rep 1	ug TOC	ppm TOC	CV %
S1 @ 10 ppm	9340161	38.4264	9.6066	"-3.9"
CV @ 10 ppm	9411619	39.1716	9.7929	-2.1

CV = Calibration Verification
Note: sample calculation does not use y-intercept, however CV calculation does use y-intercept

BW
2/16/2001

2/20/2001
BAW

Lanthanum Solutions for AA analysis

Target concentration 10% (w/w) La - Soln A
Target concentration 1% (w/w) La - Soln B
Target concentration 0.1% (w/w) La - Soln C
Balance = Mettler PM 4600
Reagents - Lanthanum chloride $LaCl_3 \cdot 7H_2O$ - Fisher
cat # L9-250, lot # 985153A
- nanopure water

Soln A $\quad 36.5\,g\ LaCl_3 \cdot 7H_2O \left(\frac{138.91\ g\ La/mol}{371.374\ g/mol\ LaCl_3 \cdot 7H_2O}\right)$

$(100\,g\ H_2O + 36.5\,g\ LaCl_3 \cdot 7H_2O)$

mass of beaker (g)	mass of beaker + water (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
29.95	129.95	166.48

Soln B $\quad 8.52(g)\ LaCl_3 \cdot 7H_2O \left(\frac{138.91\ g/mol\ La}{371.374\ g/mol\ LaCl_3 \cdot 7H_2O}\right)$

$100\,g\ H_2O + 8.52\,g\ LaCl_3 \cdot 7H_2O$ $\quad -0.01000$

mass of beaker (g)	mass of beaker + water (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
36.51g	366.51	375.03

Soln C $\quad 1.34g\ LaCl_3 \cdot 7H_2O \left(\frac{138.91\ g\ La/mol}{371.374\ g\ LaCl_3 \cdot 7H_2O/mol}\right)$

$500\,g\ H_2O + 1.34\,g\ LaCl_3 \cdot 7H_2O$

mass of beaker g	mass of beaker + water (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
89.22	589.22	590.56

2/21/2001
BAW

Na/K/Sr/Ca mixed soln stds for AA analysis

Stock solns for curve preparation

Reagents - 1000 ug/mL Sr - Spex certiprep cat # PLSR2-2Y
lot # 7-1235R, rec 5/12/2000, exp 4/15/2001
- 1000 ug/mL K - Spex certiprep cat # PLK2-2Y
lot # ~~PLK~~ 2/21/01 7-93K, rec 5/12/00, exp 4/15/2001
- 1000 ug/mL Na - Spex certiprep cat # PLNA2-2Y
lot # 7-87Na, rec 5/12/2000, exp 4/15/2001
- 1000 ug/mL Ca - Spex certiprep cat # PLCA2-2X
lot # 7-114Ca, rec 1/31/2001, exp 1/31/2002
- nanopure water

Target conc Sr at 25 ug/mL - Added 5 mL (vol pipet) of 1000 ug/mL Sr to a ~~B~~ 2/21/01 200 mL (vol flask) and diluted to mark with nanopure water (Sr Stock)

Target conc K at 10 ug/mL - Added 1 mL (vol pipet) of 1000 ug/mL K to a 100 mL (vol flask) and diluted to mark with nanopure water. (K Stock)

Target conc Na at ~~3 ug/mL - Added 0.6 mL~~ BAW 2/21/2001
100 ug/mL - Added 1 mL (vol pipet) of 1000 ug/mL Na to a 10 mL vol flask and diluted to mark w/ nanopure water. (Soln Na)

Target conc Na at 3 ug/mL - Added 6 mL (1+5 vol pipet) of 100 ug/mL Na (soln Na 309/163) to a 200 mL vol flask and diluted to mark with nanopure water (Na Stock)

Target conc Ca at 20 ug/mL - Added 2 mL of (vol pipet) 1000 ug/mL Ca to a 100 mL vol flask and diluted to mark with nanopure water (Ca Stock)

2/21/2001
cont BAW

Stock solns for high level conc

Reagents - NaCl - Fisher S2713, lot# 986412
- $SrCl_2 \cdot 6H_2O$ - Fisher S541-500, lot# 000052
rec 2/9/01, open 2/21/2001
- $CaCl_2 \cdot 2H_2O$ - Fisher C79-500, lot 995698

- KCl - Fisher P217-500, lot# 006242
- nanopure water

Balance: Mettler PM4600

Na high - Target conc 23000 ug/mL Na
Na mol wt = 22.9898 g
NaCl mol wt = 58.44 g

$$\frac{29.23\text{ g NaCl}\left(\frac{22.9898}{58.44}\right)\times 10^6}{500} = 23{,}000\text{ ug/mL Na}$$

Diluted 29.23 g NaCl to 500 mL (vol flask) to mark with nanopure water

wt of beaker (g)	wt of beaker + NaCl (g)
53.77	83.01

Ca high - Target conc 20,000 ug/mL Ca
Ca mol wt = 40.08 g
$CaCl_2 \cdot 7H_2O$ mol wt = 147.02 g

$$\frac{36.68\text{ g } CaCl_2 \cdot 7H_2O\left(\frac{40.08}{147.02}\right)\times 10^6}{500\text{ mL}} = 20{,}000\text{ ug/mL Ca}$$

2/21/2001
cont BAW

wt of beaker + ~200 mL H_2O (g)	wt. with $CaCl_2 \cdot 7H_2O$ added (g)
465.05	501.75

Sr High - Target conc 44,000 ug/mL Sr
Sr mol wt = 87.62 g
$SrCl_2 \cdot 6H_2O$ mol wt = 266.62 g

$$\frac{66.94\text{ g } SrCl_2 \cdot 6H_2O\left(\frac{87.62}{266.62}\right)\times 10^6}{500\text{ mL}} = 44000\text{ ug/mL Sr}$$

wt of beaker + ~200 mL H_2O (g)	wt with $SrCl_2 \cdot 6H_2O$ added (g)
472.16	539.10

K High - Target conc 40,000 ug/mL K
K mol wt = 39.102 g
KCl mol wt = 74.56 g

$$\frac{38.14\text{ g KCl}\left(\frac{39.102}{74.56}\right)\times 10^6}{500\text{ mL}} = 40{,}000\text{ ug/mL K}$$

wt of beaker (g)	wt of beaker and KCl (g)
56.71	94.85

2/21/2001
cont BAW

Blanks with high metal conc - non Sr analysis

2/21/01
Cont BW

BlkCa - Target conc 10000 ppm Ca
Added 25mL (vol pipet) of 20000 ppm Ca (309/164) and 5mL (vol pipet) of 1% La (309/162) to a 50mL vol flask and diluted to mark with nanopure water.

Blk Na - Target conc 11500 ppm Na
Added 25mL (vol pipet) of 23000 ppm Na (309/164) and 5mL (vol pipet) of 1% La (309/162) to a 50 mL Vol flask and diluted to mark with nanopure water

BlkSr - Target conc 22000 ppm Sr
Added 25 mL (vol pipet) of 44000 ppm Sr (309/165) and 5mL (vol pipet) of 1% La (309/162) to a 50 mL Vol flask and diluted to mark with nanopure water.

Blk K - Target conc 20000 ppm K
Added 25 mL (vol pipet) of 40000 ppm K (309/165) and 5mL (vol pipet) of 1% La (309/162) to a 50 mL Vol flask and diluted to mark with nanopure water.

Working level Calcium solutions

2/21/2001
BW

All final volumes were 50 mL (volumetric flasks)
Volumetric pipets were used to transfer all solutions in the 0.5 mL to 25 mL volume range
High concentrations represent 0.5 N solutions of the cations
% LaCl is w/w

BW
2/2/2001

Calcium calibration curve

Soln ID	Target Conc of Ca (ppm)	Target Conc of LaCl (%)		Vol (mL) of 309/163 (20 ppm Ca)	Vol (ml) of 309/162 (1% LaCl)
Ca1*	4	0.1		10	5
Ca2	2	0.1		5	5
Ca3	1.2	0.1		3	5
Ca4	0.8	0.1		2	5
Ca5	0.4	0.1		1	5
Ca6	0.2	0.1		0.5	5

* AA sensitivity check

Calcium solutions with high potassium concentrations

Soln ID	Target Conc of Ca (ppm)	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/165 40000 ppm K	Vol (ml) of 309/162 1% LaCl
HCaK	4	20000	0.1	10	25	5
MCaK	1.2	20000	0.1	3	25	5
LCaK	0.2	20000	0.1	0.5	25	5

Calcium solutions with high sodium concentrations

Soln ID	Target Conc of Ca (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/164 23000 ppm Na	Vol (ml) of 309/162 1% LaCl
HCaNa	4	11500	0.1	10	25	5
MCaNa	1.2	11500	0.1	3	25	5
LCaNa	0.2	11500	0.1	0.5	25	5

Calcium solutions with high stronium concentrations

Soln ID	Target Conc of Ca (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/165 44000 ppm Sr	Vol (ml) of 309/162 1% LaCl
HCaSr	4	22000	0.1	10	25	5
MCaSr	1.2	22000	0.1	3	25	5
LCaSr	0.2	22000	0.1	0.5	25	5

2/21/2001 cont BAW

Calcium solution with equal concentration of sodium

BW 2/21/01

Soln ID	Target Conc of Ca (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/163 3 ppm Na	Vol (ml) of 309/162 1% LaCl
ECaKNa	0.4	0.6	0.1	1	10	5

BW 2/21/2001

Calcium solution with equal concentration of potassium

Soln ID	Target Conc of Ca (ppm)	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/163 10 ppm K	Vol (ml) of 309/162 1% LaCl
ECaK	0.4	0.4	0.1	1	2	5

Calcium solution with equal concentration of strontium

Soln ID	Target Conc of Ca (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 20 ppm Ca	Vol (mL) of 309/163 25 ppm Sr	Vol (ml) of 309/162 1% LaCl
ECaSr	1.2	1	0.1	3	2	5

2/22/2001 BW

AA analysis of solution for calcium

Perkin Elmer 3100 Atomic Absorbtion Spectrophotometer
Ca-Mg Lamp- 6mV, λ=422.7nm, slit= 0.7nm
Air-acetylene flame
Blank= 0.1% La (w/w) 309/162
Integration time for samples= 35ec

Absorbance of Standards (309/167)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Ca1	4	0.341	0342	0.342	0.342	0.342
Ca2	2	0.175	0.176	0.176	0.177	0.175
Ca3	1.2	0.106	0.105	0.106	0.106	0.106
Ca4	0.8	0.078	0.078	0.078	0.078	0.078
Ca5	0.4	0.035	0.035	0.036	0.035	0.035
Ca6	0.2	0.020	0.020	0.020	0.020	0.020

2/22/2001 cont BW

Absorbance of Samples (309/167, 309/168 + 309/169) BW 2/22/2001

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ECaNa	0.036	0.036	0.036	0.036	0.036
ECaSr	0.108	0.108	0.108	0.108	0.108
ECaK	0.036	0.036	0.036	0.037	0.035
HCaNa	0.277	0.277	0.277	0.278	0.277
MCaNa	0.089	0.089	0.088	0.088	0.088
LCaNa	0.021	0.021	0.021	0.022	0.021
BlkNa	0.008	0.008	0.008	0.008	0.008
Ca3	0.110	0.110	0.111	0.111	0.111
HCaK	0.260	0.258	0.259	0.260	0.260
MCaK	0.081	0.081	0.081	0.081	0.080
LCaK	0.013	0.013	0.013	0.013	0.013
BlkK	-0.000	-0.000	0.000	0.000	-0.000
HCaSr	0.282	0.282	0.281	0.282	0.282
MCaSr	0.156	0.157	0.157	0.156	0.156
LCaSr	0.114	0.113	0.114	0.115	0.115
BlkSr	0.100	0.100	0.100	0.100	0.101
Ca3	0.109	0.109	0.109	0.109	0.109
Ca6	0.020	0.020	0.020	0.020	0.020

Max A/Z correction = 0.004, Normal A/Z correction = 0.000

2/22/2001 BW

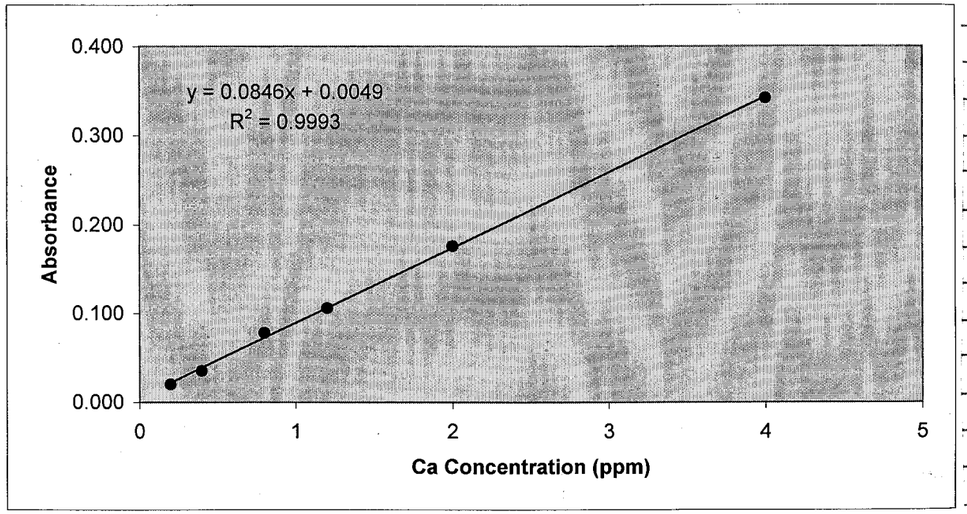
2/22/2001
cont BLW

Calcium Concentration in Various Binary Solutions (309/168 + 169)

Ca Std Data

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
Ca6	0.2	0.020	0.020	0.020	0.020	0.020	0.0200
Ca5	0.4	0.035	0.035	0.036	0.035	0.035	0.0352
Ca4	0.8	0.078	0.078	0.078	0.078	0.078	0.0780
Ca3	1.2	0.106	0.105	0.106	0.106	0.106	0.1058
Ca2	2	0.175	0.176	0.176	0.177	0.175	0.1758
Ca1	4	0.341	0.342	0.342	0.342	0.342	0.3418

Ca Calibration Curve



All calibration verification samples analyzed with the samples were within 5%

BLW 2/23/2001

2/22/2001 BLW

2/22/2001
cont BLW

Calcium Concentration in Various Binary Solutions (309/168 + 169)

Summary Data for equal concentrations of Ca and X

Sample ID	Conc of Ca (ppm)	Conc of cation X	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
ECaNa	0.4	0.6	0.0360	0.36761	8.1
ECaSr	1.2	1	0.1080	1.21868	-1.6
ECaK	0.4	0.4	0.0360	0.36761	8.1

Summary Data for Ca in high concentration of Na

Sample ID	Conc of Ca (ppm)	Conc of cation X	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
HCaNa	4	11500	0.2772	3.21868	19.5
MCaNa	1.2	11500	0.0884	0.98700	17.8
LCaNa	0.2	11500	0.0212	0.19267	3.7
BlkNa	0	11500	0.0080	0.03664	

Summary Data for Ca in high concentration of K

Sample ID	Conc of Ca (ppm)	Conc of K (ppm)	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
HCaNa	4	20000	0.2594	3.00827	24.8
MCaNa	1.2	20000	0.0808	0.89716	25.2
LCaNa	0.2	20000	0.0130	0.09574	52.1
BlkNa	0	20000	0.0000	-0.05792	

Summary Data for Ca in high concentration of Sr

Sample ID	Conc of Ca (ppm)	Conc of Sr (ppm)	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
HCaNa	4	22000	0.2818	3.27305	18.2
MCaNa	1.2	22000	0.1564	1.79078	-49.2
LCaNa	0.2	22000	0.1142	1.29196	-546.0
BlkNa	0	22000	0.1002	1.12648	

BLW 2/22/2001

2/22/2001 BLW

2/23/2001 BW

AA Calcium analysis w/ Strontium present w/ 1% La
Initial Ca analysis (309/167-171) used 0.1% La solns,

20 ppm Ca (Target conc.) Stock soln - labeled Ca Stock
added 2 mL (vol pipet) of 1000 ppm Ca std (Spex
certiprep cat# PLCA2-2X, lot# 7-114CA, open 2/21/2001,
exp 1/31/2002) to a 100 mL vol flask and diluted to
mark with nanopure water.

All final volumes were 50 mL (volumetric flasks)
Volumetric pipets were used to transfer all solutions in the 0.5 mL to 25 mL volume range
High concentrations represent 0.5 N solutions of the cations
% LaCl is w/w

Calcium calibration curve (1% La)

Soln ID	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/172 (20 ppm Ca)	Vol (ml) of 309/162 (10% LaCl)
Ca1*	4	1	10	5
Ca2	2	1	5	5
Ca3	1.2	1	3	5
Ca4	0.8	1	2	5
Ca5	0.4	1	1	5
Ca6	0.2	1	0.5	5

* AA sensitivity check

Calcium solutions with high strontium concentrations (1% La)

Soln ID	Target Conc of Ca (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/172 20 ppm Ca	Vol (mL) of 309/165 44000 ppm Sr	Vol (ml) of 309/162 10% LaCl
HCaSr	4	22000	1	10	25	5
MCaSr	1.2	22000	1	3	25	5
LCaSr	0.2	22000	1	0.5	25	5
BlkSr	0	22000	1	0	25	5

Calcium solution with equal concentration of strontium (1% La)

Soln ID	Target Conc of Ca (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/172 20 ppm Ca	Vol (mL) of 309/163 25 ppm Sr	Vol (ml) of 309/162 10% LaCl
ECaSr	1.2	1	1	3	~~2~~ 25	5

To 2/23/01 BW 2/23/2001

BAW initials to acknowledge page damage 03/06/02

2/23/2001 cont BW

tape removal marks (A)

Perkin Elmer 3100 Atomic Absorption Spectrophotometer BW 2/23/2001
Ca-Mg Lamp - 6 mV, λ = 422.7, slit
Air-acetylene flame
Blank = 1% La (w/w) 309/162
Integration time for samples = 3 sec (A)

Absorbance of Standards (309/172)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Ca1	4	0.255	0.259	0.258	0.257	0.254
Ca2	2	0.143	0.143	0.142	0.143	0.142
Ca3	1.2	0.080	0.081	0.081	0.080	0.080
Ca4	0.8	0.055	0.056	0.055	0.055	0.055
Ca5	0.4	0.028	0.028	0.028	0.027	0.028
Ca6	0.2	0.014	0.015	0.014	0.014	0.015

Absorbance of Samples (309/172)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ECaSr	0.084	0.084	0.084	0.084	0.084
HCaSr	0.222	0.223	0.224	0.223	0.223
MCaSr	0.123	0.124	0.123	0.124	0.123
LCaSr	0.089	0.088	0.088	0.089	0.088
BlkSr	0.080	0.079	0.080	0.080	0.079
Ca3	0.083	0.084	0.083	0.084	0.083
Ca6	0.015	0.015	0.015	0.014	0.015

A/Z max correction = 0.003, normal A2 correction = 0.001
Δ Slit from 0.7 to 0.2 on Ca BW 2-27-2001 no difference in Absorbance
Δ Hi to Low - absorbance dropped some.

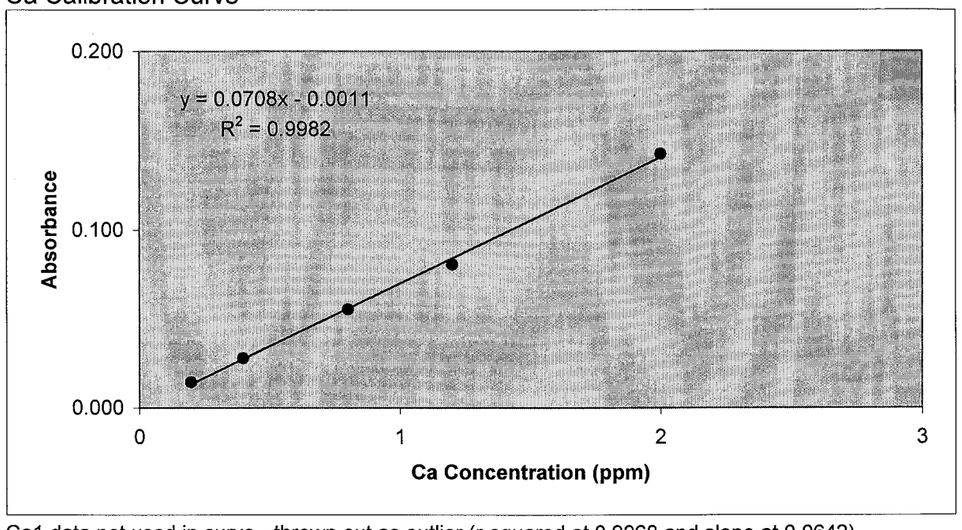
2/26/2001
BAW

Calcium Concentration in Strontium solutions with 1% La (w/w) (309/172)

Ca Std Data

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
Ca6	0.2	0.014	0.015	0.014	0.014	0.015	0.0144
Ca5	0.4	0.028	0.028	0.028	0.027	0.028	0.0278
Ca4	0.8	0.055	0.056	0.055	0.055	0.055	0.0552
Ca3	1.2	0.080	0.081	0.081	0.080	0.080	0.0804
Ca2	2	0.143	0.143	0.142	0.143	0.142	0.1426
Ca1	4	0.255	0.259	0.258	0.257	0.254	0.2566

Ca Calibration Curve



Ca1 data not used in curve - thrown out as outlier (r-squared at 0.9968 and slope at 0.0642)

All calibration verification samples analyzed with the samples were within 5%

BW 2/26/2001

2/26/2001 BW

2/26/2001
BAW cont

Calcium Concentration in Strontium solutions with 1% La (w/w) (309/172)

Summary Data for equal concentrations of Ca and Sr (1% w/w La)

Sample ID	Conc of Ca (ppm)	Conc of cation Sr	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
ECaSr	1.2	1	0.0840	1.20198	-0.2

Summary Data for Ca in high concentration of Sr (1% w/w La)

Sample ID	Conc of Ca (ppm)	Conc of Sr (ppm)	Average Absorbance	Measured Ca Conc (ppm)	Ca conc % difference
HCaNa	4	22000	0.2230	3.16525	20.9
MCaNa	1.2	22000	0.1234	1.75847	-46.5
LCaNa	0.2	22000	0.0884	1.26412	-532.1
BlkNa	0	22000	0.0796	1.13983	

BW 2/26/2001

Lanthanum soln for AA analysis

Reagents - Lanthanum Chloride, $LaCl_3 \cdot 7H_2O$, Fisher
cat# L9-250, lot# 985153A
- nanopure water

Target concentration 10% (w/w) La

$$36.5\text{ g } LaCl_3 \cdot 7H_2O \left(\frac{138.91\text{ g La/mol}}{371.374\text{ g } LaCl_3 \cdot 7H_2O\text{/mol}} \right) \Big/ (100\text{ g } H_2O + 36.5\text{ g } LaCl_3 \cdot 7H_2O)$$

BW 2/26/2001

mass of beaker (g)	mass of beaker + H_2O (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
30.12	130.12	166.66

2/26/2001 cont BWS

Strontium Solutions For AA Analysis

All final volumes were 50mL (vol flask)
Vol pipets used to transfer solns in 0.5mL - 25mL range
High conc represent 0.5N soln of the cation
%LaCl is w/w

Strontium calibration curve

Soln ID	Target Conc of Sr (ppm)	Target Conc of LaCl (%)		Vol (mL) of 309/163 (25 ppm Sr)	Vol (ml) of 309/175 (10% LaCl)
Sr1*	5	1		10	5
Sr2	2.5	1		5	5
Sr3	1.5	1		3	5
Sr4	1	1		2	5
Sr5	0.5	1		1	5
Sr6	0.25	1		0.5	5

* AA sensitivity check

Strontium solutions with high potassium concentrations

Soln ID	Target Conc of Sr (ppm)	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/165 40000 ppm K	Vol (ml) of 309/175 (10% LaCl)
HSrK	5	20000	1	10	25	5
MSrK	1.5	20000	1	3	25	5
LSrK	0.5	20000	1	1	25	5

Strontium solutions with high sodium concentrations

Soln ID	Target Conc of Sr (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/164 23000 ppm Na	Vol (ml) of 309/175 (10% LaCl)
HSrNa	5	11500	1	10	25	5
MSrNa	1.5	11500	1	3	25	5
LSrNa	0.5	11500	1	1	25	5

Strontium solutions with high calcium concentrations

Soln ID	Target Conc of Sr (ppm)	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/164 20000 ppm Ca	Vol (ml) of 309/175 (10% LaCl)
HSrCa	5	10000	1	10	25	5
MSrCa	1.5	10000	1	3	25	5
LSrCa	0.5	10000	1	1	25	5

BW 2/26/2001

2/26/2001 cont BW

BW 2/26/2001

Strontium solution with equal concentration of sodium

Soln ID	Target Conc of Sr (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/163 3 ppm Na	Vol (ml) of 309/175 (10% LaCl)
ESrNa	0.5	0.6	1	1	10	5

Strontium solution with equal concentration of potassium

Soln ID	Target Conc of Sr (ppm)	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/163 10 ppm K	Vol (ml) of 309/175 (10% LaCl)
ESrK	1	1	1	2	5	5

Strontium solution with equal concentration of calcium

Soln ID	Target Conc of Sr (ppm)	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (25 ppm Sr)	Vol (mL) of 309/172 20 ppm Ca	Vol (ml) of 309/175 (10% LaCl)
ESrCa	1	1.2	1	2	3	5

High calcium concentration blank for strontium analysis

Soln ID	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/164 20000 ppm Ca	Vol (ml) of 309/175 10% LaCl
SrBlkCa	10000	1	25	5

BW 2/26/2001

High sodium concentration blank for strontium analysis

Soln ID	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/164 23000 ppm Na	Vol (ml) of 309/175 10% LaCl
SrBlkNa	11500	1	25	5

High potassium concentration blank for strontium analysis

Soln ID	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/165 40000 ppm K	Vol (ml) of 309/175 10% LaCl
SrBlkK	20000	1	25	5

BW 2/26/2001

2/28/2001
BAW

AA Analysis of Strontium solutions in various cation mixtures

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
Sr Hollow cathode lamp = 12mA Fisher 14-388-106W
λ = 460.7nm, slit = 0.7nm, Air-acetylene flame
Blank = 1% La_2O_3 (w/w) 309/162
Integration time for samples = 3 sec

Absorbance of Standards (309/176)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Sr1	5	0.262	0.261	0.263	0.261	0.261
Sr2	2.5	0.133	0.131	0.132	0.132	0.131
Sr3	1.5	0.080	0.081	0.081	0.080	0.080
Sr4	1.0	0.053	0.054	0.054	0.054	0.053
Sr5	0.5	0.026	0.026	0.026	0.026	0.026
Sr6	0.25	0.013	0.013	0.012	0.013	0.012

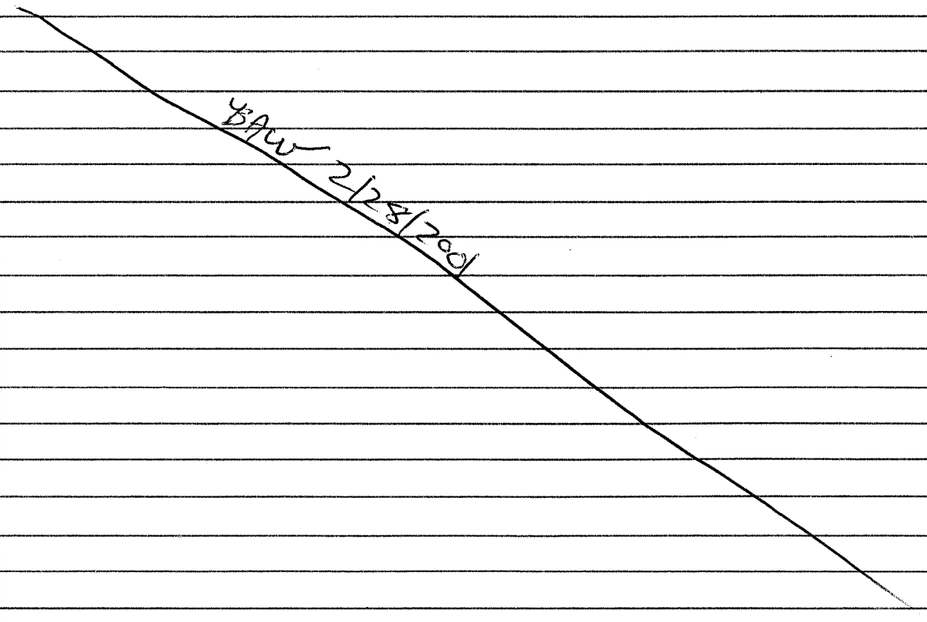
Absorbance of Samples (309/176+177)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ESrK	0.052	0.053	0.052	0.052	0.053
ESrNa	0.026	0.026	0.026	0.026	0.026
ESrCa	0.052	0.052	0.052	0.052	0.052
HSrK	0.198	0.199	0.198	0.199	0.198
MSrK	0.060	0.060	0.060	0.060	0.060
LSrK	0.020	0.019	0.020	0.019	0.020
SrBlkK	-0.000	-0.000	-0.000	-0.000	-0.000
Sr3	0.079	0.079	0.078	0.079	0.079

2/28/2001
last BAW

Sample ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
HSrNa	0.199	0.199	0.199	0.200	0.199
MSrNa	0.062	0.061	0.062	0.062	0.061
LSrNa	0.020	0.020	0.020	0.020	0.020
SrBlkNa	0.000	0.000	0.000	0.000	0.000
HSrCa	0.301	0.301	0.301	0.302	0.302
MSrCa	0.202	0.202	0.201	0.202	0.203
LSrCa	0.174	0.173	0.172	0.173	0.173
SrBlkCa	0.159	0.158	0.159	0.158	0.158
Sr3	0.077	0.077	0.077	0.077	0077
Sr6	0.013	0.012	0.013	0.013	0.013

AZ max correction = 0.001, normal AZ correction = 0.000
Solid formed on burner head and caused flame to become "non continuous". Burner cleaned after analysis
All CCV's within 5% of target conc.



