Geochemistry
Research
Lab Notebook

Vol. GC-10

GEOCHEMISTRY RESEARCH
LAB NOTEBOOK
GC-10

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6/8/93 JP
0840 hrs. **Preparation of saturated SiO_2 and Al 0.1 m NaCl + 0.01 m $NaHCO_3$ solution for reverse analcime and clinoptilolite experiments**

Objective - prepare a 0.1 m NaCl + 0.01 m $NaHCO_3$ solution containing 200 ppb Al and 30 ppm SiO_2

Method - Dissolve NaCl + $NaHCO_3$ in solutions containing 200 ppb Al + 30 ppm SiO_2.

Equipment + Supplies:
- 4L PP bottle
- 2L volumetric flasks.
- volumetric pipets
- eppendorf pipets + tips
- Mettler AE24 balance (SN-101237)
- Mettler PM460 balance (SN-20467)
- Orion pH meter + pH electrode
- Certified buffers.
- Constant temp bath or shaker

Reagents -
a) NaCl (lot 914193)
b) $NaHCO_3$ (lot 897186A)
c) Alumin standard (1000ppm) (lot 915747-18)
d) Silica standard (1000ppm). (lot 3131-3)

6/8/93
0900hrs
JP

Procedure:

a) Prepare a 10 ppm Al solution by adding 10 ml of ~~the~~ Al standard to a 1L volumetric flask and making up to mark with H_2O.

b) Into each of two 2L volumetric flasks add 60 ml of SiO_2 standard and 40 ml of ~~the~~ 10 ppm Al solution and make up to mark with H_2O.

c) Place 23.38g NaCl + 3.36g $NaHCO_3$ in a 4L PP bottle. Add the solution prepared in step b and label bottle "Reverse Analcime and Clinoptilolite Standard Solution."

d) Leave bottle open to atmosphere to equilibrate with air by placing kimwipe over mouth. Place bottle on gyratory shaker for continuous agitation. Check pH periodically to determine equilibrium.

Solution Measurements - Standards

Date	Time	pH	Na (ppm)
6/11/93	1350	8.91/22.5°C	
6/23/93	0950	8.89/21.2°C	2440
6/29/93	1045	8.97/22.0°C	

6/8/93
1100hrs. JP

c) Prepare matrix solutions to be used in spectrophotometric analysis of reverse analcime + clinoptilolite samples. Place 5.844 g NaCl + 0.8401 g $NaHCO_3$ in each of 4 1L PP bottles and dissolve in 1L of H_2O.
Label Bottles: Reverse Matrix - A
" -B
" -C
" -D

5) Place bottles on gyratory shaker for continuous agitation. Leave open to atmosphere to equilibrate with air by placing kimwipe over mouth. Check pH periodically to determine equilibrium

Solution Measurements - Matrix

Date	Time	pH	Na (ppm)	Reverse Matrix
6/11/93	1352	8.81/22.5°C		A
6/11/93	1354	8.90/22.5°C		B
6/11/93	1356	8.83/22.5°C		C
6/11/93	13.57	8.81/22.5°C		D
6/23/93	0951	8.99/21.2°C	2540	A
6/23/93	0952	9.01/21.2°C	2570	B
6/23/93	0954	8.99/21.2°C	2570	C
6/23/93	0955	8.99/21.2°C	2550	D

6/8/93 GJP

Recovery of clinoptilolite from samples CDVSE1 and CDVSE2.

a) Recovered remaining solutions from CDVSE1 and CDVSE2 by filtering through a 2 µm filter. Placed solutions in 60 ml PP bottle labelled "CDVSE1 Remain solution" + "CDVSE2 Remain solution."

Before recovery solution data, time, and weight of sample were noted in GC/03-300 for final analysis of the solutions.

b) Solids (clinoptilolite) remain in sample bottles. Were vacuum filtered. Solids were rinsed out of bottle with ultrapure H_2O & vacuum filtered.

c) Solids were allowed to air dry on filter paper then placed in plastic containers labelled CDVSE1 * REC + CDVSE2 * REC.