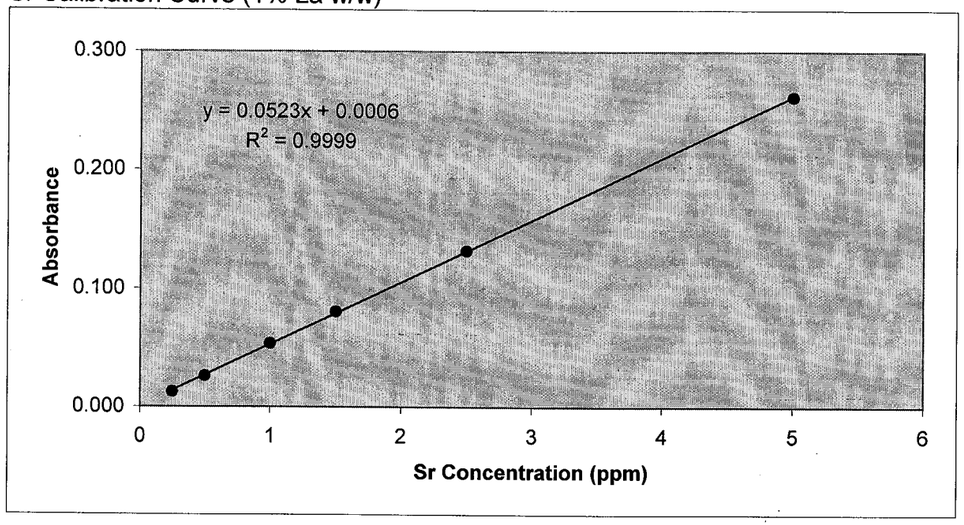
3/1/2001 BAW

Strontium Concentration in Various Binary Solutions (309/176 + 177)

Sr Std Data (1% La w/w)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
Sr1	0.25	0.013	0.013	0.012	0.013	0.012	0.0126
Sr2	0.5	0.026	0.026	0.026	0.026	0.026	0.0260
Sr3	1	0.053	0.054	0.054	0.054	0.053	0.0536
Sr4	1.5	0.080	0.081	0.081	0.080	0.080	0.0804
Sr5	2.5	0.133	0.131	0.132	0.132	0.131	0.1318
Sr6	5	0.262	0.261	0.263	0.261	0.261	0.2616

Sr Calibration Curve (1% La w/w)



All calibration verification samples analyzed with the samples were within 5% BW 3/1/2001

3/1/2001 BW

3/1/2001 cont BW

Strontium Concentration in Various Binary Solutions (309/176 + 177)

Summary Data for equal concentrations of Sr and X

Sample ID	Conc of Sr (ppm)	Conc of cation X	Average Absorbance	Measured Sr Conc (ppm)	Sr conc % difference
ESrNa	0.5	0.6	0.0260	0.48566	2.9
ESrK	1	1	0.0524	0.99044	1.0
ESrCa	1	1.2	0.0520	0.98279	1.7

Summary Data for Sr in high concentration of Na

Sample ID	Conc of Sr (ppm)	Conc of Na (ppm)	Average Absorbance	Measured Sr Conc (ppm)	Sr conc % difference
HSrNa	5	11500	0.1992	3.79732	24.1
MSrNa	1.5	11500	0.0616	1.16635	22.2
LSrNa	0.5	11500	0.0200	0.37094	25.8
SrBlkNa	0	11500	0.0000	-0.01147	

Summary Data for Sr in high concentration of K

Sample ID	Conc of Sr (ppm)	Conc of K (ppm)	Average Absorbance	Measured Sr Conc (ppm)	Sr conc % difference
HSrK	5	20000	0.1984	3.78203	24.4
MSrK	1.5	20000	0.0600	1.13576	24.3
LSrK	0.5	20000	0.0196	0.36329	27.3
SrBlkK	0	20000	0.0000	-0.01147	

Summary Data for Sr in high concentration of Ca

Sample ID	Conc of Sr (ppm)	Conc of Ca (ppm)	Average Absorbance	Measured Sr Conc (ppm)	Sr conc % difference
HSrCa	5	10000	0.3014	5.75143	-15.0
MSrCa	1.5	10000	0.2020	3.85086	-156.7
LSrCa	0.5	10000	0.1730	3.29637	-559.3
SrBlkCa	0	10000	0.1584	3.01721	

BW 3/1/2001

3/1/2001 BW

3/2/2001
BAW

Sodium Solutions for AA Analysis

All final volumes were 50 mL (volumetric flasks)
Volumetric pipets were used to transfer all solutions in the 0.5 mL to 25 mL volume range
High concentrations represent 0.5 N solutions of the cations
% LaCl is w/w

Sodium calibration curve

Soln ID	Target Conc of Na (ppm)	Target Conc of LaCl (%)		Vol (mL) of 309/163 (3 ppm Na)	Vol (ml) of 309/183 (1% LaCl)
Na1	0.6	0.1		10	5
Na2	0.3	0.1		5	5
Na3	0.18	0.1		3	5
Na4	0.12	0.1		2	5
Na5	0.06	0.1		1	5
Na6	0.03	0.1		0.5	5

* AA sensitivity check 0.5ppm

Sodium solutions with high calcium concentrations

Soln ID	Target Conc of Na (ppm)	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (3 ppm Na)	Vol (mL) of 309/164 20000 ppm Ca	Vol (ml) of 309/183 1% LaCl
HNaCa	0.6	10000	0.1	10	BW ~~25~~ 25	5
MNaCa	0.12	10000	0.1	2 2/27/02	25	5
LNaCa	0.03	10000	0.1	0.5	25	5

Sodium solutions with high potassium concentrations

Soln ID	Target Conc of Na (ppm)	Target Conc of K (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (3 ppm Na)	Vol (mL) of 309/165 40000 ppm K	Vol (ml) of 309/183 1% LaCl
HNaK	0.6	20000	0.1	10	25	5
MNaK	0.12	20000	0.1	2	25	5
LNaK	0.03	20000	0.1	0.5	25	5

Sodium solutions with high stronium concentrations

Soln ID	Target Conc of Na (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (3 ppm Na)	Vol (mL) of 309/165 44000 ppm Sr	Vol (ml) of 309/183 1% LaCl
HNaSr	0.6	22000	0.1	10	25	5
MNaSr	0.12	22000	0.1	2	25	5
LNaSr	0.03	22000	0.1	0.5	25	5

BW
3/2/2001

3/2/2001
cont BAW

Sodium solution with equal concentration of strontium

Soln ID	Target Conc of Na (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (3 ppm Na)	Vol (mL) of 309/163 25 ppm Sr	Vol (ml) of 309/183 1% LaCl
ENaSr	0.6	0.5	0.1	10	1	5

Potassium solution with equal concentration of sodium

Soln ID	Target Conc of K (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (10 ppm K)	Vol (mL) of 309/163 3 ppm Na	Vol (ml) of 309/183 1% LaCl
EKNa	0.6	0.6	0.1	3	10	5

Target conc. 1% La (w/w)

$8.52\text{g } LaCl_3 \cdot 7H_2O \left(\frac{138.91\text{ g La/mol}}{371.374\text{ g } LaCl_3 \cdot 7H_2O\text{/mol}}\right)$

$(310\text{g } H_2O + 8.52\text{g } LaCl_3 \cdot 7H_2O)$

Balance = Mettler PM 4600
Reagents = Lanthanum Chloride, $LaCl_3 \cdot 7H_2O$ - Fisher
cat# L9-250, lot# 985153A
- nanopure water

mass of beaker (g)	mass of beaker + H_2O (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
56.71	366.71	375.23

AA Analysis of Sodium Solutions in various cation mixtures

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
Na Hollow Cathode Lamp @ 8 mA, Fisher 14-386-106 S
λ = 589.0 nm, slit = 0.7 nm, Air-acetylene flame, "high"
Blank = 0.1 % La (309/162 soln C)
Integration time for samples

3/2/2001
cont BAW

Absorbance of Standards (309/182)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Na1	0.6	0.139	0.140	0.140	0.140	0.141
Na2	0.3	0.067	0.065	0.066	0.066	0.066
Na3	0.18	0.042	0.043	0.043	0.043	0.042
Na4	0.12	0.031	0.031	0.028	0.029	0.029
Na5	0.06	0.015	0.015	0.015	0.015	0.015
Na6	0.03	0.008	0.008	0.008	0.008	0.008

Absorbance of Samples (309/182, 183, 166, 168)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ECaNa	0.134	0.133	0.133	0.133	0.133
EKNa	0.135	0.135	0.136	0.135	0.137
ENaSr	0.132	0.133	0.132	0.132	0.131
HNaCa	0.240	0.237	0.238	0.239	0.240
MNaCa	0.155	0.156	0.154	0.156	0.156
LNaCa	0.145	0.144	0.144	0.145	0.145
BlkCa	0.134	0.136	0.135	0.135	0.136
Na3	0.041	0.042	0.042	0.041	0.041
HNaK	0.225	0.225	0.227	0.226	0.225
MNaK	0.135	0.134	0.134	0.133	0.134
LNaK	0.130	0.131	0.130	0.131	0.130
BlkK	0.103	0.103	0.104	0.105	0.105
HNaSr	0.981	0.970	0.973	0.976	0.977
MNaSr	0.939	0.932	0.937	0.937	0.946
LNaSr	0.927	0.929	0.931	0.933	0.929
BlkSr	0.924	0.927	0.919	0.925	0.918
Na3	0.042	0.042	0.041	0.041	0.044

3/2/2001
cont BAW

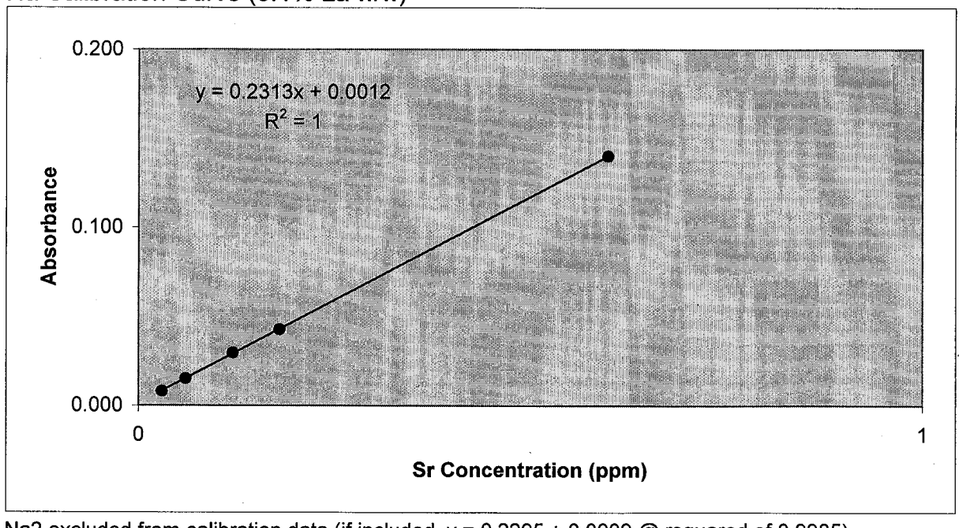
Analyzed SrBlkK and SrBlkNa (309/177) for verification of high level metal (0.5N)
- Verified- K was lavender and Na was orange
#3 max correction = 0.066 Normal = 0.002

Sodium Concentration in Various Binary Solutions (309/182 & 183)

Na Standard Data (0.1% La w/w)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
Na1	0.6	0.139	0.140	0.140	0.140	0.141	0.1400
Na2	0.3	0.067	0.065	0.066	0.066	0.066	0.0660
Na3	0.18	0.042	0.043	0.043	0.043	0.042	0.0426
Na4	0.12	0.031	0.031	0.028	0.029	0.029	0.0296
Na5	0.06	0.015	0.015	0.015	0.015	0.015	0.0150
Na6	0.03	0.008	0.008	0.008	0.008	0.008	0.0080

Na Calibration Curve (0.1% La w/w)



Na2 excluded from calibration data (if included, y = 0.2295 + 0.0009 @ rsquared of 0.9985)

All calibration verification samples analyzed with the samples were within 5% BW 3/2/2001

3/2/2001
cont BAW

Sodium Concentration in Various Binary Solutions (309/182 & 183)

Summary Data for equal concentrations of Na and X

Sample ID	Conc of Na (ppm)	Conc of cation X	Average Absorbance	Measured Na Conc (ppm)	Na conc % difference
ENaSr	0.6	0.5	0.1320	0.56550	5.8
EKNa	0.6	0.6	0.1356	0.58106	3.2
ECaNa	0.6	0.4	0.1332	0.57069	4.9

Summary Data for Na in high concentration of Sr

Sample ID	Conc of Na (ppm)	Conc of Sr (ppm)	Average Absorbance	Measured Na Conc (ppm)	Na conc % difference
HNaSr	0.6	22000	0.9754	4.21185	-602
MNaSr	0.12	22000	0.9382	4.05102	-3276
LNaSr	0.03	22000	0.9298	4.01470	-13282
BlkSr	0	22000	0.9226	3.98357	

Summary Data for Na in high concentration of K

Sample ID	Conc of Na (ppm)	Conc of K (ppm)	Average Absorbance	Measured Na Conc (ppm)	Na conc % difference
HNaK	0.6	20000	0.2256	0.97017	-62
MNaK	0.12	20000	0.1340	0.57415	-378
LNaK	0.03	20000	0.1304	0.55858	-1762
BlkK	0	20000	0.1036	0.44272	

Summary Data for Na in high concentration of Ca

Sample ID	Conc of Na (ppm)	Conc of Ca (ppm)	Average Absorbance	Measured Na Conc (ppm)	Na conc % difference
HNaCa	0.6	10000	0.2388	1.02724	-71
MNaCa	0.12	10000	0.1554	0.66667	-456
LNaCa	0.03	10000	0.1446	0.61997	-1967
BlkCa	0	10000	0.1352	0.57933	

BW
3/2/2001

BW 3/2/2001

Potassium

3/6/2001 BW 3/6/01
BAW Section Solutions for AA Analysis

All final volumes were 50 mL (volumetric flasks)
Volumetric pipets were used to transfer all solutions in the 0.5 mL to 25 mL volume range
High concentrations represent 0.5 N solutions of the cations
% LaCl is w/w

Potassium calibration curve

Soln ID	Target Conc of k (ppm)	Target Conc of LaCl (%)		Vol (mL) of 309/163 (10 ppm K)	Vol (ml) of 309/183 (1% LaCl)
K1*	2	0.1		10	5
K2	1	0.1		5	5
K3	0.6	0.1		3	5
K4	0.4	0.1		2	5
K5	0.2	0.1		1	5
K6	0.1	0.1		0.5	5

* AA sensitivity check

Potassium solutions with high calcium concentrations

Soln ID	Target Conc of K (ppm)	Target Conc of Ca (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (10 ppm K)	Vol (mL) of 309/164 20000 ppm Ca	Vol (ml) of 309/183 1% LaCl
HKCa	2	10000	0.1	10	25	5
MKCa	0.6	10000	0.1	3	25	5
LKCa	0.1	10000	0.1	0.5	25	5

Potassium solutions with high sodium concentrations

Soln ID	Target Conc of K (ppm)	Target Conc of Na (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (10 ppm K)	Vol (mL) of 309/164 23000 ppm Na	Vol (ml) of 309/183 1% LaCl
HKNa	2	11500	0.1	10	25	5
MKNa	0.6	11500	0.1	3	25	5
LKNa	0.1	11500	0.1	0.5	25	5

Potassium solutions with high stronium concentrations

Soln ID	Target Conc of K (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (10 ppm K)	Vol (mL) of 309/165 44000 ppm Sr	Vol (ml) of 309/183 1% LaCl
HKSr	2	22000	0.1	10	25	5
MKSr	0.6	22000	0.1	3	25	5
LKSr	0.1	22000	0.1	0.5	25	5

BW 3/6/2001

3/6/2001 cont BW

Potassium solution with equal concentration of strontium

Soln ID	Target Conc of K (ppm)	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 309/163 (10 ppm K)	Vol (mL) of 309/163 25 ppm Sr	Vol (ml) of 309/183 1% LaCl
EKSr	1	1	0.1	5	2	5

BW 3/6/2001

- AA analysis of Potassium solutions in various cation mixtures

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
K Hollow Cathode Lamp - 8mA Fisher 14-386-106H
λ = 766.5nm, slit = 0.7nm, air-acetylene flame, high
Blank = 0.1% La (w/w) 309/162
Integration time for samples = 3 sec

Absorbance of Standards (309/187+188)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
K1	2	0.200	0.196	0.196	0.199	0.199
K2	1	0.100	0.096	0.098	0.101	0.099 ~~0.094~~ BW 2/27/02
K3	0.6	0.061	0.065	0.066	0.060	0.064
K4	0.4	0.041	0.041	0.041	0.043	0.042
K5	0.2	0.018	0.018	0.018	0.018	0.018
K6	0.1	0.198				

Absorbance of Samples (309/187+188)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ECaK					
EKNa					

BW 3/6/2001

3/7/~~2001~~ ~~2000~~
BW 3/7/2001
BAW

3/6/2001 cont BAW

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
EKSr					
HKCa					
MKCa					
LKCa					
BIKCa					
K					
HKNa					
MKNa					
LKNa					
BIKNa					
HKSr					
MKSr					
LKSr					
BIKSr					
K					

BW 3/6/2001

Analysis stopped. Absorbance values fluctuated unsteadily over ~ 0.015 for a measured value. Cleaned burner w/ water + ensured drain line flowing properly.

Copy of notebook made for QA archives

Target Conc. 0.1% La (w/w)
Balance = Mettler PM4600
Reagents - Lanthanum Chloride, $LaCl_3 \cdot 7H_2O$ - Fisher
cat# L9-250, lot# 985153A
- nanopure water

3/7/2001 cont BAW

$1.34\,g\ LaCl_3 \cdot 7H_2O \left(\frac{138.91\ g\ La/mol}{371.374\ g\ LaCl_3 \cdot 7H_2O/mol}\right) \times 100 = 0.1000$
$(500\,g\ H_2O + 1.34\,g\ LaCl_3 \cdot 7H_2O)$

mass of beaker (g)	mass of beaker + water (g)	mass of beaker + H_2O + $LaCl_3 \cdot 7H_2O$ (g)
57.19	557.19	558.53

3/8/2001 BAW

Re-attempt at Potassium analysis of 3/6/2001.

Perkin Elmer 3100 Atomic Adsorption Spectrophotometer
K - Hollow cathode lamp 8mA Fisher 14-386-106H
λ = 766.5 nm, slit = 0.7 nm, high, Air-acetylene flame
Blank = 0.1% La (w/w) 309/190
Integration time for samples = 3 sec

Absorbance of Standards (309/187+188)

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
K1	2	0.242	0.241	0.240	240	0.240
K2	1	0.123	0.123	0.123	0.123	0.123
K3	0.6	0.074	0.074	0.075	0.074	0.075
K4	0.4	0.048	0.048	0.048	0.048	0.048
K5	0.2	0.024	0.024	0.024	0.024	0.024
K6	0.1	0.011	0.011	0.011	0.011	0.012

Absorbance of Samples (309/187+188)

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
ECaK	0.051	0.050	0.050	0.051	0.050
EKNa	0.074	0.075	0.075	0.075	0.075

3/8/2001 cont BAW

ID	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
EKSr	0.123	0.124	0.124	0.123	0.123
HKCa	0.232	0.232	0.233	0.234	0.234
MKCa	0.077	0.077	0.077	0.077	0.076
LKCa	0.019	0.019	0.019	0.019	0.018
BIKCa	0.010	0.009	0.009	0.008	0.009
K3	0.073	0.073	0.073	0.073	0.073
HKNa	0.355	0.355	0.354	0.354	~~0.03~~ 0.355 BWS 3/8/2001
MKNa	0.159	0.158	0.159	0.157	0.158
LKNa	0.151	0.151	0.151	0.154	0.153
BIKNa	0.068	0.068	0.068	0.068	0.067
HKSr	0.290	0.294	0.292	0.292	0.292
MKSr	0.110	0.110	0.110	0.110	0.110
LKSr	0.034	0.034	0.034	0.034	0.034
BIKSr	0.028	0.028	0.028	0.028	0.028
K2	0.131	0.131	0.131	0.132	0.132
K2	0.129	0.129	0.129	0.129	0.128
K6	0.011	0.011	0.011	0.011	0.011

aspirated 1% HNO_3 solution for 5 minutes then began analysis of

AZ max correction = 0.008, AZ normal correction = 0.002
ALL calibration checks within 7%

Aspirated 1% HNO_3 solution for 5 minutes after all analysis completed.

Small "fluctuations" in values started with high Na conc.

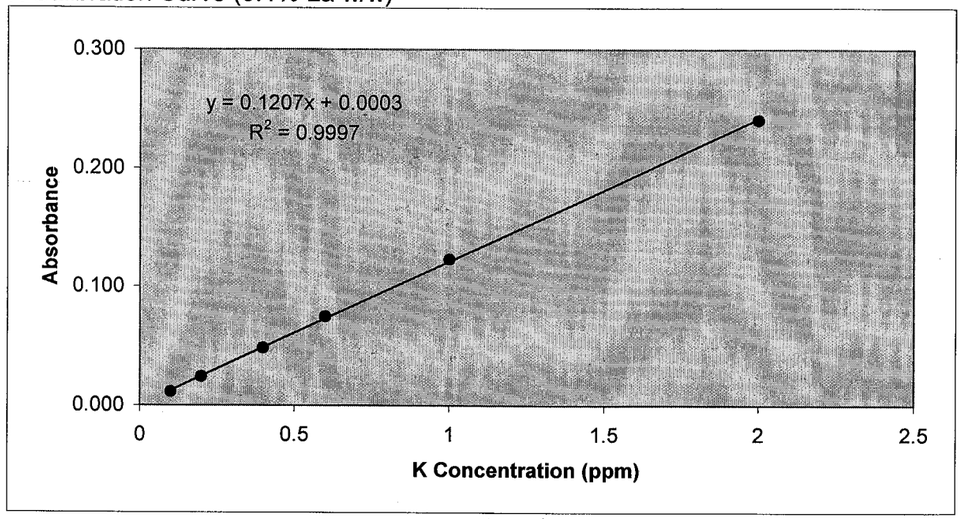
3/12/2001 BAW

Potassium Concentration in Various Binary Solutions (309/187 & 188)

K Standard Data (0.1% La w/w)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
K1	2	0.242	0.241	0.240	0.240	0.240	0.2406
K2	1	0.123	0.123	0.123	0.123	0.123	0.1230
K3	0.6	0.074	0.074	0.075	0.074	0.075	0.0744
K4	0.4	0.048	0.048	0.048	0.048	0.048	0.0480
K5	0.2	0.024	0.024	0.024	0.024	0.024	0.0240
K6	0.1	0.011	0.011	0.011	0.011	0.012	0.0112

K Calibration Curve (0.1% La w/w)



All calibration verification samples analyzed with the samples were within 7%

3/12/2001 BW

BW 3/12/2001

3/12/2001 (cont) BAW

Potassium Concentration in Various Binary Solutions (309/187 & 188)

Summary Data for equal concentrations of K and X

Sample ID	Conc of K (ppm)	Conc of cation X	Average Absorbance	Measured K Conc (ppm)	K conc % difference
EKCa	0.4	0.4	0.0504	0.41508	-3.8
EKNa	0.6	0.6	0.0748	0.61723	-2.9
EKSr	1.0	1.0	0.1234	1.01988	-2.0

Summary Data for K in high concentration of Sr

Sample ID	Conc of K (ppm)	Conc of Sr (ppm)	Average Absorbance	Measured K Conc (ppm)	K conc % difference
HKSr	2.0	22000	0.2920	2.41674	-21
MKSr	0.6	22000	0.1100	0.90886	-51
LKSr	0.1	22000	0.0340	0.27920	-179
BlkSr	0	22000	0.0280	0.22949	

Summary Data for K in high concentration of Na

Sample ID	Conc of K (ppm)	Conc of Na (ppm)	Average Absorbance	Measured K Conc (ppm)	K conc % difference
HKNa	2.0	11500	0.3546	2.93538	-47
MKNa	0.6	11500	0.1582	1.30820	-118
LKNa	0.1	11500	0.1520	1.25684	-1157
BlkK	0	11500	0.0678	0.55924	

Summary Data for K in high concentration of Ca

Sample ID	Conc of K (ppm)	Conc of Ca (ppm)	Average Absorbance	Measured K Conc (ppm)	K conc % difference
HKCa	2.0	10000	0.2330	1.92792	4
MKCa	0.6	10000	0.0768	0.63380	-6
LKCa	0.1	10000	0.0188	0.15327	-53
BlkCa	0	10000	0.0090	0.07208	

BW 3/12/2001

3/12/01 gp

Petrographic analyses of Nye County EWDP well cutting samples.

Obj. 03/08/02 PB

~~Obj~~ - describe mineralogic components of thin sections of samples collected from wells drilled for the EWDP.

Method - petrographic analysis

Equipment and materials
- Nikon Optiphot Pol microscope with 35 mm camera attachment.
- thin sections of Nye County EWDP samples (see p 100 for list of samples).

Following are descriptions and observations about the mineralogic and textural components of thin sections of samples from the Nye County EWDP. 35 mm slides of thin sections are stored in a 3-ring binder entitled "Thin section photomicrographs of Nye County EWDP Samples." The slides are labeled with the thin section number along with the horizontal field of view scale (eg., 578 2.6mm). Digital images of selected 35 mm slides are included in the following descriptions for illustrative purposes.

The materials from the Nye County EWDP are well cuttings of alluvium and tuff. The alluvium is epiclastic or sedimentary in nature (i.e., it was originally formed at the surface by consolidation of fragments of preexisting rocks [eg. sandstone]). The well cuttings are disturbed samples of these original sedimentary rocks. The samples have disaggregated during the drilling process and in most cases the cements holding rock fragments together have been totally or partially removed.

ok 03/08/02 PB

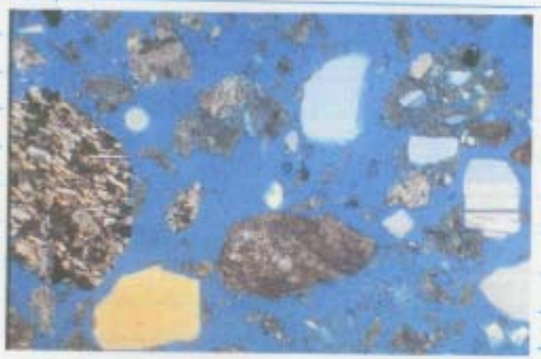
Thin Section No.

578 - Components included volcanic rock fragments (VRFs), quartz, and Feldspar.

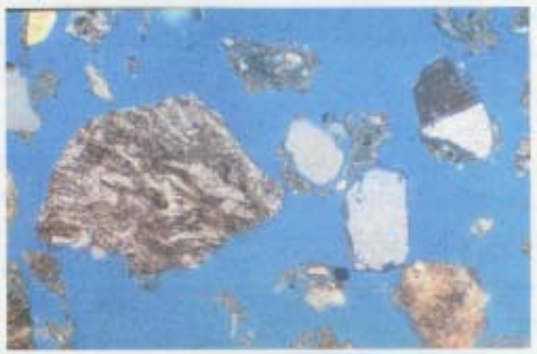
VRFs are texturally varied: aphanitic to porphyritic, pumiceous, and variolitic. Some VRFs have a trachytic fabric where microlites of feldspar composing the groundmass are disposed in a subparallel manner due to flow of magma. Quartz and Feldspar grains are predominately volcanic in origin and are often rimmed by cements or remnants of an aphanitic groundmass. Also present are clastic particles (VRFs, quartz, ± Feldspar) cemented by silt-sized particles (predominantly feldspar). These clastic particles are examples of what the alluvium looked like before disruption by drilling.

578 cont

Sample 578 (2.6mm cross) - VRFs with porphyritic texture - phenocrysts in aphanitic groundmass.



Sample 578 (2.6mm)
Numerous silt and Feldspar particles. Also a clastic fragment at top right and a fragment with variolitic texture at left (Feldspar laths in glassy matrix)

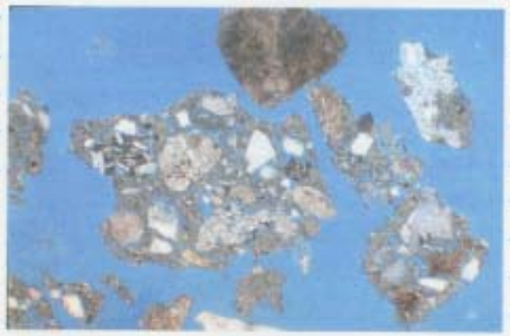


Sample 578 (2.6mm)

Quartz + Feldspar particles VRF with pumiceous fabric at left center.

584

Compositional thin sample is very similar to 578. VRFs, quartz, and Feldspar are predominate components. Thin sample however contains more numerous clastic fragments.



Sample 584 (2.6 mm)

Both photos show example of clastic fragments - VRFs, quartz, and Feldspar dominated by fine silt-sized Feldspar particles.

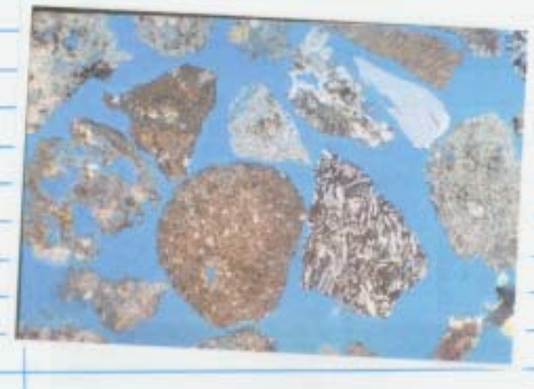
649 - Major composite grains include VRFs, quartz, & feldspar. Clasts & vesicles are less common. XRD indicates the presence of a possible zeolite (clinoptilolite) which is a alteration product of glass. This sample contains a few fragments of composite quartz which appear to be plutonic in origin.

Sample 649 (2.6mm) -

Top photo show patch composed of equigranular quartz.

Sample contains mostly VRFs with aphanitic texture. Other VRF texture include porphyritic and vesicular.

650- VRFs, quartz, & feldspar are major components. VRFs predominate and have varied textures like sample 649, this sample may contain zeolites as indicated by XRD.



gp 3/2/01

Sample ~~649~~ 650 (2.6mm)

These photos show VRFs with variable textures.

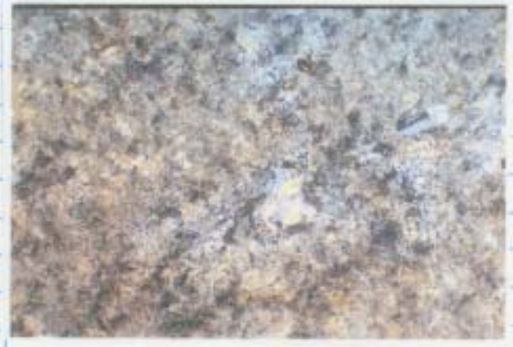
654AT Single volcanic rock fragment. Composed of phenocrysts of quartz and Feldspar in an aphanitic (Feldspar) groundmass. Groundmass has a pumiceous texture (ghosts of glass shards are replaced by cristobalite).

Sample 654AT (2.6mm)

Qtz phenocryst in felty groundmass

Sample 654AT (1.0mm) Ghosts of glass shards depicted by cristobalite.

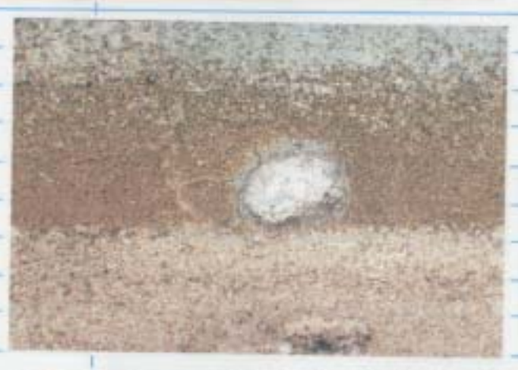
656AT - Volcanic rock fragment. Feldspar phenocrysts (minor) in an aphanitic groundmass. Cristobalite replaces glass shards in groundmass.



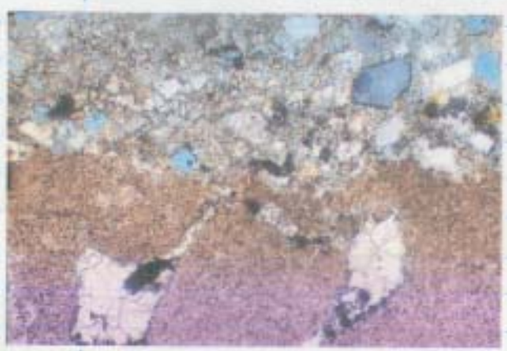
656AT (2.6mm)

656 BT - Appears to be a layered sedimentary rock fragment composed of coarse to fine sandstone cemented by calcite and/or dolomite. Finer layers could be considered as siltstone. Often spaces and voids are filled by spurry calcite and/or dolomite.

656 BT 2.6 mm
Layered SRF composed of sandstone and siltstone. Finer layer of cement has been altered to dolomite.

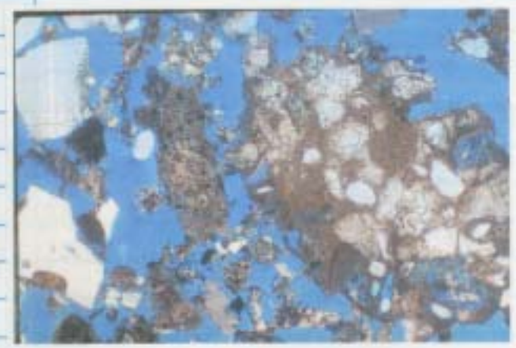


656 BT 2.6 mm
Vug in siltstone filled by coarse calcite/dolomite.

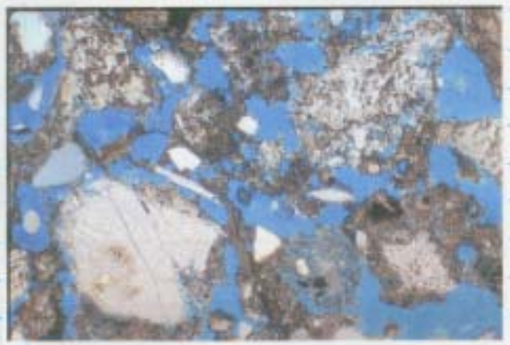


656 BT 1.0 mm
Contact between layers with vugs filled by calcite/dolomite.

657 - Composed of ~~particles~~ particles 03/03/12 TB of quartz, FLP, VRFs, and clastic fragments to be fine matrix or cement. Voids in VRFs are filled by calcite/dolomite. Cement may also be replaced by dolomite, as are some of the VRFs.

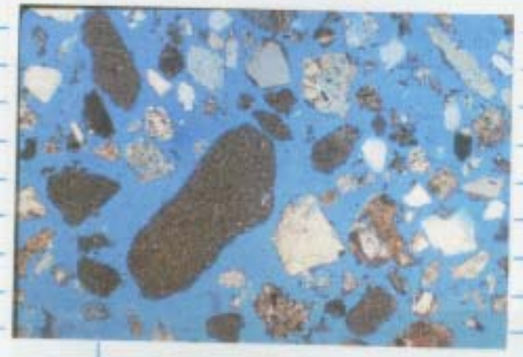


657 (2.6 mm)
VRF replaced by calcite/dolomite.



657 (2.6 mm)
Particles cemented by what appear to be dolomite.

661 - Composed of VRFs, quartz, + feldspar. The sample also contains numerous dolomite fragments which occur as dark reddish brown fine crystalline particles. Dolomite is also present as a feldspar replacement. A few coarse crystalline calcite fragments are also present.



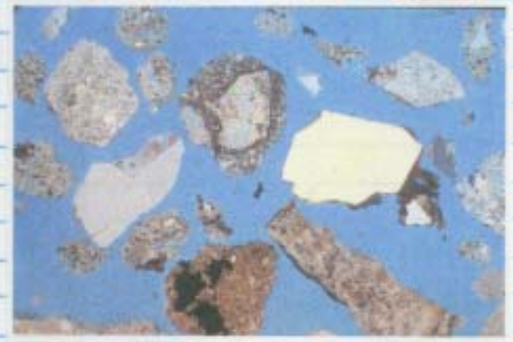
Sample 661 (2.6mm)

Photo shows numerous dolomite fragments which are dark-colored and fine crystalline.

Sample 661 (1.0mm)

Fragment to right is dolomite. The particle at left is feldspar being replaced by dolomite.

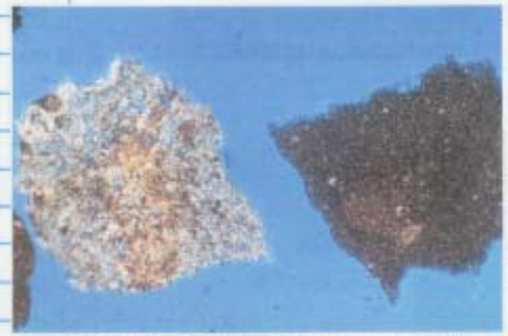
664 Similar to sample 661 (previous page) but contains fewer dolomite fragments. Some of the VRFs appear to be undergoing replacement by dolomite - i.e. feldspar groundmass undergoing alteration to dolomite, and feldspar phenocrysts replaced by spurry calcite/dolomite.



Sample 664 (2.6mm)
Top 2 photos show VRFs, quartz, feldspar, and dolomite fragments.

Sample 664 (1.0mm)
VRF being replaced by dolomite.

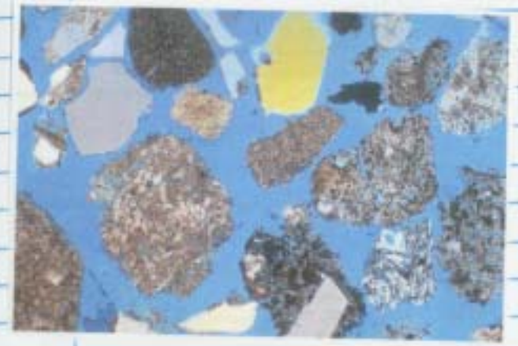
Sample 665 Sample is composed of VRFs and Dolomite particles. Many of the VRFs are undergoing replacement by Dolomite.



665 2.6mm
VRF adjacent to Dolomite particle

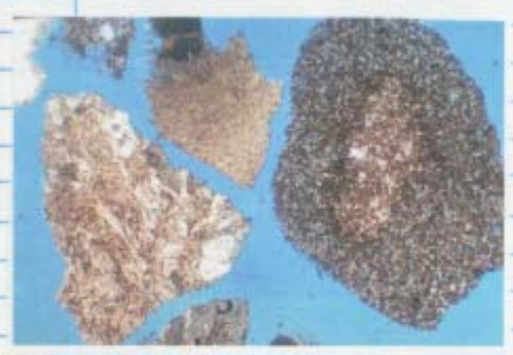
665 1.0mm
This photo shows Dolomite replacement of a VRF.

666 Composed of VRFs, quartz, feldspar, & dolomite & evaporite. Some replacement of groundmass of VRFs by dolomite. VRFs are again texturally varied.



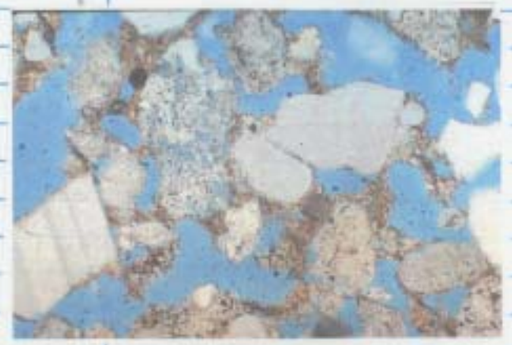
666 2.6mm

Photos of VRFs showing a variety of textures; aphanitic, vuggy, porphyritic, porcellanous.



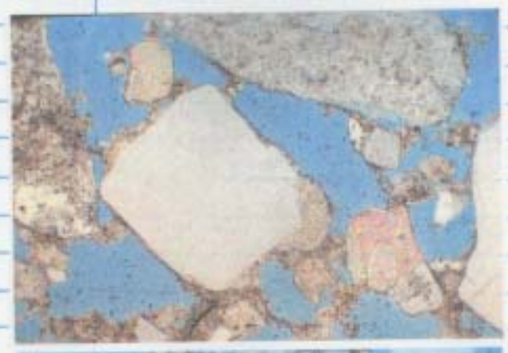
Bottom photo shows VRF with equigranular type texture.

684 Composed of VRFs, quartz, feldspar, carbonate fragments cemented by calcite. Cement is not well developed and thus the rock is very porous looking.



684 (2.6mm)

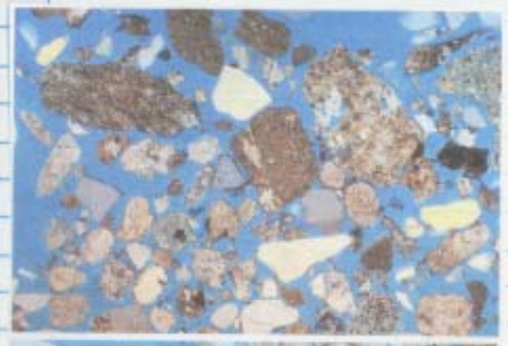
Photo of VRFs, quartz, + feldspar particles with calcite cement.



684 (1.0 mm)

Both these photos show grains cemented by calcite.

690 Composed of VRFs, quartz, feldspar, dolomite fragments, & calcite fragments. VRFs show some replacement by dolomite. Calcite occurs as coarse crystalline fragments.



690 (2.6mm)

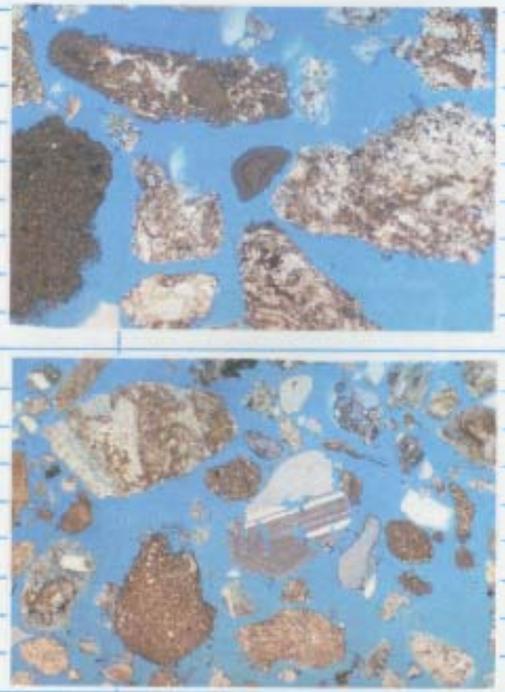
Photo shows VRFs, quartz, feldspar, dolomite, & calcite fragments.

Calcite fragment

690 (1.0mm)

Shows VRF begin replaced by dolomite/calcite and a calcite fragment at the center right of photo.

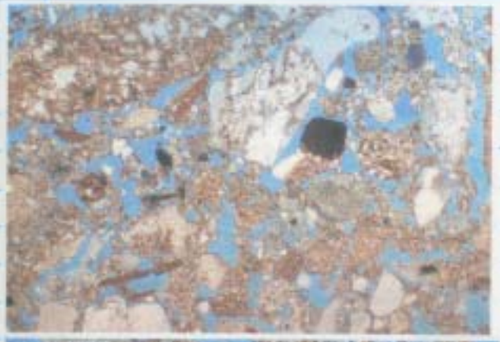
690BT Composed of VRFs, qtz, fldspr, Dolomite, & calcite fragments. Calcite fragments are coarse crystalline. Some VRFs appear to be replaced by dolomite and/or calcite. Drusy in some VRFs are filled by coarse calcite.



690BT 2.6 mm

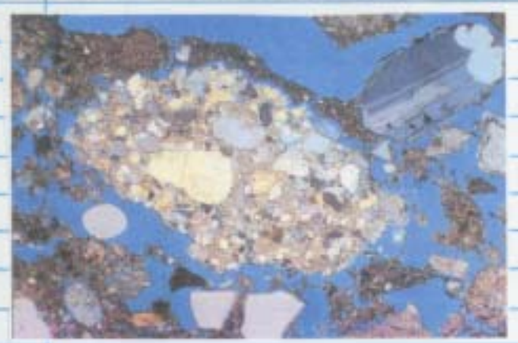
Photos of VRFs, qtz, fldspr, dolomite, and calcite fragments. VRFs often show replacement by calcite and/or dolomite.

701 This sample displays calcite cementation of VRFs, qtz, fldspr, dol fragments, and subbituminous fragments. Subbituminous fragments are made up of qtz and fldspr particles cemented by calcite. VRFs are often being replaced by calcite and/or dolomite.



701 (2.6mm)

Top two photos show VRFs, qtz, fld. & dol fragments which are cemented by calcite. Rock is still very porous.



This photo shows a Subbituminous fragment (SRF) composed of qtz + fldspr cemented by calcite.

Sample 855 This thin section is composed of three large volcanic rock fragments.

The first is composed of quartz and feldspar phenocrysts in a felsic matrix. The matrix has a layered texture and trachytic fabric indicative of flow is present.

Sample 855 (2.6mm)

Photos show felsic matrix with trachytic texture.

855 (cont)

The second VRF is composed of phenocrysts of qtz and fld in a groundmass of feldspar and cristobalite

855 (2.6mm)

Phenocrysts in groundmass of feldspar & cristobalite. Both photo shows resorbed quartz texture.

855 (cont)

The third VRF is composed of qtz + feldspar phenocrysts in an aphanitic groundmass. This sample contains more phenocrysts than previous two samples. Also some biotite phenocrysts.

855 (2.6 mm)

Phenocrysts of qtz, feldspar, and biotite in aphanitic feldspar matrix.

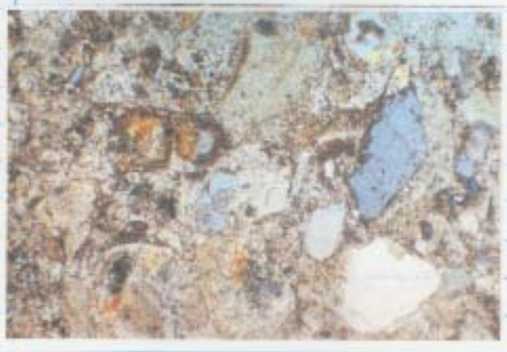
856 This section composed of several VRFs. all with different textures. Some are described with photos below.

856 (2.6 mm)
This VRF is aphanitic in texture and also has a pumiceous fabric in parts of the fragment. Glass shards are replaced by zeolite (clinoptilolite most likely). Also some vugs are lined with zeolite. Vugs may be dissolved out feldspar.

856 (cont)

856 2.6mm

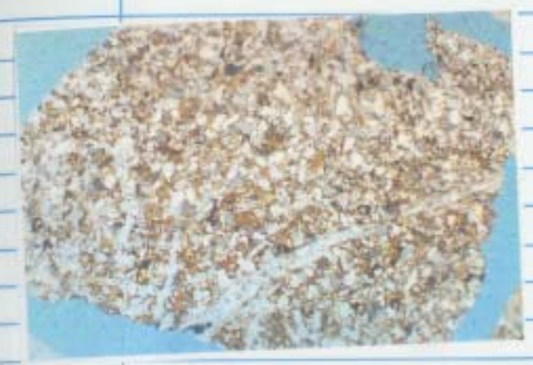
This VRF is composed of lath of feldspar in glass matrix.



856 (2.6mm)

Sample composed of phenocrysts of alt feldspar in aphanitic matrix.

864 The sample is composed mostly of VRFs with a few SR-FS sandstone cemented by calcite (dolomite). VRFs have a variety of texture.



864 (2.6mm)

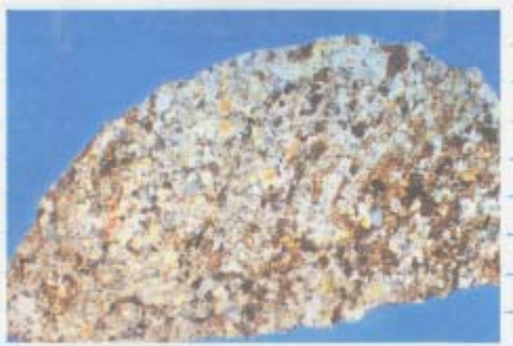
Top photo shows sandstone fragment composed of qtz + fld in calcite cement.

Bottom two photos show VRFs with variety of texture.

867 Composed of mostly VRFs that are relatively coarse (>1.0 mm). VRFs have lathoid texture. Also a certain few SRFs (sandstone fragments).