Weights

	Container	Container + Clinoptilolite	Clinoptilolite
CDVSE1 * REC	13.938 g	14.907 g	.969 g
CDVSE2 * REC	13.781 g	14.762 g	.981 g

6/8/93 gp

Recovery of analcime from samples ASE1, ASE2, and ASE3

a) Recovered remaining solution by filtering thru a 2 [illegible] filter. Placed solution in ~~250~~ 125 ml bottles labeled "ASE1 remaining solution" "ASE2 remaining solution," and "ASE3 remaining solution". Before removing solution date, time, + weights were noted in GC-03/241 for final analysis of solution.

b) Solids (analcime) were rinsed out of bottles with ultrapure H_2O & vacuum filtered.

c) Solids were allowed to air dry on filter paper + placed in plastic containers labelled ASE1*REC, ASE2*REC, and ASE3*REC.

Weights	Container	Container + Analcime	Analcime
ASE1*REC	14.063 g	17.605 g	3.542 g
ASE2*REC	13.718 g	17.158 g	3.44 g
ASE3*REC	13.892 g	17.604 g	3.712 g

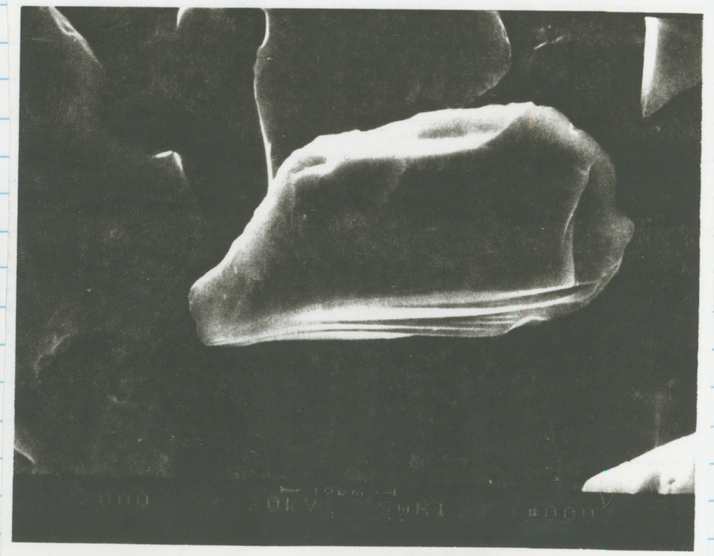
6/9/93 1430hrs. A small amount of ASE1*REC and CDVSE1*REC were mounted on aluminum stubs for examination by scanning electron microscope to observe secondary growth, etch pits, etc...

6/16/93 SEM analyses of ASE1*REC and CDVSE1*REC. Samples were carbon coated and analyzed on the AMRAY in Div 06.

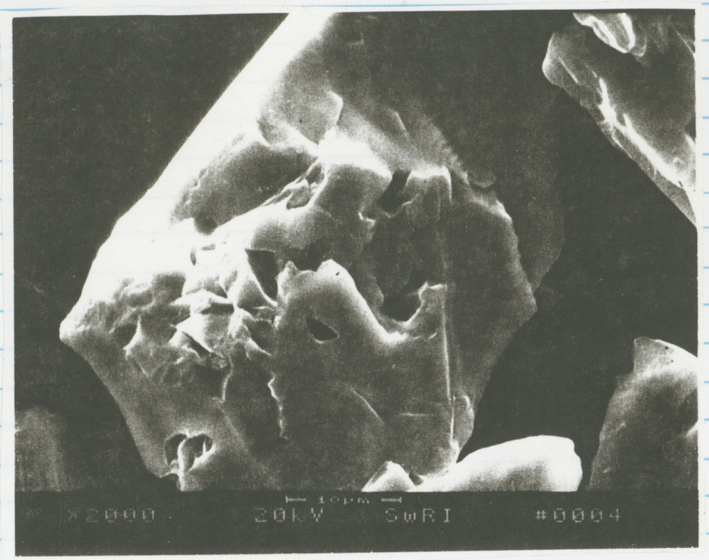
Microphotographs:

#62453
ASE1*REC

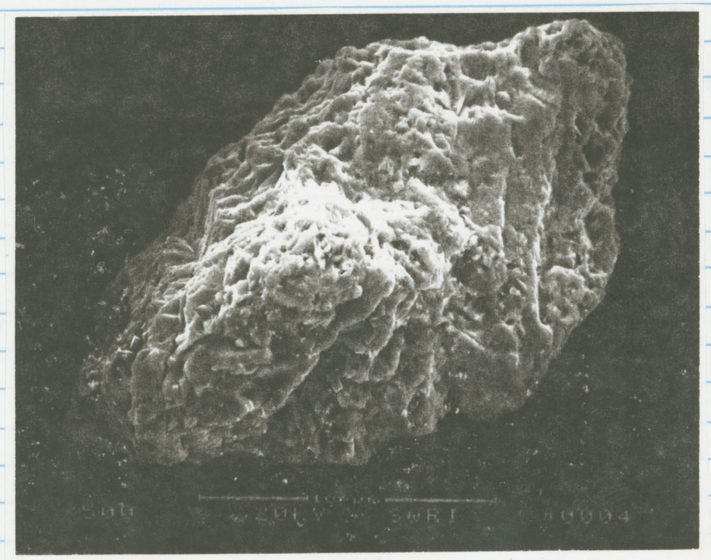
ASE1*REC #62454



ASE1*REC #62455

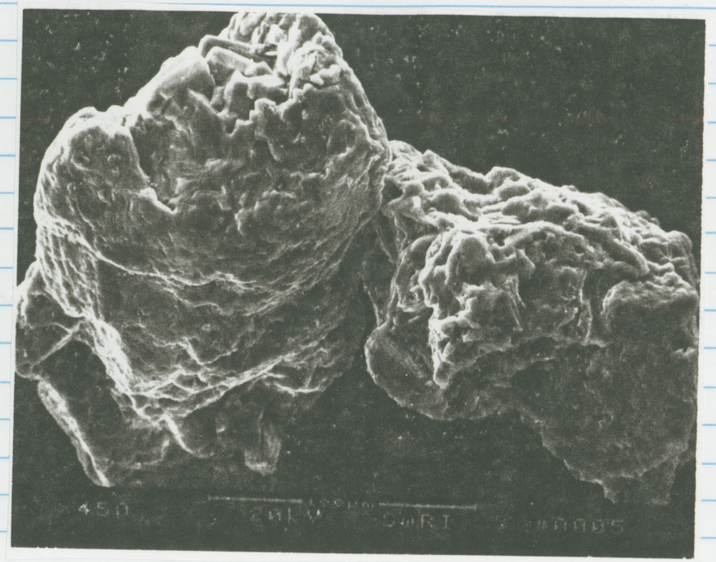


ASE1*REC #62456

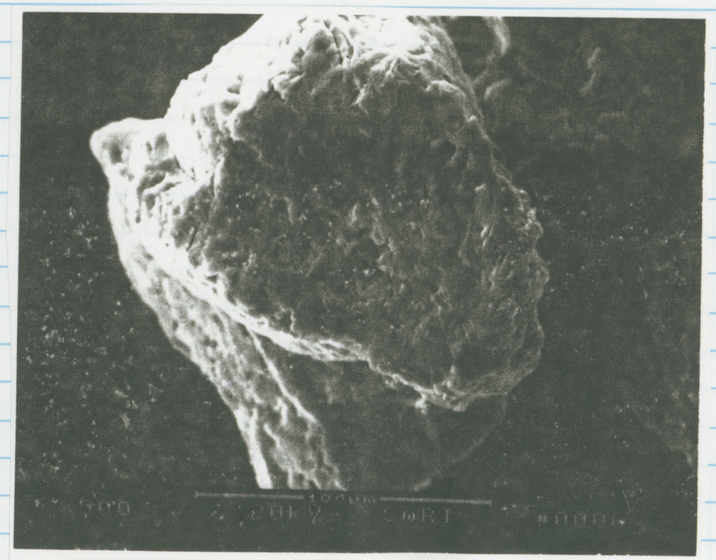


COVSE1*REC #62457

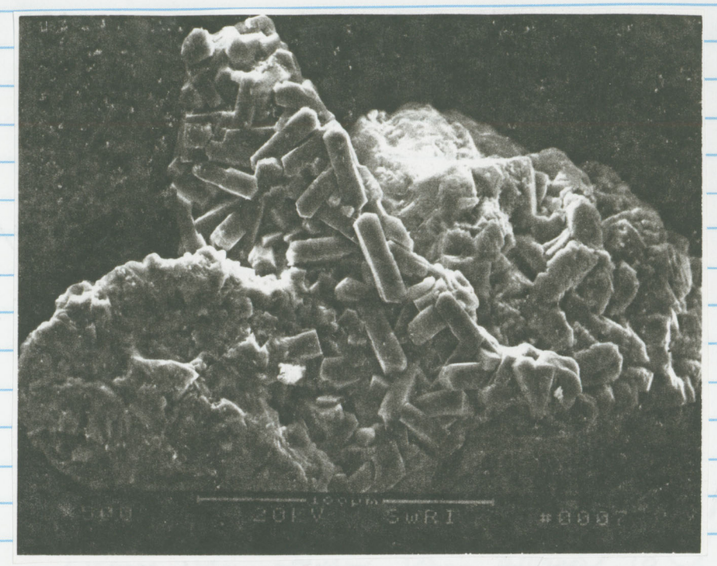
The original photomicrographs are kept in a 3-ring binder entitled "Analcime-Clinoptilolite Geochemistry Experiments".



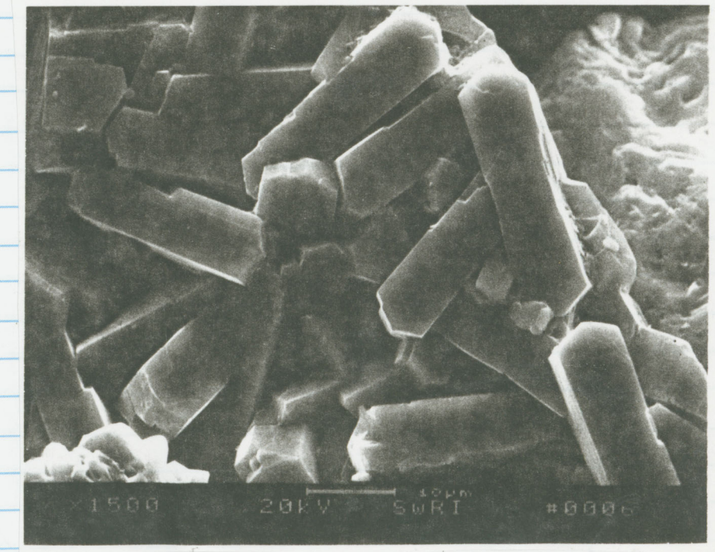
COVSE1*REC #62458



COVSE1*REC #62459



COVSE1*REC #62461



COVSE1*REC #62460

6/21/93 JP Additional 450/635 mesh aNalcime was prepared for reversal experiments.
Bulk ASH (analcime from Mt. St. Hilaire) was crushed using an Al ceramic mortar + pestle and sieved to collect the 450-635 mesh fraction.

The collected 450-635 fraction was placed in a 1L beaker with ~ 500 ml H_2O and ultrasonically cleaned for about 10 min. After ultrasonically cleaning the solute was allowed to stand for ~ 5 min and then the supernatant was poured off. This process was repeated 2 more times.

The solution was then washed by adding approximately 500 ml H_2O to the sample and allowing the suspension to settle for ~ 10 min and then pouring off the supernatant. This process was repeated 4 more times.