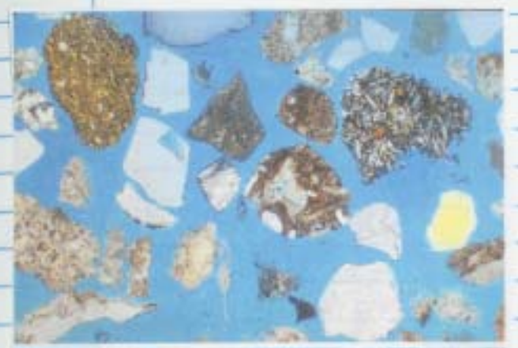
867 (2.6 mm)

Top 2 photos of VRFs with lathoid texture.



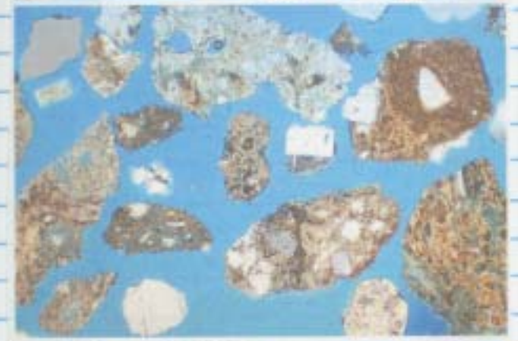
Bottom photo is of a sandstone fragment

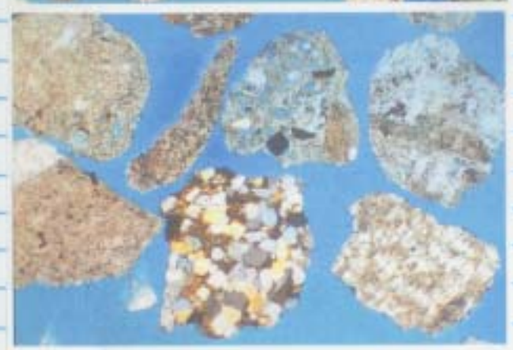
868 Composed predominantly of VRFs with varied texture. Minor Vol Fragments of SRFs (sandstone fragments). Also grain + feldspar fragments.



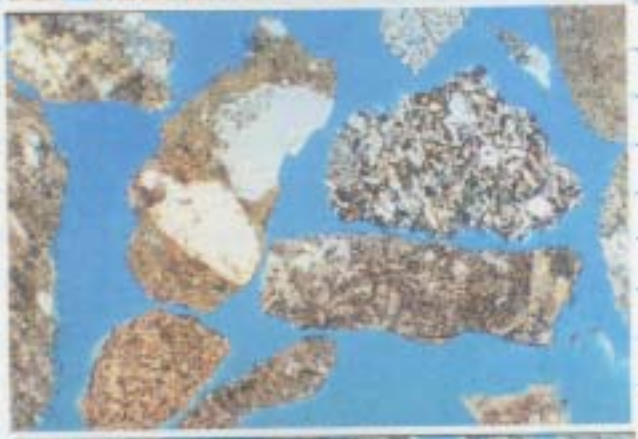
868 2.6mm

Photos showing 03/08/02 VRFs, grain feldspar, & minor Vol Fragments of SRFs.





869 Composed of VRFs, groundmass, and feldspar. VRFs have variety of texture. Porphyritic, inplonitic, devolite, pumiceous.



869 (2.6 mm)

Photos show VRFs, groundmass of feldspar. VRFs have variety of texture.

Blank page
BW 2-27-02

3/13/2001
BAW

Sorbent (Calcite) preparation.

Solutions: 309/2 1A, 309/146 51, and 309/146 52
Each Solution was a 125g $CaCO_3$ into 3.5L of 0.02M $NaHCO_3$ that had been aged.
Solutions were processed separately.

Solutions were filtered with a Whatman #2 (18.5cm) paper (cat# 1002 185 >8μm). Large glass funnel into a 2000mL filter flask while pulling vacuum. Then washed three times with nanopure water. The calcite was transfered from the filter paper to the freeze drying beaker with a spatula.
One "aliquot" of 309/2 1A was freeze dried for 1 hour. The other "aliquot" of 309/2 1A was frozen in a freezer before deep freezing. This last aliquot had a ~ layer of frozen water on top of the calcite.

Next attempt was to combine the two 309/2 1A aliquot together. They were rewetted with nanopure water. They were filtered through a Buchner funnel under vacuum with the same Whatman #2 filter paper (cat# 1002-185). The calcite was scraped off the filter paper with a spatula and collected in a freeze dryer beaker. The rubber cap + glass neck were placed on top of the beaker. The sample was pre-frozen by placing in the acetone/dry ice bath for 60 seconds. It was then ~~attached~~ attached onto the freeze dry for about 45 minutes. It was then removed and the sample was placed into a beaker for that particular solution (ie 309/2 1A). Six aliquots of 309/2 1A were freeze dried in order to process all of 309/2 1A. All aliquots were placed in one tightly sealed beaker with minimal headspace. This beaker was placed in a desicator with a vacuum top.

BAW 2-27-02

3/15/2001
BAW

Solns 309/146 51+52 were not processed today

3/14/2001
BAW

Sorbent (Calcite) preparation

Solns 309/146 51 and 52 were kept separate through the freeze drying process.

Solutions were filtered with a Whatman #2 (18.5cm) paper (cat# 1002 185, >8μm). A Buchner funnel under vacuum was used. The samples were washed three times in the Buchner funnel with nanopure water. The calcite was transfered to a beaker with a spatula. Rubber caps + glass necks were placed ontop of a freeze dryer beaker. This beaker, with sample, was prefrozen by placing in the acetone/dry ice solution for about 1 minute. Then it was attached to the freeze dryer and dried for about an hour. Then the sample was removed and placed in an appropriate beaker (51 for 51 and 52 in 52). 7 aliquots of 309/146 51 were processed and five aliquots of 309/146 52 were processed. The beakers with the fried dried calcite was stored in a dessicator (with a vacuum).

XRD Analysis
Portions of the three calcite sorbents (309/2 1A, 309/146 51, + 309/14652) were separately ground with a mortar + pestal and placed in vials for XRD analysis.
Containers labeled as such:
309/2-1A was 1A-XRD,
309/146-51 was 51-XRD, and
309/146-52 was 52-XRD

Target Concentration = 0.1N HNO_3

Reagents - Conc HNO_3 - trace metal grade - Fisher cat# A509212
lot# 1100046, open 3/15/2001
- nanopure water

3/15/2001 BAW cont

Conc HNO_3 @ 16N. To make 1.6L of 0.1N HNO_3,

add 10mL conc HNO_3 to a 1.6L FVOL.

$$\frac{(10\text{mL conc})(16\text{ mol/L})}{1.6\text{L}}\left(\frac{1\text{L}}{10^3\text{mL}}\right) = 0.1\text{N}$$

Added 10mL of conc HNO_3 (vol pipet) to a 1000mL vol flask and diluted to mark with nanopure water. Transfered this solution into a clean 2.5L solvent bottle. Added an additional 600mL of nanopure water (500mL + 100mL vol flask)

Target Concentration = 1.0N HNO_3

Reagents- Conc HNO_3 (Trace metal grade) Fisher cat# A509-212, lot# 1100040, open 3/15/2001 (16N)
- nanopure water

To make 400mL of 1N HNO_3, add 25mL conc HNO_3 (16N) to a 400mL FVOL in nanopure water

$$\frac{(25\text{mL conc})(16\text{ mol/L})}{0.4\text{L}}\left(\frac{1\text{L}}{10^3\text{mL}}\right) \simeq 1.00\text{N}$$

Added 25mL (vol pipet) of conc HNO_3 to a 200mL vol flask and diluted to mark with nanopure water. Transferred this to a larger container. Refilled 200mL vol flask to mark with nanopure water and added it to larger container with other aliquot for 400mL total.

Calcite solutions for ICP analysis

Soln CA51- target conc 400ppm Ca from Fisher stock reagent bottle

3/15/2001 cont BAW

reagents - Fisher $CaCO_3$ cat# C64-500, lot# 986396, open 8/3/1999
- 0.1N HNO_3 (309/223)

Add 0.1g of $CaCO_3$ to a 100mL FVOL:

$$\frac{(0.1\text{g } CaCO_3)\left(\frac{40.08\text{g Ca}}{100.09\text{g } CaCO_3}\right)\left(\frac{10^6\text{mg}}{\text{g}}\right)}{100\text{mL}} = 400.4\text{ppm Ca}$$

Balance = Mettler AE240

mass of 100mL vol flask (g)	mass of flask + sample (g)
62.1037	62.2041

Solns CA52, CA53, CA54 - target conc 400ppm Ca from various freeze dried calcite preparations
reagents - freeze dried calcite from 309/21A (309/222)
- freeze dried calcite from 309/14651 (309/223)
- freeze dried calcite from 309/14652 (309/223)
- 0.1N HNO_3 (309/223)

Balance = Mettler AE240

sample ID	Mass of 100mL vol flask (g)	Mass of flask + sample (g)	soln ID
309/21A	57.5288	57.6321	CA52
309/14651	57.2792	57.3813	CA53
309/14652	57.4531	57.5529	CA54

Solns CA55, CA56 - target conc 4000ppm Ca from newly prepared freeze dried calcite
reagents - freeze dried calcite from 309/14651 (309/223)
freeze dried calcite from 309/14652 (309/223)
~~nanopure water~~ BAW 3/15/2001 1.0N HNO_3 (309/224)

Mass of sample increased to 1g to reach 4000ppm soln conc.

3/15/2001 cont BAW

Balance = Mettler AE240

Sample ID	Mass of 100mL vol flask (g)	Mass of flask + sample (g)	Soln ID
309/146 S1	62.0378	63.0401	CAS5
309/146 S2	62.3614	63.3633	CAS6

Soln CAS7 - 400 ppm Reference Std for Calcium
reagents - 1000 ppm Ca - Spex certiprep cat# PLCA2-2X
lot# 7-114CA, open 2/21/2001, exp 1/31/2002.
- nanopure water
Added 10 mL (vol flask) of 1000 ug/mL Ca to a 25 mL vol flask and diluted to mark with nanopure water.

$$\frac{(10\,mL)(1000\,ug/mL)}{25\,mL} = 400\,ug/mL\ Ca$$

Soln CAS8 - Acid blank (0.1N HNO_3)
- Aliquot of 309/223 used.

3/16/2001 JP

Calcite samples were prepared and sent to Div 01 for SEM analysis. SEM samples of 309/2 1A, 309/146 S1, and 309/146 S2 were prepared. These freeze-dried calcite samples were prepared by placing aliquots of each sample on aluminum stubs covered with carbon tape. The carbon tape acts as a conductive surface. The aluminum stubs were labeled as followed and delivered to Div 01 for coating + SEM analysis.

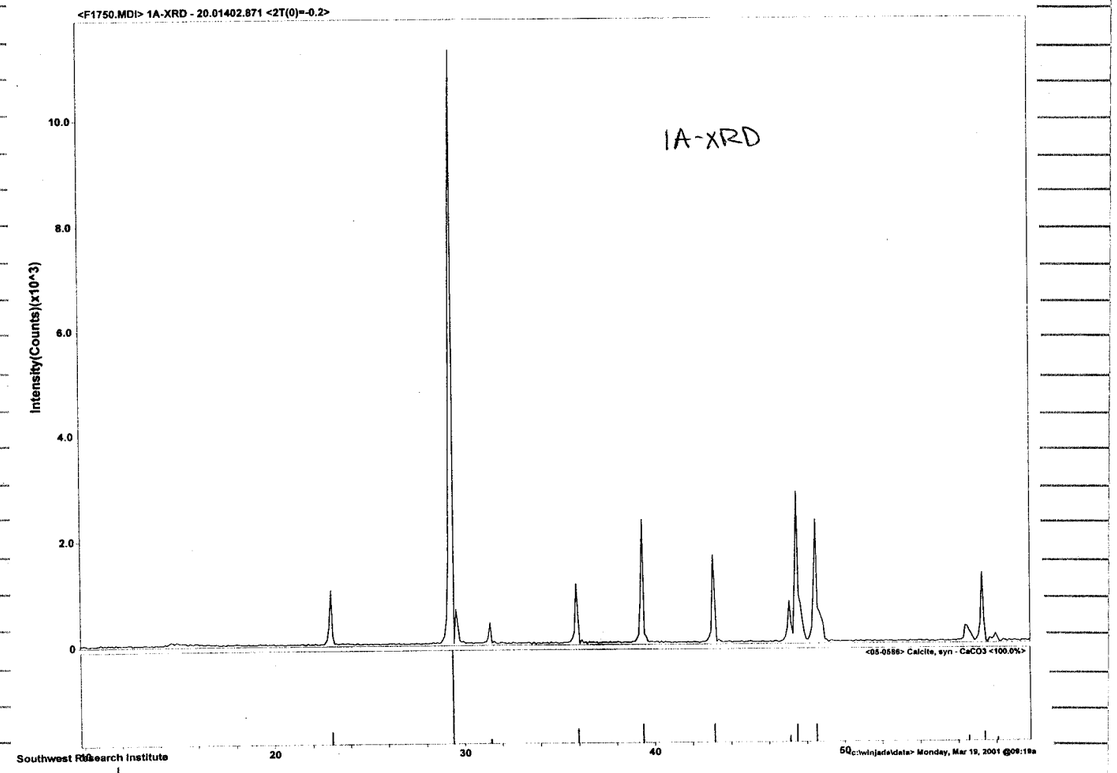
Sample	Label
309/2 1A	A1
309/146 S1	S1
309/146 S2	S2

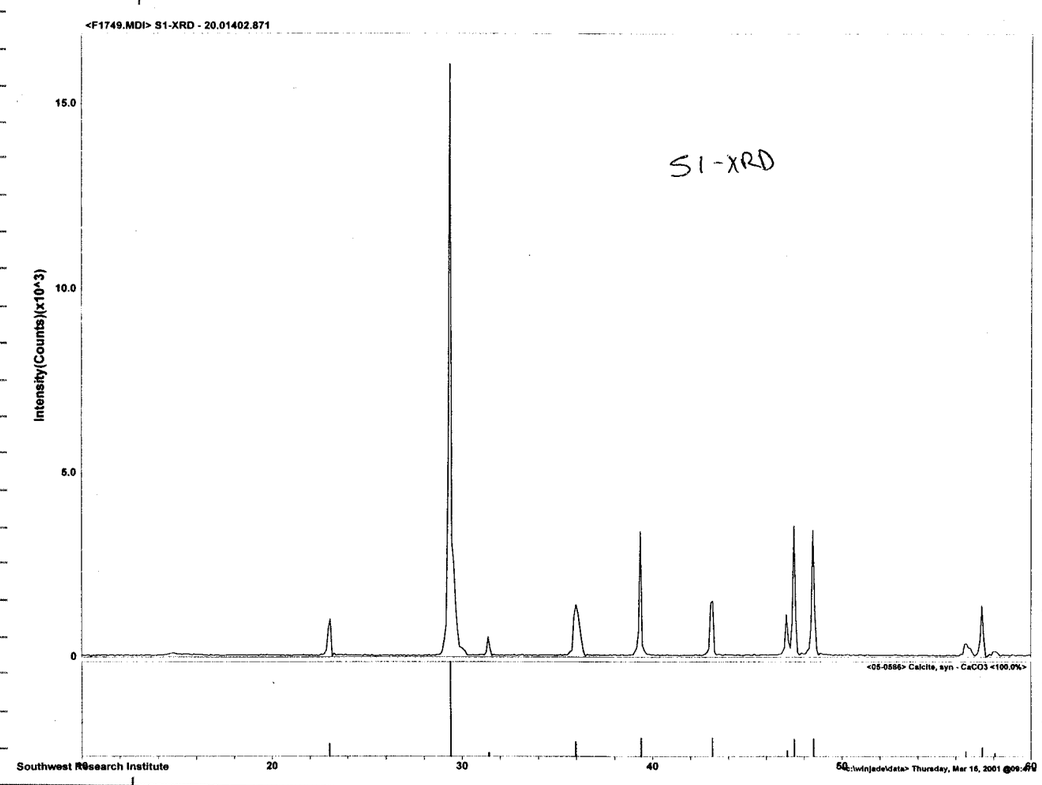
3/16/2001 cont BAW

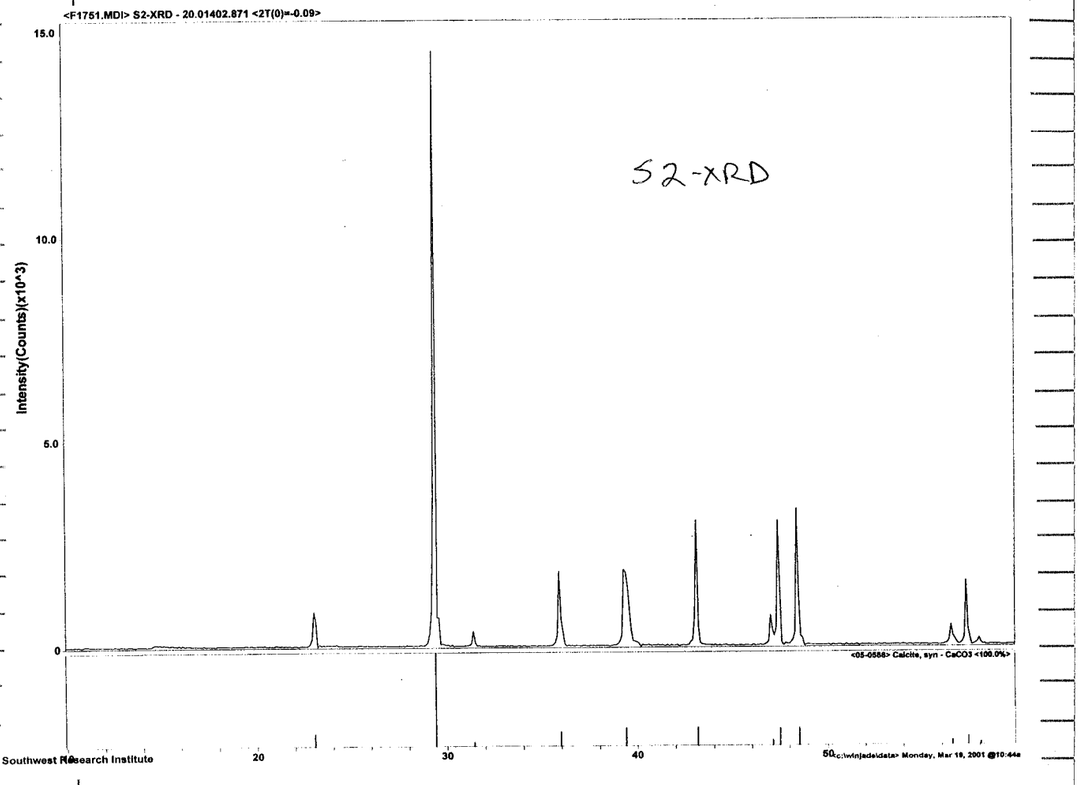
Samples CAS1 to CAS8 (309/224-226) delivered to Division 01 for analysis (major/minor elements by ICP)

3/20/2001 JP

Results of XRD analyses of calcite samples sent to Div 01 are shown below. See p 223 for sample label explanation.



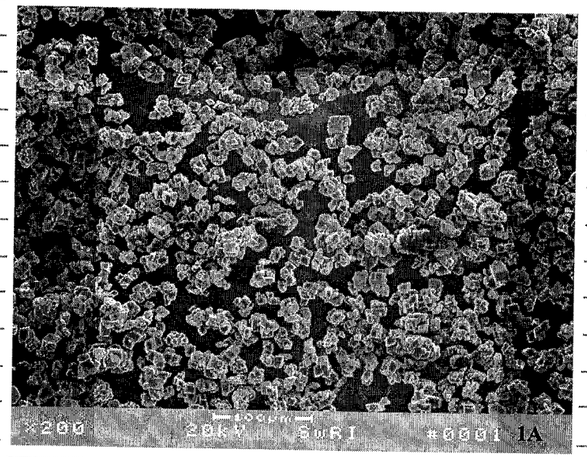

<F1749.MDI> S1-XRD - 20.01402.871
S1-XRD
Intensity(Counts)(x10^3)
15.0
10.0
5.0
0
<05-0586> Calcite, syn - CaCO3 <100.0%>
20
30
40
50
Southwest Research Institute
c:\winjade\data> Thursday, Mar 15, 2001 @09:40

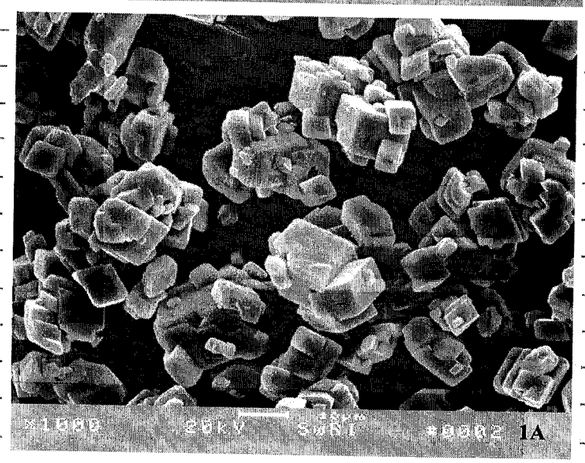

<F1751.MDI> S2-XRD - 20.01402.871 <2T(0)=-0.09>
S2-XRD
Intensity(Counts)(x10^3)
15.0
10.0
5.0
0
<05-0586> Calcite, syn - CaCO3 <100.0%>
20
30
40
50
Southwest Research Institute
c:\winjade\data> Monday, Mar 19, 2001 @10:44a

3/20/2001
JP

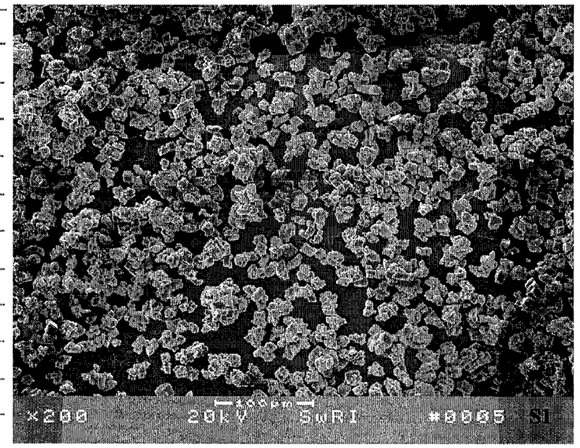
Results of SEM analyses of calcite samples sent to Div 01. Photos of calcite samples at 200x and 1000x magnification are shown below. See p226 for sample label explanation.

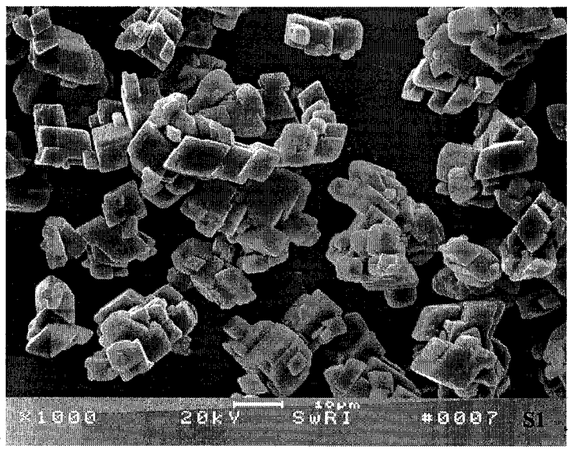
Sample 1A





Sample S1





3/28/2001 BAW

Sample Prep for XRD analysis of Clinoptilolite samples

Six samples were prepared

Sample 1 - Sodium form of clinoptilolite from
CDV * 100/200 * ML * CP * NaF wt = 48.94g
1/25/2001 AJ

Sample 2 - Potassium form of clinoptilolite from
CDV * 100/200 * HL * CP * KF wt = 70.05g
1/25/2001 AJ

Sample 3 - 100/200 mesh cleaned/processed but not exchanged
CDV * (100-200 mesh) * HL * RC * RFe 172.8gm
420/5-15 12/7/00 AJ

Sample 4 - 200-325 mesh (Roberts) from
CDV-2000 200-325 mesh 9/1/00 RC

Sample 5 old/"good" 100-200 mesh Na form in dessicator
from CDV * RC * RFe * HL 11/2/99 AJ
SA = 11.0 m^2/g satd NaCl

Sample 6 raw 200-325 mesh that Alka is currently working with from CDV-200/325-UC.WA
420/54 3/27/01

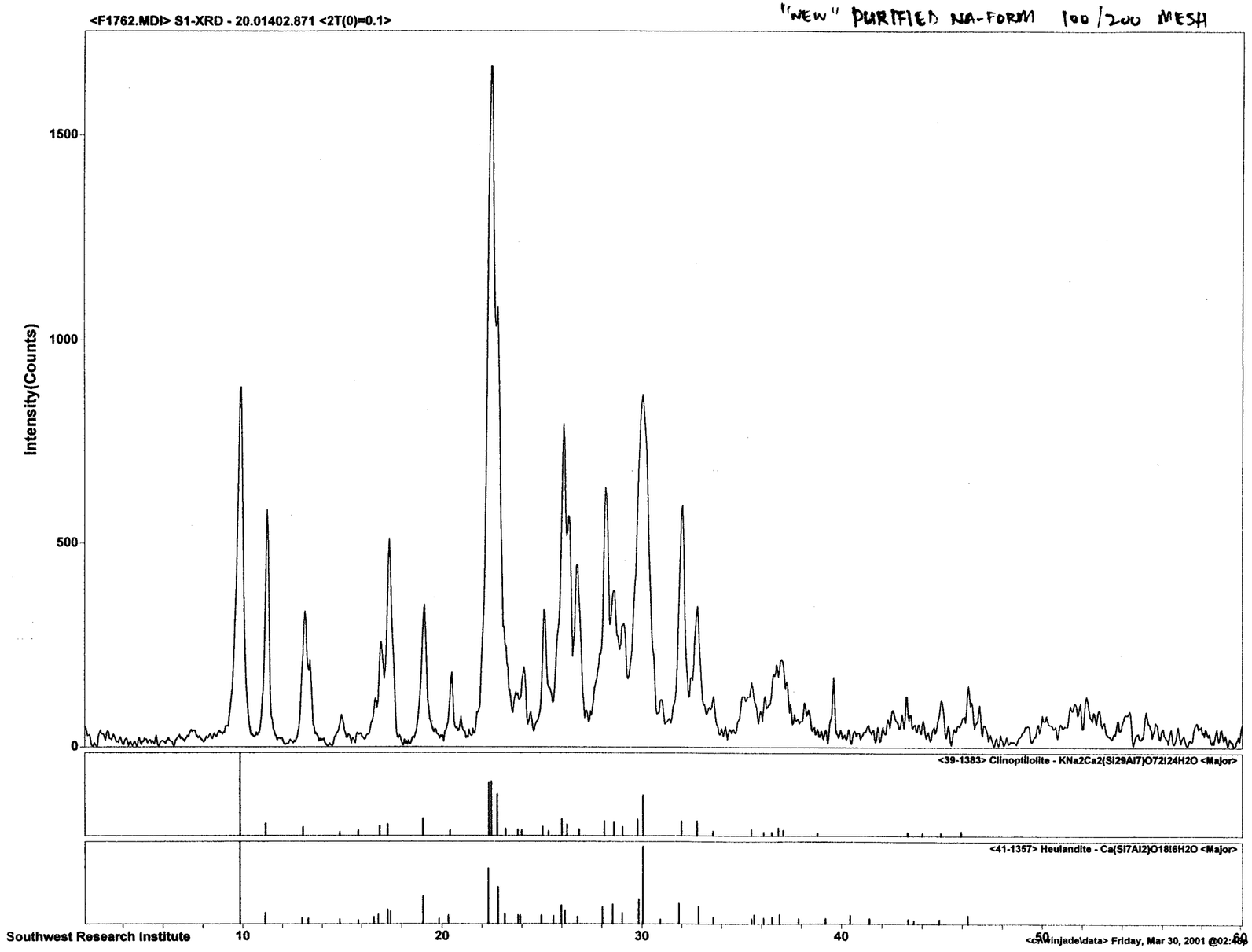
Each Sample was crushed in a mortar + pestal for about 15 min to produce a "grit free" powder. "Grit-free" test was rubbing small aliquot of crushed sample between index finger + thumb. Mortar + pestal was washed with nanopure water and hand dried between samples

3/29/2001 BAW

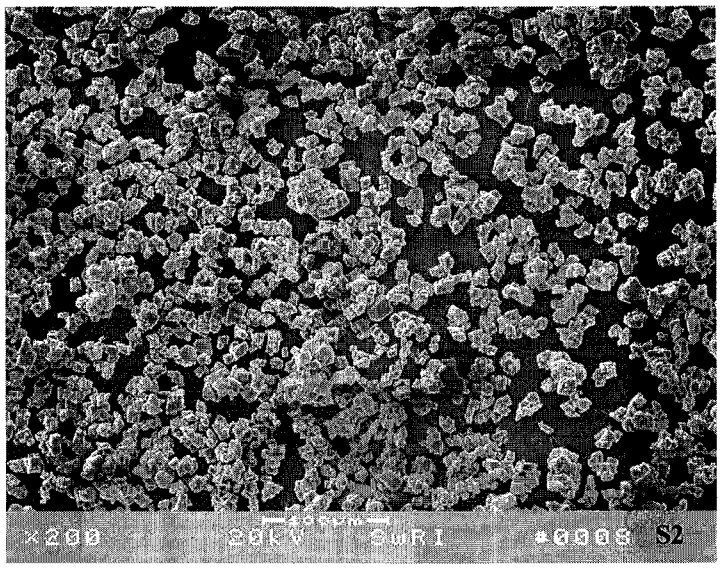
The six samples (309/232) were taken to Div 18 for XRD analysis. They were labeled as follows:

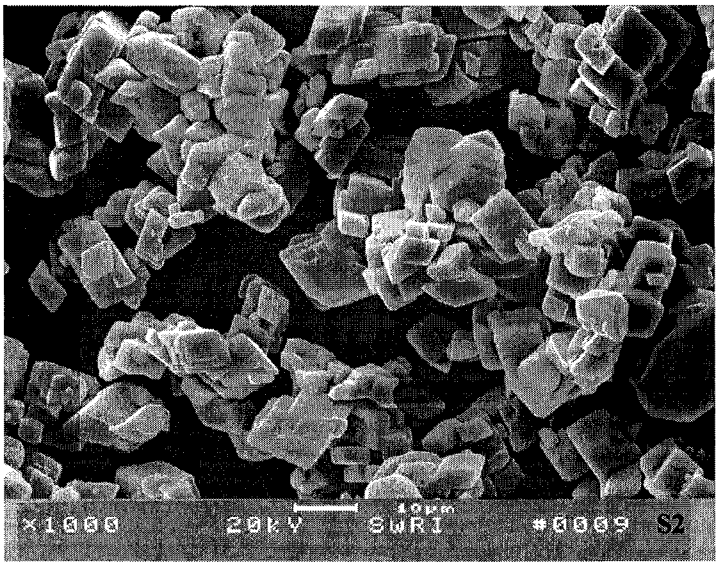
Sample 1 = S1-XRD
Sample 2 = S2-XRD
Sample 3 = S3-XRD
Sample 4 = S4-XRD
Sample 5 = S5-XRD
Sample 6 = S6-XRD

4/3/2001 XRD analysis results of clinoptilolite samples (309/232)
BAW

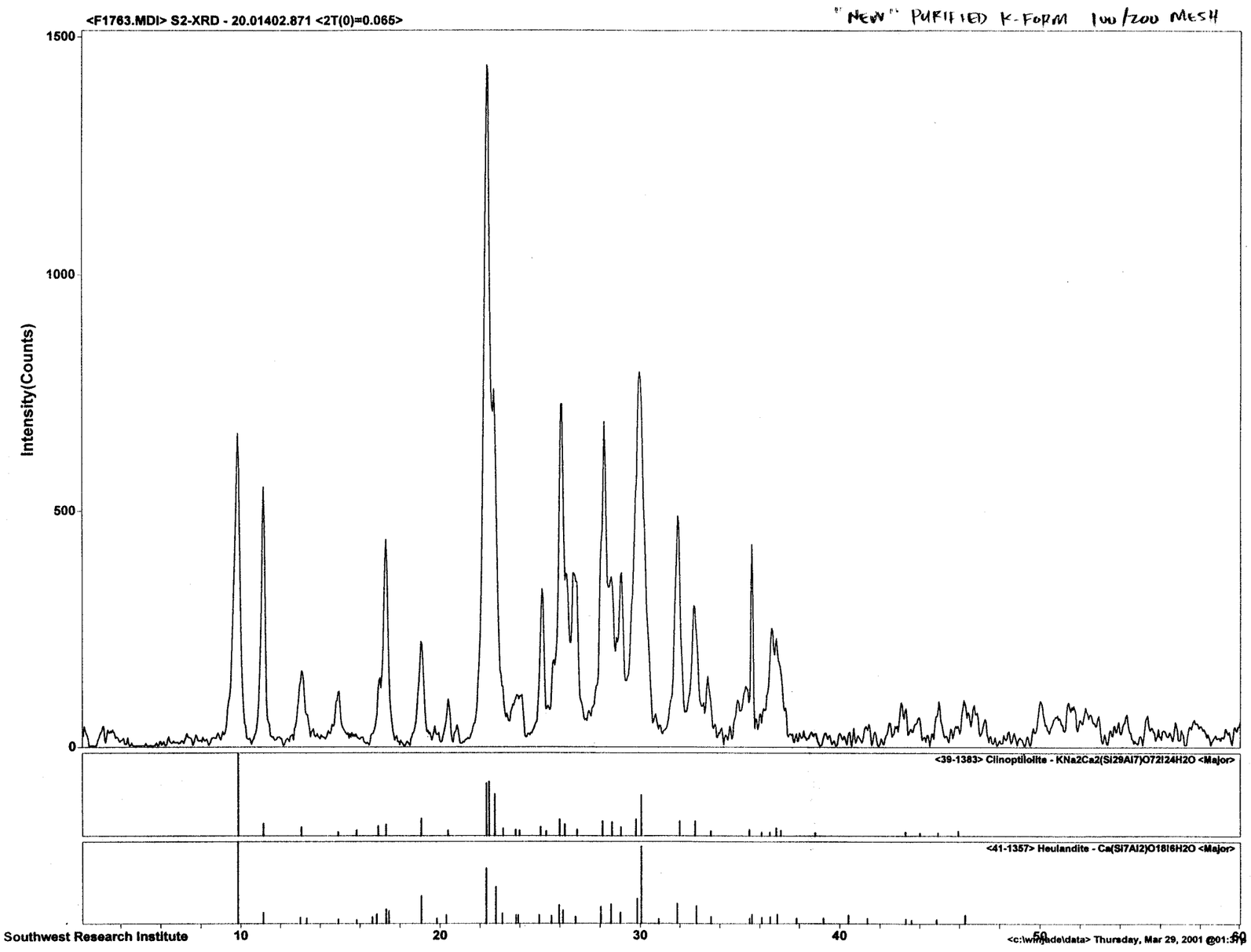


Sapk S2 see (309/230+231)

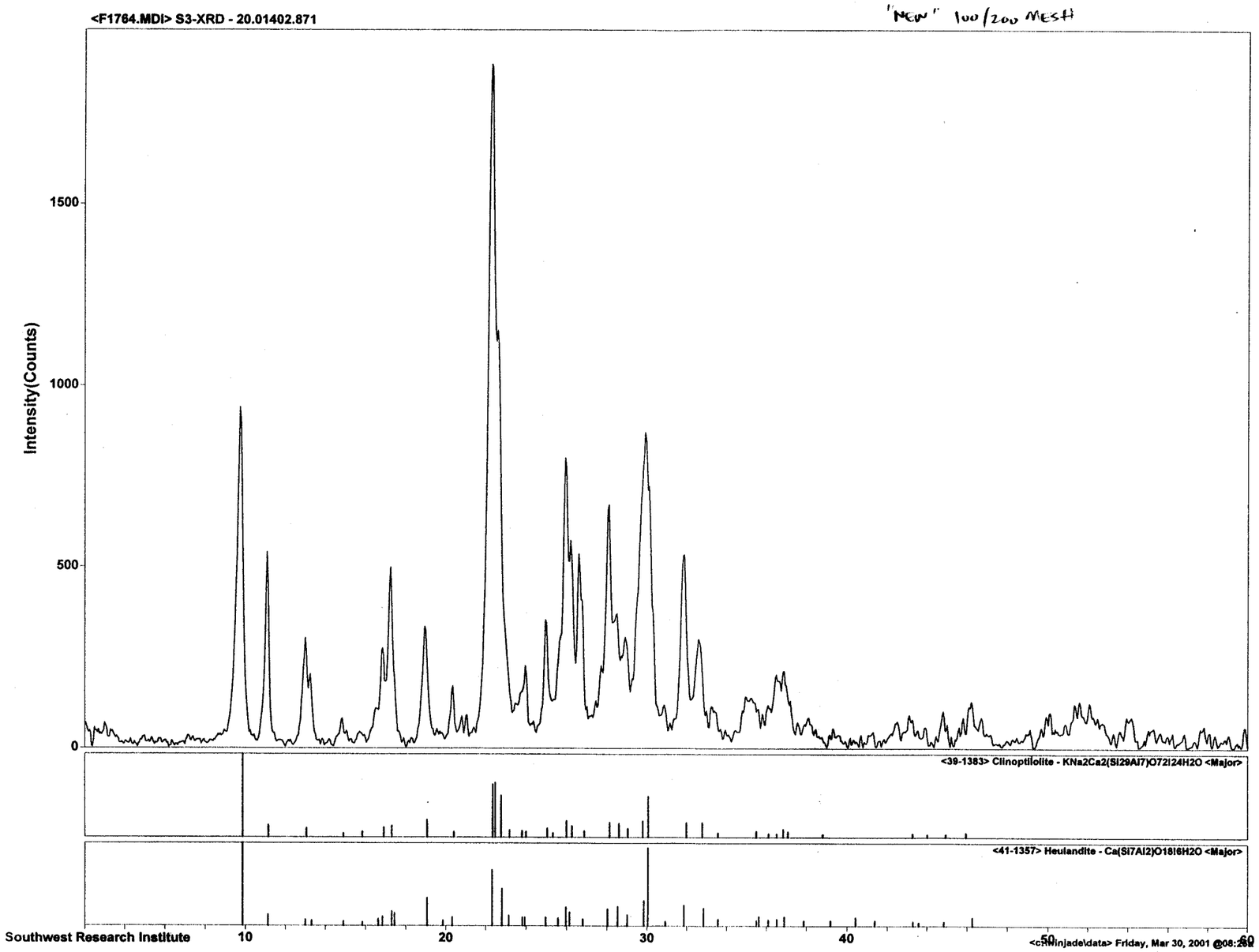




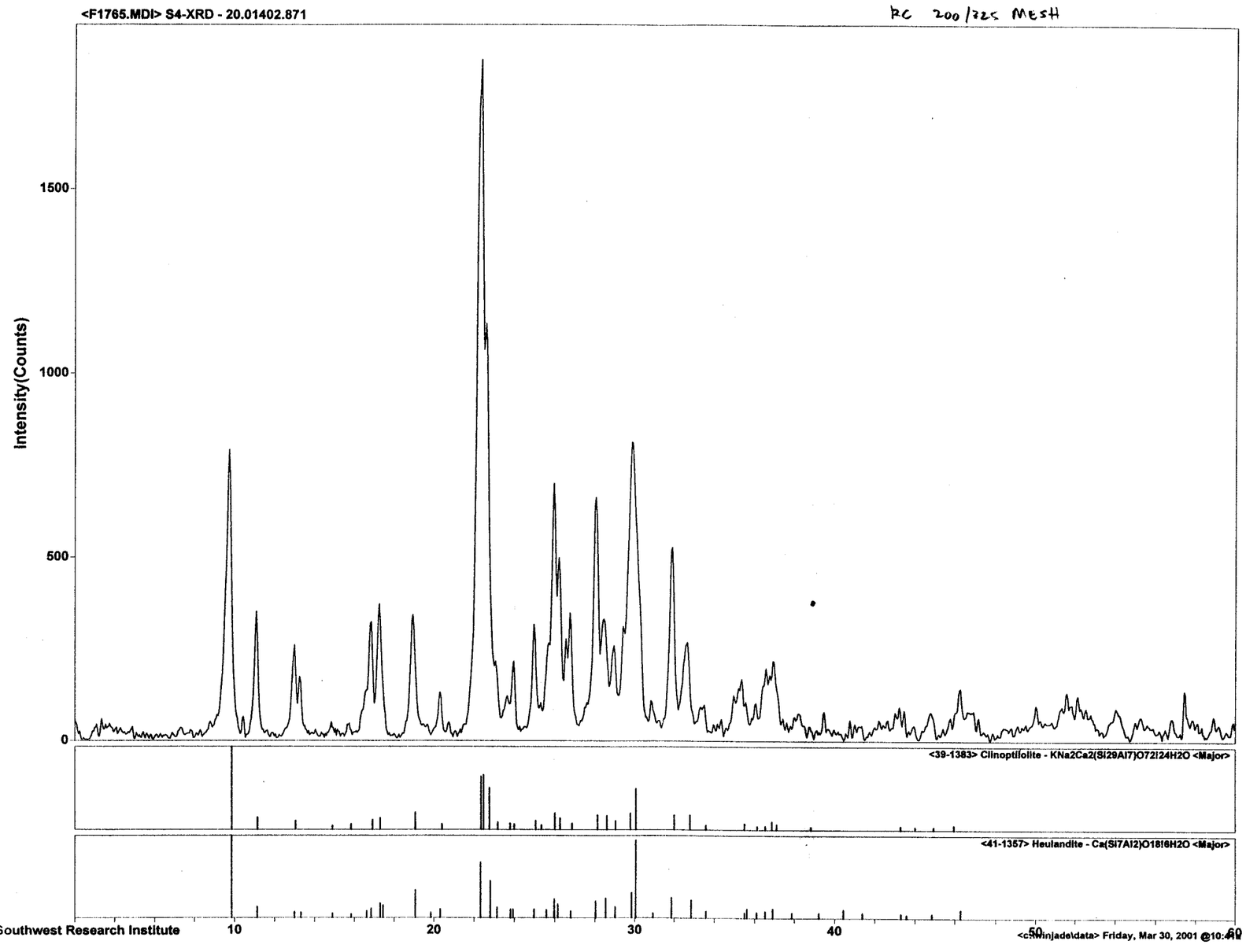
4/3/2001
cont BAW



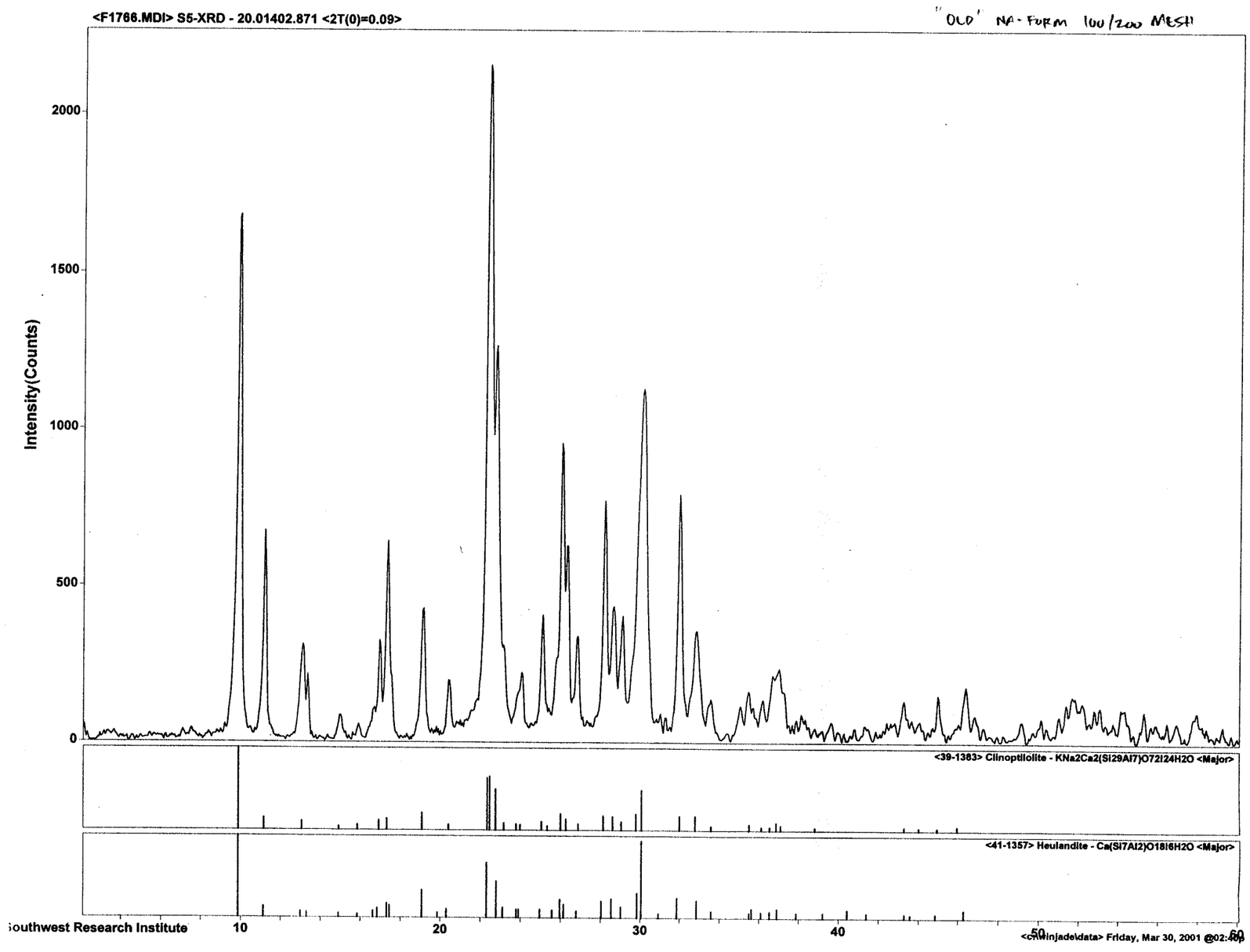
4/3/2001
cont BW



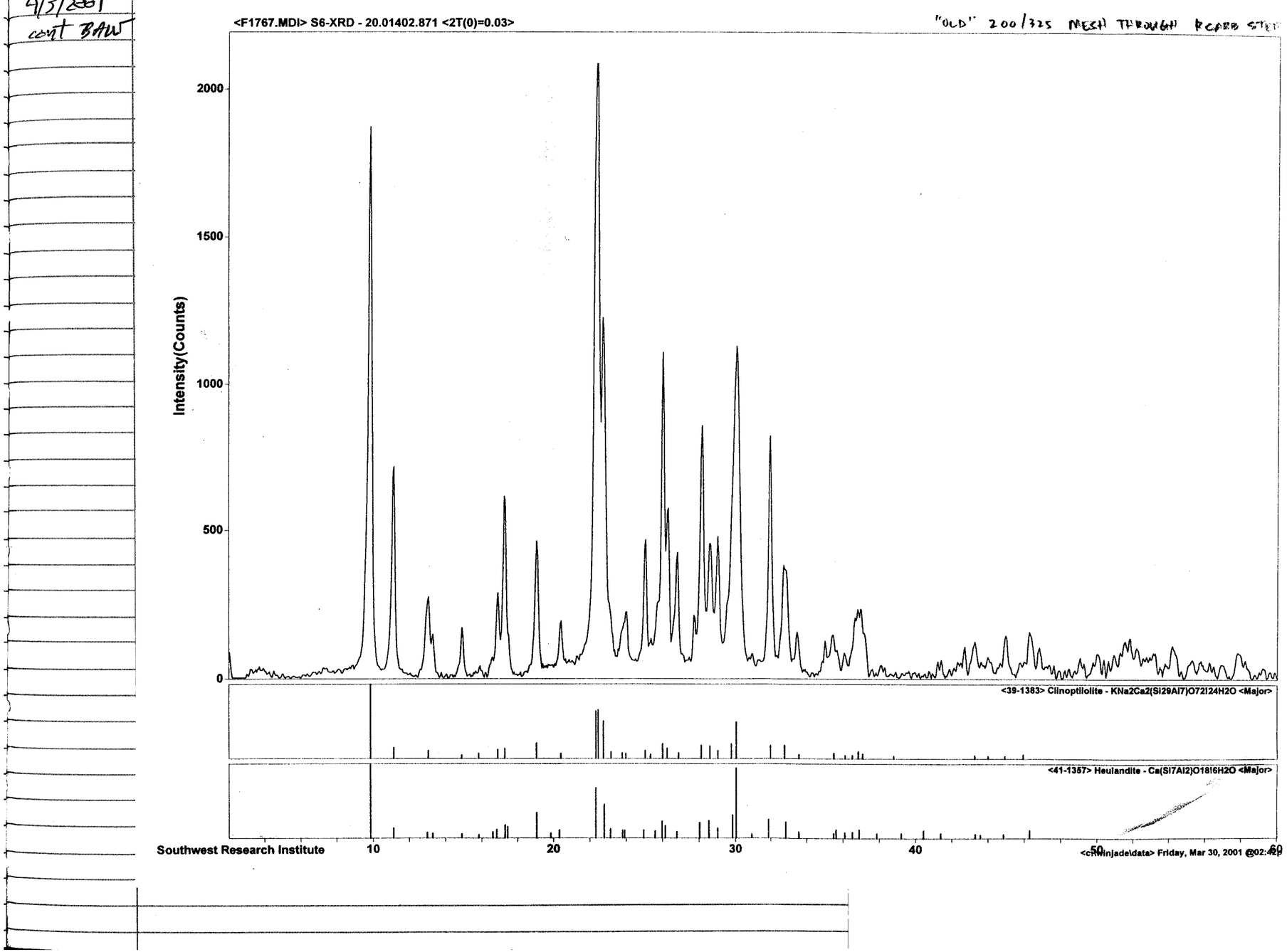
4/3/2001
cont BALLS



4/3/2001
Lout BAW



4/3/2001
cont BAW



4/10/2001
BAW

Target Concentration 0.32N HNO_3

Using concentrated HNO_3 (16M) and a 50 mL final volume,

$$\frac{(1mL)(16M)}{50mL} = 0.32M\ HNO_3$$

Reagents - Conc HNO_3; Fisher A509-212 Trace Metal Grade
lot# 1100040
- nanopure water

Added 1 mL (eppendorf pipet) of conc HNO_3 to a 50 mL (vol. flask) final volume with nanopure water.

Analysis of Freeze Dried Calcite for % moisture

Sample 309/14651 - freeze dried, ~ 1 gram (309/225)

Wt of Crucible (g)	Wt of sample + crucible (g)	
		before oven drying
28.8206	29.8398	

Sample was placed in oven at 210°C for 3 hours. It was placed in a dessicator to cool, then reweighed

after oven drying, Wt of sample + crucible (g)
29.7103

Weight before drying = 1.0192g
Weight after drying = 0.8897g
Weight of moisture/water = 0.1295g

% moisture = $\left(\frac{0.1295}{1.0192}\right) \times 100 = 12.71\%$

4/11/2001
BAW

Analysis of Freeze Dried samples for % moisture

Approximately 1 gram of sample was placed in crucible and weighed. The samples were placed in an oven at 210°C for 3 hours. They were placed in a dessicator to cool, then reweighed.