The material was then filtered and dried in an oven overnight.
The dried material was then placed in a beaker labeled ASH*450/635*uc*WA.
Weight of beaker = 13.817 g
Weight of ASH*450/635*uc*WA + beaker = 25.195g
Weight of ASH*450/635*uc*WA = 11.378g

6/25/93 gp

Analysis of saturated SiO_2 + Al 0.1 M NaCl - 0.01 M $NaHCO_3$ solutions for reversal experiments.

Objective: measure the SiO_2 and Al content of previously prepared (see GC-10/1) 30 ppm SiO_2 ad 200 ppb Al solutions to be used in reversed experiments.

Equipment + Materials:
- Milton-Roy 1201 Spectrophotometer
- plasticware as needed
- Eppendorf pipets + tips

*Note - sample solutions will not come into contact with glassware due to their high pH (≈9.00) and their corrosive nature.

Procedure:

a) Two 35ml samples were Taken from bottle labelled "Reverse Analcime and Clinoptilolite Standard Solution" and placed in 60 ml PP bottles. The SiO_2 + Al content of the two samples will be determined in accordance with "TOP-013" and "TOP-014". However the procedures will be modified to avoid the use of glassware.

b) As controls 1 35ml sample from bottle "Reverse Matrix-A" and 1 35 ml sample from bottle "Reverse Matrix-C" were placed in 60 ml PP bottles and the SiO_2 + Al contents were determined as in step a.

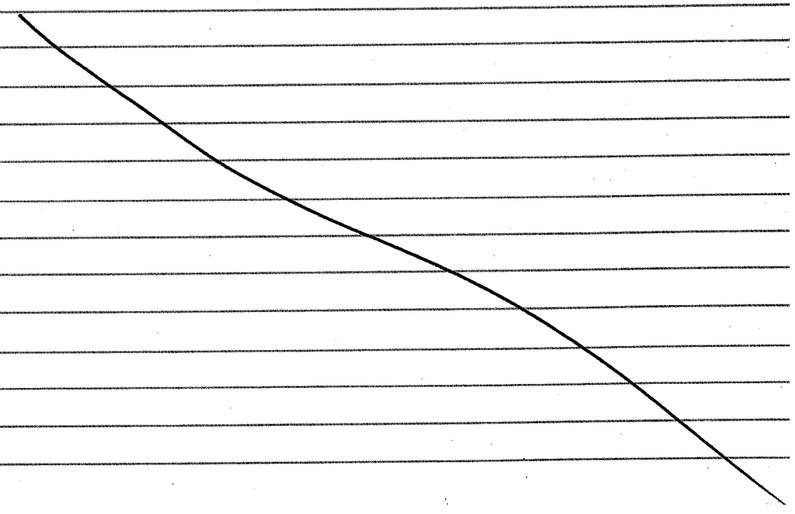
c) Results:

Reverse Analcime-Clinoptilolite Standard Solution

SiO_2 = 29.86 and 29.86 ppm

Al = 195.75 and 201.25 ppb

Spectrophotometer analyses are shown on following pages:



STD # ABS CONC

SiO_2

5.5X dilution factor

1	.100	3.640
2	.151	5.460
3	.199	7.280
4	.251	9.100

LINEAR FIT
NOT THRU ZERO
SLOPE 36.47
INTERCEPT -.0204

CORR COEF 1.0000

ID# 1 TST24
410.0NM 5.54 C — control 5.46

ID# 2 TST24
410.0NM 5.67 C

ID# 3 TST24
410.0NM 5.48 C — Reverse Avalcime-Clinoptilolite Std. Soln

ID# 4 TST24
410.0NM 5.48 C

ID# 5 TST24
410.0NM .03*C — Reverse Matrix A

ID# 6 TST24
410.0NM .10*C — Reverse Matrix C

ID# 7 TST24
410.0NM .05*C — Blank control

STD # ABS CONC

Al - 2.5x dilution factor

1	.124	50.00
2	.199	75.00
3	.266	100.0

LINEAR FIT
NOT THRU ZERO
SLOPE 352.1
INTERCEPT 5.836

CORR COEF .9996

ID# 1 TST24
534.0NM 75.0 C — 75 control

ID# 2 TST24
534.0NM 77.7 C — Rev Aval-clinop

ID# 3 TST24
534.0NM 79.9 C — Std Soln

ID# 4 TST24
534.0NM 6.6*C — Rev Matrix B

ID# 5 TST24
534.0NM 7.2*C — control blank

6/25/93JP Prepared additional reagents for spectrophotometric analysis of SiO_2 and Al as described below:

"Buffer Reagent" for Al analysis:
Dissolved 136g sodium acetate (lot 905630) in ~800 ml H_2O in a 1L volumetric flask. Diluted 5.8 ml glacial acetic acid (lot 893419) to 100 ml H_2O in a 100 ml volumetric flask. Added 40 ml of the acetic acid solution to the sodium acetate solution + diluted to 1L with H_2O. Transferred solution to a 1L PP bottle and labelled "Buffer Reagent".

"Stock Dye Solution" for Al analysis:
Dissolved 300 mg Eriochrome cyanine R (lot K5501 249482 58) in about 100 ml H_2O in a 250 ml glass beaker. Diluted 5 ml glacial acetic acid (lot 893419) to 10 ml in a 10 ml volumetric flask. Adjusted pH of the Eriochrome cyanine R solution to about 2.9 with the acetic acid solution. Transferred solution to a 200 ml volumetric flask + diluted with H_2O to 200 ml. Transferred solution to a 500 ml PP bottle and labelled "Stock Dye Solution".

HCl, 1+1 for SiO_2 analysis:
Added 250 ml concentrated HCl (lot 930085) to 250 ml of H_2O in a volumetric flask. Transferred to a 500 ml PP bottle + labelled "HCl, 1+1".

Oxalic Acid Solute for SiO_2 analysis:
Dissolved 15 g oxalic acid (lot 905504) in 200 ml H_2O in a volumetric flask. Transferred solute to a 500 ml PP bottle labelled "Oxalic Acid Solute".

6/29/93 JF

Reverse Clinoptilolite and Analcime Experiment

Objective: determine kinetics of clinoptilolite and analcime precipitation

Method: monitor SiO_2 and Al of saturated SiO_2-Al solutions containing clinoptilolite or analcime.

Equipment:
a) Mettler PM4600 balance accurate to 0.1 g (SN 20467)
b) Mettler AE240 balance accurate to 0.001 g (SN 101237)
c) ORION pH meter + pH electrode + Na ion selective electrode.
d) Plastic ware + glass ware as needed.

Materials:
a) saturated 30 ppm SiO_2 - 200 ppb Al 0.1 m NaCl - 0.01 m $NaHCO_3$ solution prepared earlier (GC-10/1-4)
b) Matrix solutions prepared earlier (GC-10/1-4)

c) CDVSE2*REC, CDVSE1*REC
CDV*100/200*UC*WA*HL*CPT*NaF
ASE1*REC, ASE2*REC, ASE3*REC
ASH*450/635*UC*WA

Procedure:

This experiment will consist of 10 500 ml PP bottles initially containing 250 ml of the saturated 30 ppm SiO_2 - 200 ppb Al 0.1 m NaCl - 0.01 m $NaHCO_3$ solution + either analcime or clinoptilolite or NO solid.

The schedule below will be followed for sampling and analysis:

Water Mass	Cumulative time	Analyses
250 ml	0	none
-35 ml = 215 ml	2 days	Al, Si, Na
-35 ml = 180 ml	3 weeks	Al, Si
-35 ml = 145 ml	6 weeks	Al, Si, Na
-35 ml = 110 ml	9 weeks	Al, Si
-35 ml = 75 ml	12 weeks	Al, Si, Na

Bottles will be labelled as follows:

RCDV1 + RCDV2 - will contain clinoptilolite recovered from solubility experiments CDVSE1 + CDVSE2.