Sample	Crucible mass(g) before drying	Crucible + sample before drying	Crucible + sample after drying
Fisher*	34.0011	35.0021	35.0018
309/2 1A BAW 4/11/01	~~36~~ 30.0600	31.0634	30.8899
309/14652	28.7168	29.7135	29.6115

Fisher* = Fisher $CaCO_3$ cat# C64-500, lot# 986596

Results of major/minor element analysis by ICP of calcite samples sent to Div01 (see 309/227) are shown below. See pages 309/224-226 for explanation of labels

BAW 4/11/2001

4/11/2001
BAW cont

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 1

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 03/16/01

Matrix: Liquid

Project No.: 20.01402.871

Lab System ID: 158382

Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	0.007	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	386	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.006	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.108	0.005
Sulfur	0.078	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	0.038	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
DUPLICATE SUMMARY

Sample ID
CAS 1

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 03/16/01

Matrix: Liquid

Project No.: 20.01402.871

Lab System ID: 158382

Work Order: 19781

Analysis	Sample Result (mg/L)	Duplicate Result (mg/L)	RPD
Aluminum	<0.05	<0.05	0.00%
Antimony	<0.02	<0.02	0.00%
Arsenic	<0.01	<0.01	0.00%
Barium	0.007	0.007	2.08%
Beryllium	<0.005	<0.005	0.00%
Bismuth	<0.01	<0.01	0.00%
Boron	<0.05	<0.05	0.00%
Cadmium	<0.005	<0.005	0.00%
Calcium	386	382	1.18%
Chromium	<0.005	<0.005	0.00%
Cobalt	<0.005	<0.005	0.00%
Copper	0.006	0.006	2.67%
Iron	<0.05	<0.05	0.00%
Lanthanum	<0.005	<0.005	0.00%
Lead	<0.005	<0.005	0.00%
Lithium	<0.005	<0.005	0.00%
Magnesium	<0.05	<0.05	0.00%
Manganese	<0.005	<0.005	0.00%
Molybdenum	<0.005	<0.005	0.00%
Nickel	<0.005	<0.005	0.00%
Palladium	<0.005	<0.005	0.00%
Phosphorus	<0.05	<0.05	0.00%
Potassium	<0.1	<0.1	0.00%
Selenium	<0.02	<0.02	0.00%
Silicon	<0.05	<0.05	0.00%
Silver	<0.005	<0.005	0.00%
Sodium	<0.1	<0.1	0.00%
Strontium	0.108	0.107	0.68%
Sulfur	0.078	0.082	4.89%
Thallium	<0.05	<0.05	0.00%
Thorium	<0.01	<0.01	0.00%
Tin	0.038	0.037	1.01%
Titanium	<0.005	<0.005	0.00%
Tungsten	<0.01	<0.01	0.00%
Uranium	<0.1	<0.1	0.00%
Vanadium	<0.005	<0.005	0.00%
Yttrium	<0.005	<0.005	0.00%
Zinc	<0.005	<0.005	0.00%
Zirconium	<0.005	<0.005	0.00%

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 2

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Liquid
Lab System ID: 158383

Client: Division 20
Date Received: 03/16/01
Project No.: 20.01402.871
Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	0.006	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	335	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.092	0.005
Sulfur	0.072	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	0.038	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
MATRIX SPIKE SUMMARY

Sample ID
CAS 2

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Liquid
Lab System ID: 158383

Client: Division 20
Date Received: 03/16/01
Project No.: 20.01402.871
Work Order: 19781

Analysis	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery
Aluminum	<0.05	2.06	2.00	102.8%
Antimony	<0.02	0.530	0.500	105.9%
Arsenic	<0.01	2.12	2.00	105.8%
Barium	0.006	2.05	2.00	102.3%
Beryllium	<0.005	0.048	0.050	95.7%
Bismuth	NA	NA	NA	NA
Boron	NA	NA	NA	NA
Cadmium	<0.005	0.050	0.050	99.1%
Calcium	335	350	20	73.8%
Chromium	<0.005	0.198	0.200	99.1%
Cobalt	<0.005	0.499	0.500	99.9%
Copper	<0.005	0.256	0.250	102.5%
Iron	<0.05	0.957	1.00	95.7%
Lanthanum	NA	NA	NA	NA
Lead	<0.005	0.526	0.500	105.2%
Lithium	NA	NA	NA	NA
Magnesium	<0.05	20.3	20	101.3%
Manganese	<0.005	0.495	0.500	99.0%
Molybdenum	NA	NA	NA	NA
Nickel	<0.005	0.489	0.500	97.7%
Palladium	NA	NA	NA	NA
Phosphorus	NA	NA	NA	NA
Potassium	<0.1	22.2	20	111.1%
Selenium	<0.02	2.38	2.00	118.8%
Silicon	NA	NA	NA	NA
Silver	<0.005	0.050	0.050	100.6%
Sodium	<0.1	20.7	20	103.7%
Strontium	NA	NA	NA	NA
Sulfur	NA	NA	NA	NA
Thallium	<0.05	2.13	2.00	106.5%
Thorium	NA	NA	NA	NA
Tin	NA	NA	NA	NA
Titanium	NA	NA	NA	NA
Tungsten	NA	NA	NA	NA
Uranium	NA	NA	NA	NA
Vanadium	<0.005	0.495	0.500	98.9%
Yttrium	NA	NA	NA	NA
Zinc	<0.005	0.495	0.500	99.0%
Zirconium	NA	NA	NA	NA

NA- Not Applicable.

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 3

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 158384

Client: Division 20

Date Received: 03/16/01

Project No.: 20.01402.871

Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	0.007	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	350	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.098	0.005
Sulfur	0.071	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	0.037	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

4/11/2001
cont

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 4

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 158385

Client: Division 20

Date Received: 03/16/01

Project No.: 20.01402.871

Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	0.007	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	345	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.096	0.005
Sulfur	0.066	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	0.029	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.011	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 5

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 03/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 158386
Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.060	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	3514	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.024	0.005
Iron	0.086	0.05
Lanthanum	<0.005	0.005
Lead	0.008	0.005
Lithium	<0.005	0.005
Magnesium	0.053	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.137	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	0.326	0.1
Strontium	0.924	0.005
Sulfur	0.433	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	0.158	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.008	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 6

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 03/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 158387
Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.060	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	3568	0.1
Chromium	**<0.005**	0.005
Cobalt	**<0.005**	0.005
Copper	**0.024**	0.005
Iron	0.087	0.05
Lanthanum	<0.005	0.005
Lead	0.006	0.005
Lithium	<0.005	0.005
Magnesium	0.051	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.155	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	0.402	0.1
Strontium	0.939	0.005
Sulfur	0.433	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	0.158	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.008	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 7

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 03/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 158388
Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	0.007	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	386	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	0.071	0.005
Lead	0.036	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.023	0.005
Sulfur	0.083	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	<0.01	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.007	0.005
Zirconium	<0.005	0.005

4/11/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 8

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 03/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 158389
Work Order: 19781

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.02	0.02
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	<0.1	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.02	0.02
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	<0.005	0.005
Sulfur	<0.05	0.05
Thallium	<0.05	0.05
Thorium	<0.01	0.01
Tin	<0.01	0.01
Titanium	<0.005	0.005
Tungsten	<0.01	0.01
Uranium	<0.1	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

4/12/2001
BAW

Analysis of Calcium in Calcite Samples

Sample ID$	Calcite Mass (g)	Solution Vol (mL)	Calculated Ca Conc (ppm)	Percent moisture*	Corrected Ca conc (ppm)	Div 01 results# Ca (ppm)	Percent Difference
CAS1	0.1004	100	402.0	0.03	401.9	386	3.96
CAS2	0.1033	100	413.7	17.29	342.2	335	2.10
CAS3	0.1021	100	408.8	12.71	356.9	350	1.93
CAS4	0.0998	100	399.6	10.23	358.7	345	3.82
CAS5	1.0023	100	4014	12.71	3504	3514	0.28
CAS6	1.0019	100	4012	10.23	3601	3568	0.92

Sample ID$	Vol (mL) of 1000 ppm Ca	Final Volume (mL)	Calculated Conc (ppm)	Analytical Conc (ppm)	Percent Difference
CAS7	10	25	400	386	3.98
CAS8	0	15	0	0	na

$See 309/227 for sample ID information
*See 309/240-241 for percent moisture raw data
#See 309/242-251 for Div 01 results

Oven Drying Calcite Sample

Samples
CAS-A Fisher $CaCO_3$ cat # C64-500, lot 986396
CAS-B Freeze dried calcite from 309/211A (309/222)
CAS-C Freeze dried calcite from 309/14651 (309/223)
CAS-D Freeze dried calcite from 309/14652 (309/223)

Approximately 1gm of each sample was precisely weighed in a 60mL LDPE bottle,*. These bottles were then dried in an oven at °C for minutes. These bottles were reweighed in order to determine the % moisture of the calcite

* with samples

BAW 4/12/2001

4/12/2001 cont BAW

Sample	Crucible mass (g)	Before Drying Crucible + Sample mass (g)	After Drying Crucible + Sample mass (g)
CAS-A	11.1964	12.2062	12.2073
CAS-B	11.1655	12.1982	12.0570
CAS-C	11.1760	12.2011	12.1213
CAS-D	11.1965	12.2012	12.1336

Target Concentration 0.1N HNO_3

Add 10mL conc HNO_3 (16M) to a 1.6L Final volume

$$\left(\frac{(10\text{mL conc})(16\text{mol/L})}{1.6\text{L}}\right)\left(\frac{1\text{L}}{10^3\text{mL}}\right) = 0.1\text{M}$$

reagents: conc HNO_3 - Fisher trace metal grade - cat # A509-212
lot # 1100040
nanopure water

Added 10mL (vol pipet) of conc HNO_3 to a 1000mL vol flask + diluted to mark with nanopure water. Transferred this to a clean 2.5L solvent bottle. Added an additional 600mL of nanopure water (500mL + 100mL vol flask).

Sample Prep for oven dried samples for major/minor elements by ICP.

Target concentration 4000 ppm Ca via 1gm calcite to 100mL

$$\frac{1\text{g } CaCO_3\left(\frac{40.08\text{g Ca}}{100.09\text{g } CaCO_3}\right)\left(\frac{10^6\mu\text{g}}{\text{g}}\right)}{100\text{mL}} = 4004\text{ ppm Ca}$$

4/12/2001 cont BAW — unable to completely dissolve 1g samples w 0.1N acid. Will retry with 1.0N acid.

4/13/2001 BAW

Target Concentration 1.0N HNO_3

To make 800mL of 1.0N HNO_3 from conc (16M)

$$\frac{(50\text{mL conc})(16\text{ mol/L})}{800\text{ mL}} = 1\text{ N } HNO_3$$

reagent 5: Conc HNO_3 - fisher trace metal grade cat# A509-212, lot # 1100040
nanopure water

Added 50mL (vol pipet) of conc HNO_3 to a 1000mL graduated cylinder and diluted to 800mL mark with nanopure water

Oven drying Calcite

Samples
CAS-9 Fisher $CaCO_3$ cat# C64-500, lot 986596
CAS-10 Freeze dried calcite from 309/21A (309/222)
CAS-11 Freeze dried calcite from 309/14651 (309/223)
CAS-12 Freeze dried calcite from 309/14652 (309/223)

Four 100mL vol flasks were dried in a oven at 54°C for 60 minutes. They were allowed to cool in a dessicator. Each flask was weighed. The weight was recorded. Approximately 1 gram of calcite was placed in each flask (one sample- one flask). Each was reweighed. They were then placed in an oven at 54 °C for 67 ~~hours 8 minutes~~ 4/16/01, these were reweighed to determine the percent moisture in the calcite. The remaining portions of 309/21A, 309/14651, & 309/14652 were oven dried at 54 °C for 5 days

4/13/2001 cont BAW

Sample	Flask mass (g)	Before Drying Sample + Flask mass (g)	After Drying Sample + Flask mass (g)
CAS 9	64.0151	65.0422	65.0428
CAS 10	57.7994	58.7835	58.6931
CAS 11	57.4719	58.4717	58.4128
CAS 12	57.1605	58.1621	58.1353

4/16/2001 BAW

Sample Prep for calcite solutions- major/minor element analysis by ICP

Target Concentration 4000 ppm Ca via 1g calcite to 100mL

$$\frac{1\text{g } CaCO_3\left(\frac{40.08\text{g Ca}}{100.09\text{g } CaCO_3}\right)\left(\frac{10^6\text{ mg}}{\text{g}}\right)}{100\text{ mL}} = 4004\text{ ppm Ca}$$

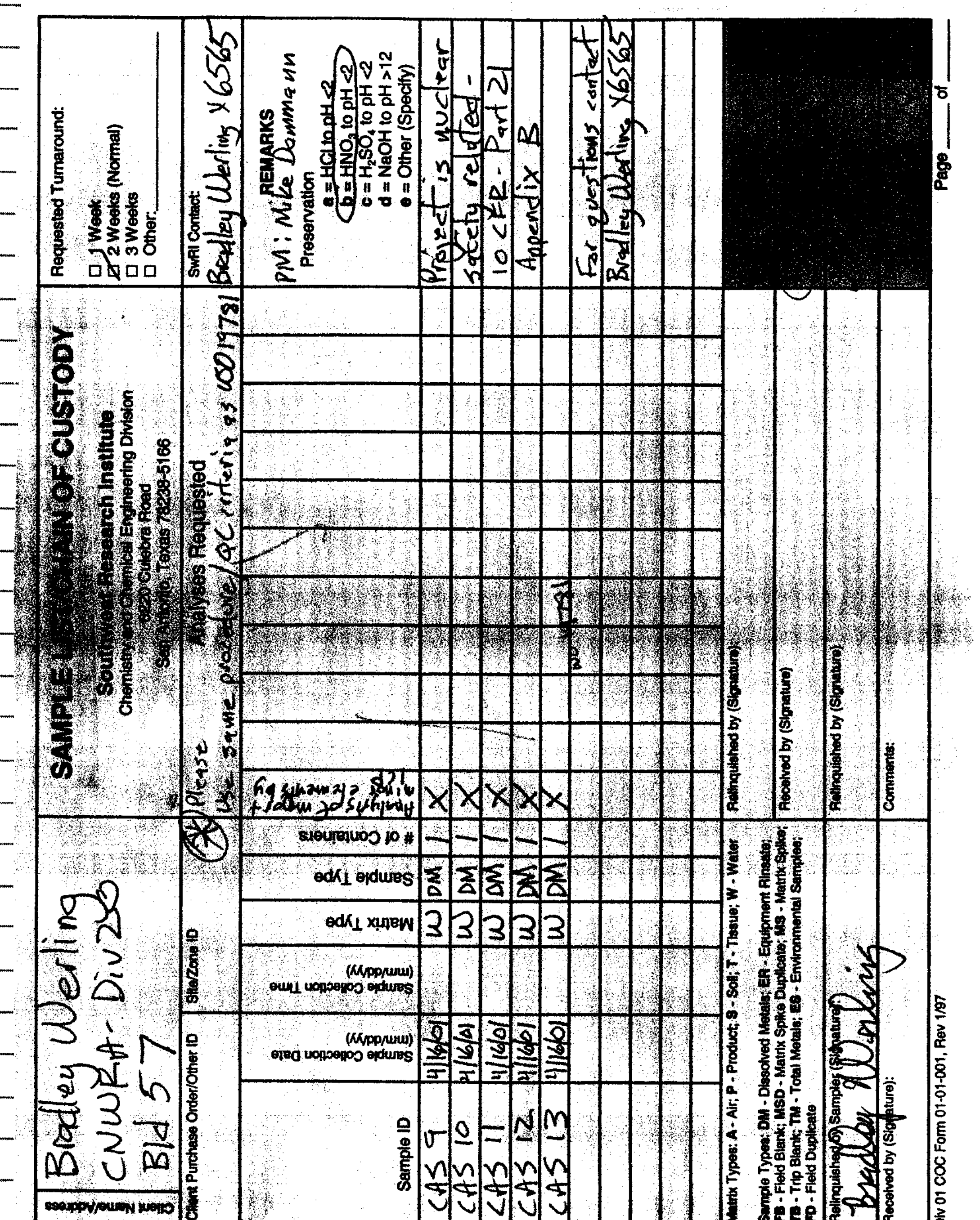
reagents- calcite samples CAS9-12 (309/255)
- 1.0N HNO_3 (309/254)

Samples were previously oven dried in 100mL Vol flask. These were diluted to mark with 1.0N HNO_3

15mL aliquots of these samples were transferred to 15mL poly propylene bottles and delivered to Division 01 for analysis. In addition to these 4 samples, a 15mL aliquot of 1000ppm Ca std* was also delivered as CAS13 for analysis.

*Ca 1000ppm Std = Spex Certiprep Cat# PLC.A2-2X, lot # 7-114CA, open 2/21/2001.

4/16/2001 cont BAW — Below is a copy of the Sample Chain of Custody for samples from 309/255



SAMPLE CHAIN OF CUSTODY

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

Requested Turnaround: ☐ 1 Week ☑ 2 Weeks (Normal) ☐ 3 Weeks ☐ Other:

SwRI Contact: Bradley Werling X6565

Client Name/Address: Bradley Werling CNWRA - Div 20 Bld 57

Analyses Requested: (*) Please use same procedure/QC criteria as 20-01978

REMARKS: PM: Mike Dammann

Preservation: a = HCl to pH <2; b = HNO_3 to pH <2; c = H_2SO_4 to pH <2; d = NaOH to pH >12; e = Other (Specify)

Project is nuclear safety related - 10 CFR Part 21 Appendix B

For questions contact Bradley Werling X6565

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	Analytical method by ICP (*)
CAS 9	4/16/01		W	DM	1	X
CAS 10	4/16/01		W	DM	1	X
CAS 11	4/16/01		W	DM	1	X
CAS 12	4/16/01		W	DM	1	X
CAS 13	4/16/01		W	DM	1	X

Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water

Sample Types: DM - Dissolved Metals; ER - Equipment Rinsate; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; ES - Environmental Samples; FD - Field Duplicate

Relinquished by (Signature): Bradley Werling

Received by (Signature):

Relinquished by (Signature):

Received by (Signature):

Comments:

Page ___ of ___

Div 01 COC Form 01-01-001, Rev 1/97

4/16/2001 cont BAW

Target Concentration 0.32 N NaOH

To make 50 mL of 0.32 N NaOH,

$$\frac{(0.64\,g\ NaOH)\left(\frac{1\,mol}{40.00\,g\ NaOH}\right)}{0.050\,L} = 0.32\,N\ NaOH$$

reagents: NaOH pellets - Fisher S 318-1, lot# 976631
nanopure water.

balance: Mettler AE240 SN# 101237

Added NaOH pellets to 50 mL vol flask + diluted to mark with nanopure H_2O.

Mass of flask (g)	Mass of flask + NaOH (g)	Mass of NaOH (g)
7.0045	7.6465	0.642

$$\text{Normality of Soln} = \frac{(0.642\,g)\left(\frac{1\,mol}{40.00\,g}\right)}{0.050\,L} = 0.321\,N\ NaOH$$

4/23/2001 BAW — Preparation of calcite solutions

Calcite (Fisher cat# C64-500, lot 986396) weighed in weighing boat and transferred dry to polycarbonate container (Mettler PM 4600)

Container ID	Container Size (L)	Mass of Calcite (g)
309/257 A	1	20.00
309/257 B	1	20.00
309/257 C	1	20.00
309/257 D	1	20.00

4/23/2001 cont BW

ID

Container ID	BW 2-27-02 Container Size (L)	Calcite Mass (g)
309/258 A	2	30.00
309/258 B	2	30.00
309/258 C	2	30.00
309/258 D	2	30.00
309/258 E	2	30.00
309/258 F	2	30.00
309/258 G	2	30.00
309/258 H	2	30.00

Target Concentration 1.0 M Ca $Ca(ClO_4)_2 \cdot 4H_2O$

To make 1 Liter of 1.0 M Ca from $Ca(ClO_4)_2 \cdot 4H_2O$;

$$311.04\,g\ Ca(ClO_4)_2 \cdot 4H_2O \left(\frac{1\ mol\ Ca}{311.04\,g\ Ca(ClO_4)_2 \cdot 4H_2O}\right)$$

1 Liter → 1.0 M Ca

reagents = calcium perchlorate tetrahydrate. Aldrich cat# 401420, lot# 08524MI, rec 4/9/2001, opened 4/23/2001, nanopure water

Approximately 400mL of nanopure water was added to a 1000mL polypropylene container. This was weighed with the Mettler PM4600. About 311.04g of $Ca(ClO_4)_2 \cdot 4H_2O$ was added and reweighed. This was transfered to a 1000mL Vol. flask and diluted to mark with nanopure water.

Mass of container + 400mL water (g)	Mass of container + water + $Ca(ClO_4)_2 \cdot 4H_2O$ (g)
553.90	867.1

Soln labeled 309/258 Ca

4/24/2001 BAW

Calculated Ca conc of 309/258 Ca

$$313.2\,g\ Ca(ClO_4)_2 \cdot 4H_2O \left(\frac{40.08\,g\ Ca}{311.04\,g\ Ca(ClO_4)_2 \cdot 4H_2O}\right)\left(\frac{10^6\,\mu g}{g}\right)\left(\frac{1\,L}{10^3\,mL}\right)$$

$= 4.036 \times 10^4\ \mu g/mL$

Target Concentration Ca @ 20 ppm

To make 100mL of 20ppm Ca from 1000ppm stock,

$$\frac{(2\,mL)(1000\,ppm)}{100\,mL} = 20\ ppm$$

Reagents - Calcium std - 1000ppm - Spex Certiprep cat# PLCA2-2X, lot# 7-114CA, rec 1/31/01, opened 2/21/2001, nanopure water

Added 2mL of 1000ppm Ca (vol pipet) to a 100mL volumetric flask and diluted to mark with nanopure water. Labeled 309/259 Ca Std.

Calcium Curve for AA analysis

All final volumes were 50mL (vol flask)
Vol pipets were used to transfer all solns from 0.5 to 25mL. % LaCl is w/w.

Calcium calibration curve

Soln ID	Target Conc of Ca (ppm)	Target Conc of LaCl (%)		Vol (mL) of 309/259 Ca Std (20 ppm Ca)	Vol (ml) of 309/183 (1% LaCl)
Ca1*	4	0.1		10	5
Ca2	2	0.1		5	5
Ca3	1.2	0.1		3	5
Ca4	0.8	0.1		2	5
Ca5	0.4	0.1		1	5
Ca6	0.2	0.1		0.5	5

* AA sensitivity check

4/24/2001 cont BAW

Sample 309/258Ca dilution for AA analysis

DF100 – 1mL (Vol pipet) of 309/258Ca added to a 100 mL vol. flask and diluted to mark with nanopure water

DF 10,000 – 1mL (Vol pipet) of DF 100 (above) added to a 100mL vol flask and diluted to mark with nanopure water.

AA Analysis of Sample 309/258Ca for Calcium

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
Ca-Mg Lamp – 6mV λ = 422.7nm, slit = 0.7nm, high
Air-acetylene flame
Blank = 0.1% La (w/w) 309/190
Integration time = 3 sec

Absorbance Values

ID	Conc (ppm)	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Ca1	4	0.258	0.260	0.259	0.259	0.260
Ca2	2	0.134	0.134	0.134	0.133	0.133
Ca3	1.2	0.080	0.080	0.080	0.080	0.080
Ca4	0.8	0.055	0.054	0.054	0.055	0.054
Ca5	0.4	0.028	0.028	0.028	0.028	0.028
Ca6	0.2	0.016	0.016	0.016	0.016	0.015
309/258CaDF20000	ng 0.136	~~0.135~~ 0.134	0.134	0.135	0.134	0.134
309/258CaDF50000	ng	0.055	0.056	0.055	0.055	0.055
Ca4	0.4	0.055	0.055	0.055	0.055	0.055

Az max = 0.001 Az normal = 0.000

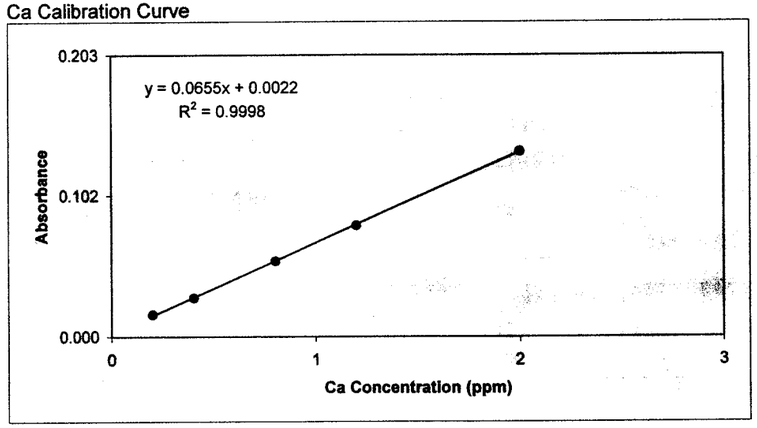
4/24/2001 cont BAW

Calcium Concentration in Sample 309/258Ca

Ca Std Data

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
Ca6	0.2	0.016	0.016	0.016	0.016	0.016	0.0160
Ca5	0.4	0.028	0.028	0.028	0.028	0.028	0.028
Ca4	0.8	0.055	0.054	0.054	0.055	0.054	0.0544
Ca3	1.2	0.080	0.080	0.080	0.080	0.080	0.08
Ca2	2	0.134	0.134	0.134	0.133	0.133	0.1336

Ca Calibration Curve



Ca Sample Data

Solution ID	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
309/258Ca/DF20000	0.136	0.134	0.135	0.134	0.134	0.1346
309/258Ca/DF50000	0.055	0.055	0.056	0.055	0.055	0.0552

All calibration verification samples analyzed with the samples were within 5%

Ca Summary Data

Solution ID	Measured conc of diluted solution (ppm)	AA conc for solution (ppm)	AA conc for solution (Molarity)
309/258Ca/DF20000	2.0214	40427	1.01
309/258Ca/DF50000	0.8092	40458	1.01

Gravimetric concentration for solution calculated at 1.007M

4/25/2001 BW

Target Conc 0.1N NaOH

Dilut-it 4687-01, lot# H33121 NaOH transfered to 1L for 0.1N solution

Various calcite solutions in equilibrium with atmospheric PCO_2.

Calculated additions of calcium perchlorate, sodium perchlorate, and sodium hydroxide will be used to bring the solution chemistry close to equilibrium conditions. Solid calcite (30 g) will be added to each solution (final volume at two liters) and air will be bubbled to ensure equilibrium with atmosphere.

Reagents: Sodium perchlorate (anhydrous): Mallinckrodt catalog # 1190, lot # KTKJ
dried at 44°C for over 24 hours
1.01M Ca from Calcium perchlorate solution 309/258Ca
0.1M Sodium hydroxide solution 309/262
Calcite from 309/258A-H
Nanopure water

The appropriate amount of sodium perchlorate was weighed (Mettler AE 240) in a weighing boat and transferred to a 600mL beaker. The beaker with sodium perchlorate was placed on the Mettler PM4600 balance and tared to zero. Next, the appropriate amount of sodium hydroxide was added by weight (Mettler PM4600). Then the appropriate amount of calcium perchlorate was added. For solutions 309/258A-E, the solution was tared to zero (Mettler PM4600) and the calcium perchlorate was added by weight. For solutions 309/258F-H, the calcium perchlorate was added by volume (micropipette). The solution in the 600mL beaker was transferred to a 2L volumetric flask and diluted to mark with nanopure water. This 2L solution was transferred to a 2L polycarbonate container that contained 30g of calcite (see table for ID of calcite containers). Gas bubblers were inserted into each container to ensure equilibrium with atmosphere.

Target pH Of solution	Mass of NaClO4 (g)	Mass of 0.1 NaOH (g)	Amount of $Ca(ClO_4)_2\ 4H_2O$	Calcite container ID
7.25	2.3968	4.41	408.52 g	309/258A
7.50	2.3826	5.40	86.71 g	309/258B
7.75	24.3670	10.00	27.49 g	309/258C
8.00	24.2925	16.00	8.19 g	309/258D
8.25	24.1737	26.02	2.58 g	309/258E
8.50	24.3682	42.01	0.94 mL	309/258F
8.75	24.3666	95.99	0.28 mL	309/258G
9.00	24.3642	185.10	0.09 mL	309/258H

4/30/2001 BAW

Results of major/minor element analysis by ICP of calcite samples sent to division 01 for analysis. Samples from 309/255.

SOUTHWEST RESEARCH INSTITUTE

SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 9

Lab Name: Southwest Research Institute
Client: Division 20
Lab Code: SwRI
Date Received: 04/16/01
Matrix: Liquid
Project No.: 20.01402.871
Lab System ID: 159539
Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	0.054	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.071	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	4050	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.069	0.005
Iron	0.103	0.05
Lanthanum	<0.005	0.005
Lead	0.009	0.005
Lithium	<0.005	0.005
Magnesium	0.051	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.126	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	0.065	0.05
Silver	<0.005	0.005
Sodium	0.366	0.1
Strontium	1.12	0.005
Sulfur	0.406	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.006	0.005
Zirconium	<0.005	0.005

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 10

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 159535

Client: Division 20

Date Received: 04/16/01

Project No.: 20.01402.871

Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	0.075	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.061	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	3553	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.053	0.005
Iron	0.088	0.05
Lanthanum	<0.005	0.005
Lead	0.009	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.122	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	0.065	0.05
Silver	<0.005	0.005
Sodium	0.312	0.1
Strontium	0.969	0.005
Sulfur	0.391	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.009	0.005
Zirconium	<0.005	0.005

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 11

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 159536

Client: Division 20

Date Received: 04/16/01

Project No.: 20.01402.871

Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	0.055	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.064	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	3705	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.048	0.005
Iron	0.096	0.05
Lanthanum	<0.005	0.005
Lead	0.010	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.152	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	0.053	0.05
Silver	<0.005	0.005
Sodium	0.307	0.1
Strontium	1.02	0.005
Sulfur	0.466	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.010	0.005
Zirconium	<0.005	0.005

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID
CAS 12

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Liquid
Lab System ID: 159537

Client: Division 20
Date Received: 04/16/01
Project No.: 20.01402.871
Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.066	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	3817	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.088	0.005
Iron	0.082	0.05
Lanthanum	<0.005	0.005
Lead	0.010	0.005
Lithium	<0.005	0.005
Magnesium	0.052	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	0.149	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	0.302	0.1
Strontium	1.05	0.005
Sulfur	0.434	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.009	0.005
Zirconium	<0.005	0.005

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
DUPLICATE SUMMARY

Sample ID
CAS 12

Lab Name: Southwest Research Institute
Lab Code: SwRI
Matrix: Liquid
Lab System ID: 159537

Client: Division 20
Date Received: 04/16/01
Project No.: 20.01402.871
Work Order: 19948

Analysis	Sample Result (mg/L)	Duplicate Result (mg/L)	RPD
Aluminum	<0.05	<0.05	0.00%
Antimony	<0.05	<0.05	0.00%
Arsenic	<0.01	<0.01	0.00%
Barium	0.066	0.066	0.30%
Beryllium	<0.005	<0.005	0.00%
Bismuth	<0.01	<0.01	0.00%
Boron	<0.05	<0.05	0.00%
Cadmium	<0.005	<0.005	0.00%
Calcium	3817	3840	0.58%
Chromium	<0.005	<0.005	0.00%
Cobalt	<0.005	<0.005	0.00%
Copper	0.088	0.081	8.30%
Iron	0.082	0.096	15.47%
Lanthanum	<0.005	<0.005	0.00%
Lead	0.010	0.010	5.09%
Lithium	<0.005	<0.005	0.00%
Magnesium	0.052	0.052	0.73%
Manganese	<0.005	<0.005	0.00%
Molybdenum	<0.005	<0.005	0.00%
Nickel	<0.005	<0.005	0.00%
Palladium	<0.005	<0.005	0.00%
Phosphorus	0.149	0.126	16.48%
Potassium	<0.1	<0.1	0.00%
Selenium	<0.1	<0.1	0.00%
Silicon	<0.05	0.050	200.00%
Silver	<0.005	<0.005	0.00%
Sodium	0.302	0.305	0.83%
Strontium	1.05	1.05	0.50%
Sulfur	0.434	0.408	6.23%
Thallium	<0.1	<0.1	0.00%
Thorium	<0.01	<0.01	0.00%
Tin	<0.1	<0.1	0.00%
Titanium	<0.01	<0.01	0.00%
Tungsten	<0.01	<0.01	0.00%
Uranium	<0.15	<0.15	0.00%
Vanadium	<0.005	<0.005	0.00%
Yttrium	<0.005	<0.005	0.00%
Zinc	0.009	0.009	4.80%
Zirconium	<0.005	<0.005	0.00%

4/30/2001 cont BAW

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Sample ID: CAS 13

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 04/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 159538
Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	0.016	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	1008	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	0.031	0.005
Iron	<0.05	0.05
Lanthanum	0.180	0.005
Lead	0.094	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	0.059	0.005
Sulfur	0.290	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.011	0.005
Zirconium	<0.005	0.005

4/30/2001 cont BAW

SOUTHWEST RESEARCH INSTITUTE
MATRIX SPIKE SUMMARY

Sample ID: CAS 13

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 04/16/01

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: 159538
Work Order: 19948

Analysis	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery
Aluminum	<0.05	2.16	2.00	108.0%
Antimony	<0.05	0.529	0.500	105.7%
Arsenic	<0.01	2.13	2.00	106.7%
Barium	0.016	2.09	2.00	103.7%
Beryllium	<0.005	0.046	0.050	92.4%
Bismuth	NA	NA	NA	NA
Boron	NA	NA	NA	NA
Cadmium	<0.005	0.050	0.050	99.8%
Calcium	1008	1420	400	102.9%
Chromium	<0.005	0.199	0.200	99.3%
Cobalt	<0.005	0.506	0.500	101.1%
Copper	0.031	0.267	0.250	94.3%
Iron	<0.05	0.961	1.00	96.1%
Lanthanum	NA	NA	NA	NA
Lead	0.094	0.618	0.500	104.8%
Lithium	NA	NA	NA	NA
Magnesium	<0.05	20.7	20	103.3%
Manganese	<0.005	0.501	0.500	100.3%
Molybdenum	NA	NA	NA	NA
Nickel	<0.005	0.492	0.500	98.4%
Palladium	NA	NA	NA	NA
Phosphorus	NA	NA	NA	NA
Potassium	<0.1	27.0	20	134.9%
Selenium	<0.1	2.14	2.00	106.8%
Silicon	NA	NA	NA	NA
Silver	<0.005	0.050	0.050	100.0%
Sodium	<0.1	23.2	20	115.8%
Strontium	NA	NA	NA	NA
Sulfur	NA	NA	NA	NA
Thallium	<0.1	2.21	2.00	110.6%
Thorium	NA	NA	NA	NA
Tin	NA	NA	NA	NA
Titanium	NA	NA	NA	NA
Tungsten	NA	NA	NA	NA
Uranium	NA	NA	NA	NA
Vanadium	<0.005	0.513	0.500	102.7%
Yttrium	NA	NA	NA	NA
Zinc	0.011	0.513	0.500	100.5%
Zirconium	NA	NA	NA	NA

NA- Not Applicable.

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
LABORATORY CONTROL SAMPLE

Sample ID: LCSW

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: NA

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: NA
Work Order: 19948

Analysis	Sample Result (mg/L)	True Value (mg/L)	Recovery
Aluminum	2.00	2.00	99.9%
Antimony	0.506	0.500	101.1%
Arsenic	2.05	2.00	102.6%
Barium	2.02	2.00	100.9%
Beryllium	0.050	0.050	99.1%
Bismuth	NA	NA	NA
Boron	NA	NA	NA
Cadmium	0.051	0.050	101.1%
Calcium	20.2	20	101.2%
Chromium	0.202	0.200	100.9%
Cobalt	0.504	0.500	100.7%
Copper	0.250	0.250	99.9%
Iron	0.977	1.00	97.7%
Lanthanum	NA	NA	NA
Lead	0.514	0.500	102.9%
Lithium	NA	NA	NA
Magnesium	20.1	20	100.5%
Manganese	0.500	0.500	99.9%
Molybdenum	NA	NA	NA
Nickel	0.500	0.500	100.0%
Palladium	NA	NA	NA
Phosphorus	NA	NA	NA
Potassium	17.0	20	85.2%
Selenium	2.13	2.00	106.5%
Silicon	NA	NA	NA
Silver	0.049	0.050	98.4%
Sodium	18.1	20	90.5%
Strontium	NA	NA	NA
Sulfur	NA	NA	NA
Thallium	2.14	2.00	106.9%
Thorium	NA	NA	NA
Tin	NA	NA	NA
Titanium	NA	NA	NA
Tungsten	NA	NA	NA
Uranium	NA	NA	NA
Vanadium	0.506	0.500	101.2%
Yttrium	NA	NA	NA
Zinc	0.514	0.500	102.9%
Zirconium	NA	NA	NA

NA- Not Applicable.

4/30/2001
cont BAW

SOUTHWEST RESEARCH INSTITUTE
BLANK SUMMARY

Sample ID: PBW

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: NA

Matrix: Liquid
Project No.: 20.01402.871

Lab System ID: NA
Work Order: 19948

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.05	0.05
Antimony	<0.05	0.05
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	<0.1	0.1
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.05	0.05
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.05	0.05
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.1	0.1
Silicon	<0.05	0.05
Silver	<0.005	0.005
Sodium	<0.1	0.1
Strontium	<0.005	0.005
Sulfur	<0.05	0.05
Thallium	<0.1	0.1
Thorium	<0.01	0.01
Tin	<0.1	0.1
Titanium	<0.01	0.01
Tungsten	<0.01	0.01
Uranium	<0.15	0.15
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

4/30/2001 cont BAW

Analysis of Calcium in Calcite Samples

Sample ID[1]	Calcite Mass dry[2] (g)	Solution Vol (mL)	Calculated Ca Conc[3] (ppm)	Div 01 results[4] Ca (ppm)	Percent Difference
CAS9	1.0277	100	4115	4050	-1.59
CAS10	0.8937	100	3579	3553	-0.72
CAS11	0.9409	100	3768	3705	-1.67
CAS12	0.9750	100	3904	3817	-2.24
CAS12Dup	0.9750	100	3904	3840	-1.65

Sample ID[4]	Vol (mL) of 1000 ppm Ca	Final Volume (mL)	Calculated Conc (ppm)	Analytical Conc (ppm)	Percent Difference
CAS13	15	15	1000	1008	0.80

[1] See 309/255 for sample ID information

[2] See 309/255 for dry weight information

[3] See 309/255 for sample calculation

[4] See 309/263-271 for Div 01 results

5/7/2001 BAW

pH Analysis of $CaCO_3$ solutions 309/258A-H

Orion model 920A pH meter ~ serial #039522
Orion model 8103 Ross combination electrode (3C) with ATC probe
Calibrated with
pH 5 buffer soln (5-7-2001) Fisher SB102-500, lot# 986990-24
pH 10 buffer soln (5-7-2001) Fisher SB116-500, lot# 987055-24
Temp = 21.7°C - set calibration at pH 5.00, 10.06

Slope = 98.6 %

Challenged with pH 7 buffer soln (5-7-2001) Fisher cat# SB108-500, lot# 000749-24

5/7/2001 cont BAW

Soln ID	pH Reading	Target pH
309/258A	7.10	7.25
309/258B	7.43	7.50
309/258C	7.64	7.75
309/258D	7.76	8.00
309/258E	8.14	8.25
309/258F	8.37	8.50
309/258 ~~XG~~ 5/7/01 BAW	9.70	8.75
309/258H	9.00	9.00
pH 7 buffer	7.03	7.02
309/2586*	9.80	8.75

* different aliquot out of 2L solution

5/8/2001 BAW

pH of solution 309/2586 was higher than expected. Noticed airation of this 2L container was lower than in the other containers. Added another pump and reconfigured system so airation was better. Took second aliquot of 309/2586* (309/273) and stirred on magnetic stirrer (setting ~4) for 15. Remeasured pH to see if airation lowered pH.

5/8/2001 BAW

5/8/2001 cont BAW

pH Analysis of 309/2586*

Orion model 920A pH meter - serial # 039522
orion model 8103 Ross combination electrode (3C) with ATC probe.
Calibrated with
pH 7 buffer (5/7/01) Fisher SB108-500 lot 000749-24
pH 10 buffer (5/7/01) Fisher SB116-500 lot 987055-24
Temp at 20.4°C ; values 7.02 and 10.07 used

Slope = 98.4%

Solution ID	pH reading	target pH
309/2586*	8.83	8.75
pH 7 buffer	7.03	7.02

5/10/2001 BAW

Aspiration tubing for solns 309/258 F+G was disconnected when inspection made at 0820. They were connected + running when I departed at 1700 on 5/9/2001. Reconnected so aeration would begin again.

pH analysis of $CaCO_3$ solutions 309/258A to H

5/10/2001 cont BAW

Orion model 920A pH meter serial # 039522
orion model 8103 Ross combination electrode (3C)
with ATC probe
calibrated with
pH 7 buffer soln (5-7-01) Fisher SB108-500 lot# 000749-24
pH 10 buffer soln (5-7-01) Fisher SB116-500 lot# 987055-24

Temp = 21.3°C ; set calibration at 7.02 and 10.06
slope = 99.5%

5/10/2001 cont BAW

Challenged with pH 9 buffer soln (5-10-01) Fisher SB114-500 lot # 007377-24

SOLN ID	pH reading	Target pH
309/258A	7.13	7.25
309/258B	7.41	7.50
309/258C	7.68	7.75
309/258D	7.94	8.00
309/258E	8.19	8.25
309/258F	8.41	8.50
309/258G	8.75	8.75
309/258H	8.99	9.00
pH 9 buffer soln	9.10	~~9.10~~ 9.05
pH 7 buffer soln	7.10	5/10/01 ~~7.10~~ 7.02

5/14/2001 BAW

pH Analysis of Eq sampling ~~of Eq~~ Np spiked solns

Orion model 920A pH meter serial # 039522
Orion model 8103 Ross combination electrode (3C)
with ATC probe
Calibrated with
pH 5 buffer soln (5/7/01) Fisher SB102-500 lot# 986990-24
pH 7 buffer soln (5/7/01) Fisher SB108-500 lot# 000749-24
pH 10 buffer soln (5/7/01) Fisher SB116-500 lot# 987055-24

5/14/2001 cont BAW

Temp = 20.0°C setpts 5.00, 7.03, and 10.07

slope @ 99.3%

Soln ID	pH Reading	pH Target
pH7	7.04	7.03
NpCA1 H1	8.97	9.00
NpCA1 G2	8.76	8.75
NpCA1 H3	~~8.78~~ BAW 5/14/01 9.03	9.00
NpCA2 G1	8.78	8.75
NpCA2 G3	8.79	8.75
NpCA2 H2	9.05	9.00
pH7	7.08	7.03

5/14/01 BAW

14 May 01 PD

Data tables for experiment initial conditions. Several experiments will be conducted that will attempt to quantify the sorption of ^{237}Np on calcite. Expts will utilize a range of pH values from 7.25 to 9 and various M/V ratios of calcite mass to solution volume. Expts will be conducted in polycarbonate test tubes using pre-equilibrated solutions and calcite.

Np-calcite sorption experiments:

	[Np]	pH range	calcite mass
Np CA1 1	~2x10^{-6}M	7.25 - 9 in 0.25 steps	0.5 g
Np CA1 2	"	"	0.5g
Np CA1 3	"	"	0.5g
Np CA2 1	"	"	0.1g
Np CA2 2	"	"	0.1g
Np CA2 3	"	"	0.1g
Np CA3 1	"	"	0.2g
Np CA3 2	"	"	0.2g

The NpCA1 and NpCA2 series will have calcite added to solutions to equilibrate prior to addition of Np spike. The NpCA3 series will be spiked with Np prior to addition of calcite.

Solutions were prepared as noted starting on p.257 of this notebook. Np spike has a concentration of ~0.32N HNO_3 so a similar amount of 0.32 N NaOH will be added to each solution as a buffer/neutralizer.

Initial weights and addition of solutions are noted on each data sheet. Dates are correct as indicated on each sheet.

All weights were recorded using the Mettler AE240 balance.

Mettler AE240

10 May 01 PB

Container and sample weight table, for Np-calcite sorption experiments.

Table inserted 14 May 01 PB

Sample Container number	Container wt. (g)	Container + calcite (g)	Container + Ca solution
NpCA1 A1	21.9648	22.4557	42.2886
NpCA1 B1	22.2028	22.7075	42.4189
NpCA1 C1	22.3758	22.8734	42.0806
NpCA1 D1	22.3469	22.8536	42.8186
Np CA1 E1	22.4004	22.9019	41.7964
NpCA1 F1	22.3664	22.8657	43.9692
Np CA1 G1	21.9650	22.4659	43.1624
Np CA1H1	22.1438	22.6443	42.6262
NpCA1 A2	22.1692	22.6643	43.1280
NpCA1 B2	22.1155	22.6138	42.7173
NpCA1 C2	22.1060	22.6019	42.7204
NpCA1 D2	22.1377	22.6349	41.7394
NpCA1 E2	22.2022	22.6981	42.7790
NpCA1 F2	22.1684	22.6648	42.1282
NpCA1G2	22.2835	22.7797	43.1858
NpCA1 H2	22.0598	22.5606	42.4389

w/cap

Mettler AE240

10 May 01 PB

Container and sample weight table for Np-calcite sorption experiments.

sample container number	Container wt. (g)	Container + calcite (g)	Container + Ca solution
Np CA1 A3	22.4904	22.9862	43.5146
Np CA1 B3	22.1641	22.6628	42.4531
Np CA1 C3	22.2506	22.7507	42.5677
Np CA1 D3	22.4125	22.9078	42.4814
Np CA1 E3	22.1423	22.6402	42.7501
Np CA1 F3	22.4271	22.9246	42.6883
Np CA1 G3	21.9223	22.4221	42.9843
Np CA1 H3	22.1214	22.6203	42.5919

calcite solutions from stock solutions A-G~~H~~H were PB 5/14/01 added to each test tube by withdrawing ~20 mL in a PP syringe and discharging the solution through a 25 mm 0.2 μm nuclepore filter w/assembly. Filter was (and housing) changed for each solution A-H.

table inserted 14 May 01 PB

10 May 01 Mettler AE 240

PB

Container and sample weight table for Np-calcite sorption experiments.

sample container number	container wt. (g)	container + calcite (g)	container + Ca solution
NpCA2A1	22.2456	22.3434	42.8142
NpCA2B1	22.0440	22.1413	42.7932
NpCA2C1	22.1205	22.2190	42.1114
NpCA2D1	22.0492	22.1457	41.9851
NpCA2E1	22.0423	22.1448	42.2057
NpCA2F1	22.2383	22.3334	42.0911
NpCA2G1	22.0923	22.1908	42.6828
NpCA2H1	21.9215	22.0209	41.6120
NpCA2A2	22.0190	22.1166	42.5689
NpCA2B2	22.0292	22.1248	41.8724
NpCA2C2	22.0061	22.1026	42.1524
NpCA2D2	22.1562	22.2547	41.8279
NpCA2E2	22.3482	22.4458	42.5337
NpCA2F2	22.0486	22.1451	41.8627
NpCA2G2	22.3079	22.4061	42.5789
NpCA2H2	22.0067	22.1054	41.9917

table inserted 14 May 01 PB

10 May 01 Mettler AE 240

PB

Container and sample weight table for Np-calcite sorption experiments.

sample container number	container wt. (g)	container + calcite (g)	container + Ca solution
NpCA2A3	21.9986	22.0953	42.5752
NpCA2B3	22.2490	22.3462	41.9798
NpCA2C3	22.3576	22.4541	42.4080
NpCA2D3	21.9692	22.0671	42.0874
NpCA2E3	21.9700	22.0675	42.1249
NpCA2F3	22.2822	22.3841	41.7676
NpCA2G3	22.0524	22.1521	42.8940
NpCA2A3	22.0899	22.1876	41.8535

PB

5/14/01

table inserted 5/14/01 PB

10 May 01 PB

Mettler AE240

Container and sample weight table for Np-calcite sorption experiments.

sample container number	container wt. (g)	container + calcite (g)	container + Cu solution
NpCA3A1	22.1596	22.3917	43.0047
NpCA3B1	22.2997	22.3932	42.2407
NpCA3C1	22.2487	42.0815 ⊛ (14May01 ↓)	42.3394
NpCA3D1	22.2734	41.8484	42.0964
NpCA3E1	22.2460	42.3321	42.5676
NpCA3F1	22.0036	41.5862	41.8600
NpCA3G1	22.3865	42.1762	42.4270
NpCA3H1	22.0618	41.5050	41.7657
NpCA3A2	22.3618	42.3430	42.5958
NpCA3B2	22.1206	41.6436	41.8861
NpCA3C2	22.1396	41.7771	42.0196
NpCA3D2	22.2771	42.0123	42.2533
NpCA3E2	22.2115	42.1950	42.4516
NpCA3F2	22.1069	41.6004	41.8581
NpCA3G2	22.1259	42.1703	42.4237
NpCA3H2	22.3242	41.6326	41.9153

14May01 PB ⊛ solutions NpCA3C1 – NpCA3H2 had calcite added after addition of Np spike. NpCA3A1 and NpCA3B1 had calcite added prior to addition of Cu solution and Np spike.

Following equilibration w/ calcite and solution, Np spike was added on 14May01 to CA1 and CA2 series. Spike added on 11 May 01 to CA3 series.

pH of some solutions checked as noted (p. 276/309)

Table inserted in reverse order for convenience.

14 May 01 PB

sample container number	container wt. (g)	container wt. after pH meas.	container + Np	container + NaOH
NpCA1A3	43.3639	—	43.4588	43.5595
NpCA1B3	42.3000	—	42.3945	42.4952
NpCA1C3	42.4124	—	42.5074	42.6070
NpCA1D3	42.3237	—	42.4190	42.5187
NpCA1E3	42.5907	—	42.6842	42.7840
NpCA1F3	42.5206	—	42.6172	42.7152
NpCA1G3	42.8127	—	42.9071	43.0070
NpCA1H3	42.4140	42.3852	42.4814	42.5822

14 May 01 PB

14 May 01 PB

Container weight table for Np-calcite sorption experiments.

Sample container number	container wt. (g)	container wt. after pH meas.	container + Np	container + NaOH
NpCA1A1	42.1200	—	42.2140	42.3133
NpCA1B1	42.2454	—	42.3375	42.4374
NpCA1C1	41.9156	—	42.0094	42.1110
NpCA1D1	42.6546	—	42.7485	42.8499
NpCA1E1	41.6275	—	41.7229	41.8230
NpCA1F1	43.8031	—	43.8979	43.9983
NpCA1G1	42.9898	—	43.0852	43.1803
NpCA1H1	42.4738	42.4486	~~43.54~~ 8w 42.5437 5/14/01	42.6444
NpCA1A2	42.9758	—	43.0737 5/14/01 8w 681	43.1692
NpCA1B2	42.5594	—	42.6559	42.7557
NpCA1C2	42.5630	—	42.6591	42.7608
NpCA1D2	41.5804	—	41.6762 5/14/01 PB	~~41.7763~~ 41.7763
NpCA1E2	42.6168	—	42.7131	42.8126
NpCA1F2	41.9574	—	42.0535	42.1463
NpCA1G2	43.0291	43.0127	43.1085	43.2098
NpCA1H2	42.2866	—	42.3826	42.4805

14 May 01 PB

14 May 01 PB

container number	Container wt. (g)	container wt. after pH meas	container + Np	container + NaOH
NpCA2A1	42.6679	—	42.7642	42.8626
NpCA2B1	42.6365	—	42.7313	42.8311
NpCA2C1	41.9560	—	42.0518	42.1513
NpCA2D1	41.8272	—	41.9246	42.0224
NpCA2E1	42.0488	—	42.1436	42.2435
NpCA2F1	41.9326	—	42.0276	42.1278
NpCA2G1	42.5283	42.5134	42.6107	42.7095
NpCA2H1	41.4542	—	41.5488	41.6491
NpCA2A2	42.4214	—	42.5767	42.6163
NpCA2B2	41.7259	—	41.8214	41.9214
NpCA2C2	41.9998	—	42.0941	42.1941
NpCA2D2	41.6757	—	41.7713	41.8714
NpCA2E2	42.3699	—	42.4646	42.5650
NpCA2F2	41.7062	—	41.8013	41.9017
NpCA2G2	42.4258	—	42.5215	42.6213
NpCA2H2	41.8376	41.8217	41.9182	42.0185

14 May 01 PB

container number	container wt. (g)	container wt. after pH meas.	container + Np	container + NaOH
NpCA2A3	42.4368	—	42.5321	42.6319
NpCA2B3	41.8388	—	41.9347	42.0340
NpCA2C3	42.2606	—	42.3563	42.4578
NpCA2D3	41.9397	—	42.0347	42.1348
NpCA2E3	41.9750	—	42.0701	42.1712
NpCA2F3	41.6039	—	41.6989	41.7981
NpCA2G3	42.7506	42.7330	42.8298	42.9294
NpCA2H3	41.7038	—	41.7982	41.8989

For NpCA3 series, the 100 µL spike of Np (#46A) (notebook p. 301/031). was added prior to addition of calcite. A3 stated previously PB 5/14/01 previously, a 100 µL aliquot of 0.321 N NaOH was also added to neutralize the HNO_3 present in the Np spike and minimize the spike's effects on the co-solutions.

NpCA3A1 and NpCA3B1 had calcite added before spike.

table inserted 14 May 01 PB

11 May 01 PB

Sample Container Number	wt container + Np	wt cont + Np + NaOH
NpCA3A1	43.0995	43.1990
NpCA3B1	42.3340	42.4353
NpCA3C1	42.4357	42.5323
NpCA3D1	42.1949	42.2908
NpCA3E1	42.6647	42.7622
NpCA3F1	41.9536	42.0506
NpCA3G1	42.5240	42.6216
NpCA3H1	41.8586	41.9567
NpCA3A2	42.6886901 PB 5/11/01	42.7890
NpCA3B2	41.9808	42.0797
NpCA3C2	42.1156	42.2144
NpCA3D2	42.3478	42.4467
NpCA3E2	42.5423	42.6403
NpCA3F2	41.9524	42.0509
NpCA3G2	42.5161	42.6142
NpCA3H2	42.0065	42.1049

14 May 01 PB — Samples of each Np CA3 solution were taken for assessment of initial Np concentrations.

11 May 01 PB

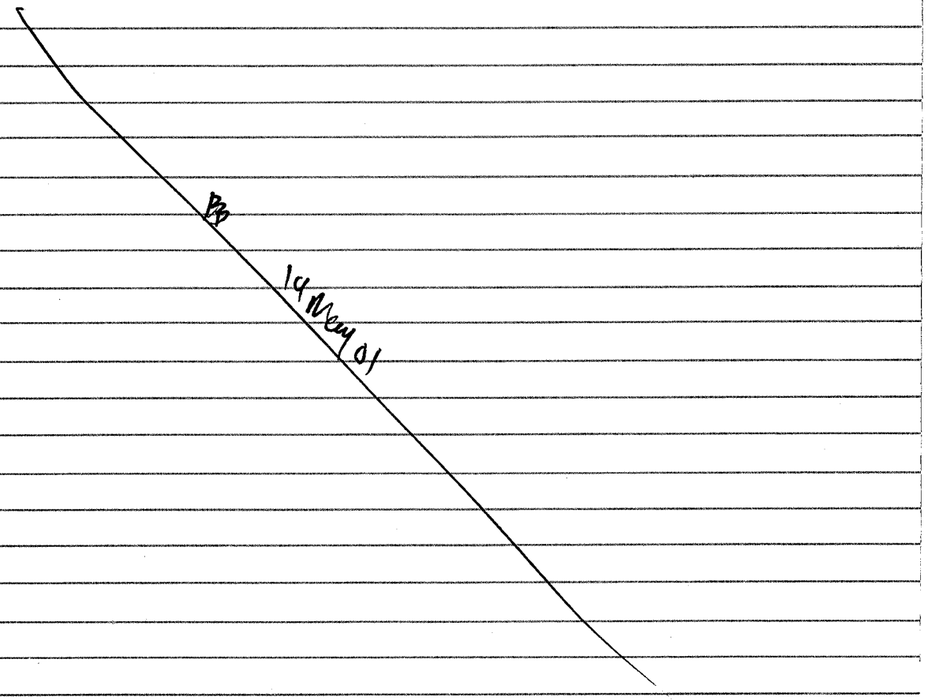
Sampling vials and initial sample weights for Np CA3

vial number	wt. vial (g)	wt. vial + sample (g)
blank	n/a	n/a
Np CA3 A1i	7.3180	7.8333
Np CA3 B1i	7.2806	7.7861
Np CA3 C1i	7.2471	7.7535
Np CA3 D1i	7.2858	7.7912
Np CA3 E1i	7.2828	7.7859
Np CA3 F1i	7.3149	7.8199
Np CA3 G1i	7.2668	7.7710
Np CA3 H1i	7.2895	7.7944
Np CA3 A2i	7.2847	7.7991
Np CA3 B2i	7.2989	7.8068
Np CA3 C2i	7.3064	7.8086
Np CA3 D2i	7.3149	7.8161
Np CA3 E2i	7.3084	7.8118
Np CA3 F2i	7.3422	7.8418
Np CA3 G2i	7.2196	7.7247
Np CA3 H2i	7.3387	7.8449

14 May 01 PB — To sample Np CA3 solutions, the tubes were centrifuged at 2000 rpm (marathon 21K) for 4 min. Then a 500 µL eppendorf pippettor was used to withdraw a sample. 500 µL of 0.1 N HNO_3 was added to each vial to minimize potential sorption onto vial walls. 5 mL of cocktail (Ultima Gold AB) was added to each vial prior to counting.

Following an equilibration period 11 May - 14 May. calcite was added to the Np CA3 series expts.

weight of vials prior to calcite addition are given on the following page.



14 May 01 PB

14 May 01 PB

container number	container wt. (g)	container wt. + calcite
NpCA3A1	42.5558	
NpCA3B1	41.7892	
NpCA3C1	41.8880	
NpCA3D1	41.6545	
NpCA3E1	42.1358	
NpCA3F1	41.3907	
NpCA3G1	41.9796	
NpCA3H1	41.3076	
NpCA3A2	42.1468	
NpCA3B2	41.4469	
NpCA3C2	41.5812	
NpCA3D2	41.8156	
NpCA3G2	41.9976	
NpCA3F2	41.4051	
NpCA3G2	41.9749	
NpCA3H2	41.4369	

↑ see other sheet

p. 292 of fm.s notebook ↓

5/18/2001 BAW

pH and mass Analysis of NpCA1A2-H2 and NpCA2A2-H2

Mettler AE240 electronic balance

Orion model 920A pH meter - serial # 039522
Orion model 8103 Ross combination electrode (3C) w/ ATC probe
Calibrated with
pH 7 soln (5/7/2001) Fisher cat # SB108-500, lot # 000749-24
pH 9 soln (5/10/2001) Fisher cat # SB114-500, lot # 007377-24
Cal temp = 21.0°C, cal set points at 7.03, 9.04
slope of cal curve = 98.6%

SAMPLE ID	Mass(g) before pH measurement	pH	Mass(g) after pH measurement
NpCA1A2	42.9598	7.17	42.9427
NpCA1B2	42.5331	7.58	42.5226
NpCA1C2	42.5407	7.77	42.5209
NpCA1D2	41.5509	8.02	41.5317
NpCA1E2	42.5865	8.21	~~42.5645~~ ~~42.65~~ 42.5645 BAW 5/18/2001
NpCA1F2	41.9191	8.43	41.8845
NpCA1G2	40.5203	8.77	40.4941
NpCA1H2	41.0893	9.06	41.0708
pH 7	—	7.05 (target 7.03)	—

5/18/2001 cont BAW

SAMPLE ID	Mass(g) before pH measurement	pH	Mass(g) after pH measurement
NpCA2A2	42.4032	6.94	42.3636
NpCA2B2	41.7092	7.50	41.6880
NpCA2C2	41.9746	7.69	41.9542
NpCA2D2	41.6460	8.08	41.6248
NpCA2E2	42.3352	8.28	42.3151 ~~42.3157~~ BAW 2-27-02
NpCA2F2	41.6780	8.40	41.6570
NpCA2G2	40.8381	8.71	40.8095
NpCA2H2	40.2776	9.04	40.2580
NpCA2A2 redo of pH 9	—	6.95	—
pH 9	—	9.04 (target 9.03)	—

Balance check: target= 20.0001g, reading 20.00005g

19 May 01 PB

Sampling of Np-calcite solutions for intermediate result analysis.

Np-calcite solutions were weighed and measured for pH as noted above. glass LSA vials are labeled and preweighed. 0.5 mL of 0.1 N HNO_3 is added to each labeled vial prior to weighing. ~0.5mL of selected solutions will be added to each vial. Then vials are reweighed to determine sample size.

18 May 01 PB

Vial	wt vial + HNO_3 (g)	wt vial + sample (g)
blank	N/A	N/A
Np CA1A2 a1	7.7509	8.2628
Np CA1A2 b1	7.8098	8.3241
Np CA1B2 a1	7.7760	8.2730
Np CA1B2 b1	7.7720	8.2717
Np CA1C2 a1	7.8110	8.3131
Np CA1C2 b1	7.8344	8.3378
Np CA1D2 a1	7.8158	8.3139
Np CA1D2 b1	7.7648	8.2679
Np CA1E2 a1	7.7566	8.2550
Np CA1E2 b1	7.8279	8.3319
Np CA1F2 a1	7.7804	8.2806
Np CA1F2 b1	7.7893	8.2926
Np CA1G2 a1	7.7348	8.2383
Np CA1G2 b1	7.7951	8.2993
Np CA1H2 a1	7.7854	8.2872
Np CA1H2 b1	7.8107	8.3158
Np CA2A2 a1	7.7792	8.2904
Np CA2A2 b1	7.7947	8.3075
Np CA2B2 a1	7.7707	8.2812
Np CA2B2 b1	7.7937	8.2965
Np CA2C2 a1	7.7711	8.2707
Np CA2C2 b1	7.7865	8.2903
Np CA2D2 a1	7.7667	8.2660
Np CA2D2 b1	7.8075	8.3112
Np CA2E2 a1	7.7951	8.2952
Np CA2E2 b1	7.8026	8.3050
Np CA2F2 a1	7.8394	8.3388
Np CA2F2 b1	7.7601	8.2625
Np CA2G2 a1	7.8067	8.3072
Np CA2G2 b1	7.8370	8.3393
NpCA2H2 a1	7.8519	8.3520
Np CA2H2 b1	7.8513	8.3532

Solution F added (0.5mL)

wt of solutions sampled after sampling

soln	wt (g)
Np CA1A2	41.9067
Np CA1D2	41.5155
Np CA1C2	41.5049
Np CA1D2	40.5195
Np CA1E2	41.5508
Np CA1F2	40.8699
Np CA1G2	39.8490
Np CA1H2	39.9570
Np CA2A2	41.3273
Np CA2B2	40.6720
Np CA2C2	40.9396
Np CA2D2	40.6105
Np CA2E2	41.3015
Np CA2F2	40.6443
Np CA2 ~~H2~~	39.7968
Np CA2 H2	39.2470

G2

PB 03/08/02

24/May 2001 BAW

Sampling Analysis of Np-Calcite ~~analysis~~ BAW 5/24/01 solutions for results analysis

pH of Np-calcite solutions were measured, Mass of each vial was recorded before and after pH analysis

Mettler AE240 electronic balance
Balance check = 20,0002g (target 20,0001g)

Orion Model 920A pH meter - serial # 039522
Orion Model 8103 Ross combination electrode (3C) w/ ATC probe
Calibrated with
pH 7 soln (5/24/01) Fisher cat# SB108-500, lot# 000749-24
pH 9 soln (5/24/01) Fisher cat# SB114-500, lot# 000737-24

Calibration #1 Cal temp = 19.9 Cal setpts = 7.03, 9.04
Cal slope = 99.0

SAMPLE ID	Mass (g) before pH measurement	pH	Mass (g) after pH measurement
NpCA1A1	41.7056	7.10	41.6731
NpCA1B1	41.7975	7.40	41.7768
NpCA1C1	41.5034	7.68	41.4854
NpCA1D1	42.2329	7.87	42.2047
NpCA1E1	41.1994	8.25	41.1756
NpCA1F1	43.3901	8.36	43.3552
NpCA1G1	40.0772	8.70	40.0503
NpCA1H1	40.7253	9.04	40.7069
NpCA1A2	41.5714	7.05	41.5440
NpCA1B2	41.1622	7.37	41.1408
NpCA1C2	41.1500	7.66	41.1219
NpCA1D2	40.1539	7.91	40.1228
NpCA1E2	41.1846	8.12	41.1554
NpCA1F2	40.5021	8.43	40.4696
NpCA1G2	39.4838	8.68	39.4578
NpCA1H2	39.6181	8.97	39.5926

BAW 5/24/01

24 May 2001 cont BAW

SAMPLE ID	Mass (g) before pH measurement	pH	Mass (g) after pH measurement
NpCA1A3	43.0054	7.09	42.9882
NpCA1B3	41.9170	7.44	41.8863
NpCA1C3	42.0190	7.69	42.0026
NpCA1D3	41.9261	7.95	41.8981
NpCA1E3	42.1949	8.25	42.1788
NpCA1F3	42.1284	8.36	42.1130
NpCA1G3	40.3383	8.72	40.3140
NpCA1H3	39.3451	9.02	39.3268
pH 9	—	9.10	(target 9.04) temp 20.6°C

The pH meter was recalibrated for the next set of solutions
Cal #2 Cal temp = 21.9 Cal setpts = 7.03, 9.04
Cal slope = 99.8

SAMPLE ID	Mass (g) before pH measurement	pH	Mass (g) after pH measurement
NpCA2A1	42.3027	7.09	42.2915
NpCA2B1	42.2282	7.42	42.2060
NpCA2C1	41.5475	7.68	41.5265
NpCA2D1	41.4042	7.87	41.3907
NpCA2E1	41.6250	8.25	41.6013
NpCA2F1	41.5097	8.40	41.4899
NpCA2GH	39.6251	8.74	39.6066
NpCA2H1	40.6623	9.00	40.6442
NpCA2A2	40.9779	6.96	40.9629
NpCA2B2	40.3181	7.44	40.2987
NpCA2C2	40.5743	7.70	40.5539
NpCA2D2	40.2411	7.99	Recalculated 40.5 40.2235
NpCA2E2	40.9275	8.24	40.9092
NpCA2F2	40.2833	8.47	40.2633
NpCA2G2	BAW 5/24/01 ~~40.~~ 39.4270	8.69	39.4089
NpCA2H2	38.9068	9.02	38.8843
pH 9	—	9.03 (target 9.04)	

24 May 2001 cont 'BAW

SAMPLE ID	Mass (g) before pH measurement	pH	Mass (g) after pH measurement
~~NpCA2B~~ BW 24 May 2001			
NpCA2A3	42.0692	6.96	A3 42.0470
NpCA2B3	41.4497	7.52	B3 41.4294
NpCA2C3	41.8533	7.74	D3 41.4979
NpCA2D3	41.5181	8.04	E3 41.5315
NpCA2E3	41.5505	8.24	F3 41.1625
NpCA2F3	41.1805	8.45	G3 39.8966
NpCA2G3	39.9319	8.69	H3 40.3101
NpCA2H3	40.3299	8.99	
pH 7	—	7.09 (target 7.03) temp = 22.4°C	

C3 41.8333

The pH meter was recalibrated for the next set of solutions
Cal #3 Cal temp = 22.5°C Cal Set, 7.03 + 9.05
Cal Slope = 98.0%

SAMPLE ID	Mass (g) before pH measurement	pH	Mass (g) after pH measurement
NpCA3A1	42.0560	6.94	42.0332
NpCA3B1	41.2190	7.51	41.1742
NpCA3C1	41.4780	7.74	41.4454
NpCA3D1	41.2727	7.99	41.2382
NpCA3E1	41.7640	8.25	41.7342
NpCA3F1	40.9538	8.41	40.9264
NpCA3G1	39.4369	8.70	39.4148
NpCA3H1	38.8853	8.98	38.8477
NpCA3A2	41.7981	6.95	41.7643
NpCA3B2	41.1164	7.51	41.0820
NpCA3C2	41.1823	7.66	41.1576
NpCA3D2	41.4414	7.90	41.4115
NpCA3E2	41.6251	8.16	41.5960
NpCA3F2	41.0114	8.48	40.9823
NpCA3G2	40.3183	8.68	40.2941
NpCA3H2	40.0373	8.92	40.0060
pH 7	—	7.05 (target 7.03)	

24 May 01 PB

Following pH measurement and mass measurement of each Np-calcite solution, two 500 µL samples were withdrawn from each container. The samples were placed in labeled 7 mL vials (glass) which had 0.5 mL of 0.1N HNO_3 added and which were pre-weighed. Following the addition of sample, each vial was re-weighed. All solution containers were also re-weighed. Measurements are recorded on the following tables. The mettler AE240 (as noted on p. 294) was used for all mass measurement. An eppendorf 500 µL pipettor w/ disposable tips was used to transfer samples from containers to sample vials.

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA1A1a1	7.8504	8.3637
NpCA1A1b1	7.8350	8.3504
NpCA1B1a1	7.7952	8.2931
NpCA1B1b1	7.7874	8.2904
NpCA1C1a1	7.7993	8.3007
NpCA1C1b1	7.6743	8.1788
NpCA1D1a1	7.9145	8.4153
NpCA1D1b1	7.8597	8.3628
NpCA1E1a1	7.7479	8.2481
NpCA1E1b1	7.7477	8.2518
NpCA1F1a1	7.8649	8.3644
NpCA1F1b1	7.7151	8.2196
NpCA1G1a1	7.8039	8.3066
NpCA1G1b1	7.8062	8.3102
NpCA1H1a1	7.7887	8.2898
NpCA1H1b1	7.7297	8.2327

Samples

24 May 01
PB

Note: Samples for CA1D2 and CA1E2 were placed in wrong vials, vials relabeled and moved in counting rack accordingly.

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA1A2a2	7.8385	8.3504
NpCA1A2b2	7.7938	8.3080
NpCA1B2a2	7.7971	8.2973
NpCA1B2b2	7.7618	8.2640
NpCA1C2a2	7.7447	8.2462
NpCA1C2b2	7.7895	8.2937
NpCA1D2a2	7.7705	8.2683
NpCA1D2b2	7.8135	8.3152
NpCA1E2a2	7.7561	8.2551
NpCA1E2b2	7.7912	8.2917
NpCA1F2a2	7.8518	8.3504
NpCA1F2b2	7.8124	8.3144
NpCA1G2a2	7.8522	8.3527
NpCA1G2b2	7.7968	8.3011
NpCA1H2a2	7.7362	8.2376
NpCA1H2b2	7.7464	8.2491

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA1A3a1	7.8046	8.3165
NpCA1A3b1	7.7499	8.2642
NpCA1B3a1	7.7963	8.2966
NpCA1B3b1	7.7962	8.2977
NpCA1C3a1	7.7759	8.2769
NpCA1C3b1	7.7488	8.2526
NpCA1D3a1	7.7615	8.2602
NpCA1D3b1	7.8209	8.3226
NpCA1E3a1	7.8665	8.3665
NpCA1E3b1	7.8710	8.3734
NpCA1F3a1	7.8243	8.3237
NpCA1F3b1	7.7791	8.2809
NpCA1G3a1	7.8449	8.3424
NpCA1G3b1	7.8381	8.3381
NpCA1H3a1	7.7842	8.2830
NpCA1H3b1	7.7500	8.2487

Samples

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA2A1a1	7.8744	8.3878
NpCA2A1b1	7.8448	8.3598
NpCA2B1a1	7.8955	8.3951
NpCA2B1b1	7.7875	8.2912
NpCA2C1a1	7.7866	8.2890
NpCA2C1b1	7.8601	8.3644
NpCA2D1a1	7.8278	8.3289
NpCA2D1b1	7.8027	8.3033
NpCA2E1a1	7.8420	8.3409
NpCA2E1b1	7.7766	8.2793
NpCA2F1a1	7.7114	8.2118
NpCA2F1b1	7.7893	8.2921
NpCA2G1a1	7.6861	8.1875
NpCA2G1b1	7.7536	8.2560
NpCA2H1a1	7.7353	8.2359
NpCA2H1b1	7.7744	8.2768

24 May 01
PB

Samples

Samples

May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA2A2a2	7.8175	8.3287
NpCA2A2b2	7.7881	8.3016
NpCA2B2a2	7.8190	8.3187
NpCA2B2b2	7.8056	8.3077
NpCA2C2a2	7.8435	8.3444
NpCA2C2b2	7.7804	8.2836
NpCA2D2a2	7.8165	8.3158
NpCA2D2b2	7.8017	8.3023
NpCA2E2a2	7.8123	8.3101
NpCA2E2b2	7.8661	8.3666
NpCA2F2a2	7.8197	8.3191
NpCA2F2b2	7.8204	8.3219
NpCA2G2a2	7.8602	8.3594
NpCA2G2b2	7.9099	8.4113
NpCA2H2a2	7.7854	8.2867
NpCA2H2b2	7.7561	8.2573

300 PB
5/24/01

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA2A3a1	7.7898	8.3010
NpCA2A3b1	7.7320	8.2424
NpCA2B3a1	7.7636	8.2597
NpCA2B3b1	7.7534	8.2514
NpCA2C3a1	7.8211	8.3199
NpCA2C3b1	7.7717	8.2722
NpCA2D3a1	7.7733	8.2720
NpCA2D3b1	7.9213	8.4212
NpCA2E3a1	7.8488	8.3467
NpCA2E3b1	7.8609	8.3609
NpCA2F3a1	7.8409	8.3390
NpCA2F3b1	7.7756	8.2757
NpCA2G3a1	7.7533	8.2524
NpCA2G3b1	7.7674	8.2688
NpCA2H3a1	7.7786	8.2771
NpCA2H3b1	7.7518	8.2520

24 May 01 PB

Samples

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA3A1a1	7.8393	8.3521
NpCA3A1b1	7.7965	8.3113
NpCA3B1a1	7.7120	8.2119
NpCA3B1b1	7.7630	8.2666
NpCA3C1a1	7.7217	8.2240
NpCA3C1b1	7.8145	8.3175
NpCA3D1a1	7.7684	8.2687
NpCA3D1b1	7.7646	8.2655
NpCA3E1a1	7.8508	8.3488
NpCA3E1b1	7.8117	8.3126
NpCA3F1a1	7.8254	8.3253
NpCA3F1b1	7.7213	8.2222
NpCA3G1a1	PB 5/24/01 ~~7.7~~ 7.7226	8.2197
NpCA3G1b1	7.8171	8.3177
NpCA3H1a1	7.8354	8.3340
NpCA3H1b1	7.7811	8.2815

24 May 01 PB

Samples

24 May 01 PB

vial name	wt. vial + acid	wt. vial + sample
NpCA3A2a1	7.7897	8.2980
NpCA3A2b1	7.8179	8.3296
NpCA3B2a1	7.7586	8.2563
NpCA3B2b1	7.8108	8.3103
NpCA3C2a1	7.7513	8.2509
NpCA3C2b1	7.7341	8.2343
NpCA3D2a1	7.7414	8.2413
NpCA3D2b1	7.8411	8.3416
NpCA3E2a1	7.7339	8.2317
NpCA3E2b1	7.8272	8.3286
NpCA3F2a1	7.8317	8.3292
NpCA3F2b1	7.7222	8.2236
NpCA3G2a1	7.7342	8.2327
NpCA3G2b1	7.8012	8.3010
NpCA3H2a1	7.8201	8.3188
NpCA3H2b1	7.8126	8.3147

After sampling

24 May 01 PB

Container name	wt. container after sampling
NpCA1A1	40.6364
NpCA1B1	40.7675
NpCA1C1	40.4704
NpCA1D1	41.1921
NpCA1E1	40.1616
NpCA1F1	42.3420
NpCA1G1	39.0329
NpCA1H1	39.6933
NpCA1A2	40.5097
NpCA1B2	40.1301
NpCA1C2	40.1075
NpCA1D2	39.1137
NpCA1E2	40.1453
NpCA1F2	39.4587
NpCA1G2	38.4434
NpCA1H2	38.5800
NpCA1A3	41.9544
NpCA1B3	40.8766
NpCA1C3	40.9893
NpCA1D3	40.8893
NpCA1E3	41.1664
NpCA1F3	41.1030
NpCA1G3	39.3069
NpCA1H3	38.3203

Samples

24 May 01 PB

24 May 01 PB

After sampling

24 May 01 PB

Container name	wt. container after sampling
NpCA2A1	41.2491
NpCA2B1	41.1988
NpCA2C1	40.5157
NpCA2D1	40.3850
NpCA2E1	40.5957
NpCA2F1	40.4828
NpCA2G1	38.5989
NpCA2H1	39.6368
NpCA2A2	39.9347
NpCA2B2	39.2932
NpCA2C2	39.5463
NpCA2D2	39.2202
NpCA2E2	39.9072
NpCA2F2	39.2565
NpCA2G2	38.4047
NpCA2H2	37.8785
NpCA2A3	41.0226
NpCA2B3	40.4325
NpCA2C3	40.8312
NpCA2D3	40.4965
NpCA2E3	40.5308
NpCA2F3	40.1615
NpCA2G3	38.8931
NpCA2H3	39.3083

After sampling

24 May 01 PB

Container name	wt. container after sampling
NpCA3A1	41.0045
NpCA3B1	40.1706
NpCA3C1	40.4396
NpCA3D1	40.2361
NpCA3E1	40.7338
NpCA3F1	39.9252
NpCA3G1	38.4166
NpCA3H1	37.8467
NpCA3A2	40.7436
NpCA3B2	40.0848
NpCA3C2	40.1571
NpCA3D2	40.4101
NpCA3E2	40.5968
NpCA3F2	39.9625
NpCA3G2	39.2952
NpCA3H2	39.0050

25 May 2001 BAW

Mass of samples removed from some Np-calcite solutions for calcium analysis by A.A.

Mettler AE240 electronic balance used
Balance check = 20.0005 (target 20.0001)

3 or 8 mL aliquots were removed from the solutions and transfered into preweighed 15 mL polypropylene bottles. These 15 mL bottles were then re weighed.

SAMPLE ID	Vol (mL) to be added.	Bottle mass (g) before sample	Bottle mass (g) with sample
NpCA1A1	3	7.0808	10.1175
NpCA1C1	8	7.2433	15.1904
NpCA1E1	8	7.1116	15.0275
NpCA1G1	8	7.0912	15.0316
NpCA1B2	3	7.0633	10.0088
NpCA1D2	3	7.0664	10.0138
NpCA1F2	3	7.1155	10.0622
NpCA1H2	8	7.1135	BAW 5/25/01 to 15.0647
NpCA2A2	3	7.1338	10.1079
NpCA2C2	8	7.1064	15.0532
NpCA2E2	8	7.0698	15.0012
NpCA2G2	8	7.0421	BAW 5/25/01 to 14.9846
NpCA2H2	8	7.0567	15.0153
NpCA3A1	3	7.2551	9.7664
NpCA3B1	3	7.1729	10.1381
NpCA3C1	8	7.1473	15.0868
NpCA3D1	3	7.1398	10.0198
NpCA3E1	8	7.1134	15.0461
NpCA3F1	3	7.0994	9.8087
NpCA3G1	8	7.0227	14.9962
NpCA3H1	8	7.0593	15.0182

03/08/02 Entry and correction review. PB

9-19-01
BAW

End of entries into notebook 309

RT studies continued in notebook 463

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9-19-01
BAW

end. of notebook - entries continued in scientific notebook # 463

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

E.C. Reed
3/27/2002

ADDITIONAL INFORMATION FOR SCIENTIFIC NOTEBOOK #: 309

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