RCDV3 + RCDV4 - will contain clinoptilolite from original stock CDV*100/200* UC*WA*HL*CPT*NaF

RASH1 + RASH2 - will contain analcime recovered from solubility experiments ASE1, ASE2, + ASE3.

RASH3 + RASH4 - will contain analcime from original stock ASH*450/635*UC*WA.

RNS1 + RNS2 - controls having NO solids.

6/29/93
1330 hrs a) Label and record weights of sample bottles:

RCDV1 - 51.3251 g
RCDV2 - 51.3830 g
RCDV3 - 51.6579 g
RCDV4 - 51.4678 g

RASH1 - 50.9608 g
RASH2 - 50.9660 g
RASH3 - 51.4224 g
RASH4 - 51.3920 g

RNS1 - 51.1834 g
RNS2 - 51.2170 g

b) Place ~.95 g of clinoptilolite into bottle, RCDV1, RCDV2, RCDV3, and RCDV4.

1335 hrs Wt RCDV1 = 51.3251 g
+CDVSE1*REC = 52.2858 g
Wt clinoptilolite = 0.9607 g

1337 hrs Wt RCDV2 = 51.3830 g
+CDVSE2*REC = 52.3581 g
Wt clinoptilolite = 0.9751 g

1340 hrs Wt RCDV3 = 51.6579 g
+ CDV*100/200*UC*WA*HL*CPT*NaS = 52.6190 g
Wt clinoptilolite = 0.9611 g

1343 hrs Wt RCDV4 = 51.4678 g
+ CDV*100/200*UC*WA*HL*CPT*NaS = 52.4284 g
Wt clinoptilolite = 0.9606 g

c) Place ~4.0 g of analcime into bottle RASH1, RASH2, RASH3, and RASH4.

1345 hrs Wt RASH1 = 50.9608 g
+ ASE1*REC+ASE3*REC = 54.9625 g
Wt analcime = 4.0017 g

1347 hrs Wt RASH2 = 50.9660 g
+ ASE2*REC + ASE3*REC = 54.9711 g
Wt analcime = 4.0051 g

1350 hrs Wt RASH3 = 51.4224 g
+ ASH*450/635*UC*WA = 55.4268 g
Wt analcime = 4.0044 g

1353 hrs Wt RASH4 = 51.3920 g
+ ASH*450/635*UC*WA = 55.4009 g
Wt analcime = 4.0089 g

6/29/93

d) Tare ~250 ml of ~~the~~ 30ppm SiO_2 - 200 ppb Al 0.1 M NaCl + 0.01 M $NaHCO_3$ solution into each of ~~the~~ 10 sample bottles.

e) Record total weights of samples.

1357 hrs RCDV1 = 302.33 g
1402 hrs RCDV2 = 302.35 g
1405 hrs RCDV3 = 302.61 g
1408 hrs RCDV4 = 302.42 g

1410 hrs RASH1 = 304.95 g
1413 hrs RASH2 = 304.98 g
1415 hrs RASH3 = 305.42 g
1417 hrs RASH4 = 305.41 g

1420 hrs RNS1 = 301.30 g
1422 hrs RNS2 = 301.24 g

f) Place kimwipes over mouths of bottles + place in shaker bath set a 25°C + 40 rpm.

g) Sample and analyze according to scheduled in pgs. SiO_2 & Al should be measured in accordance with TOP014 + TOP013 but procedure should be modified to avoid use of glassware. Na will be measured with ion selective electrode.

Results

RCDV1

Initial pH = 8.97
Start = 1357 hrs, 6/29/93
Initial wt = 302.33 g

Wt bef.	Date/ time	Wt aft.	Elapsed Time	pH	Na ppm	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al(ppb) raw	Al(ppb) corr
302.21	7/1/93 1425	266.17	48.46		2750	5.49	5.5x 30.195	34.7	2.5x 86.75
264.97	7/21/93 1015	229.39	524.29		8/11/93 2550	5.47	5.5x 30.085	5.9	2x 11.8
227.85	8/11/93 0925	192.38	1837.46 1028.12	8/11/93 9	2710	5.26	6.11x 32.138	4.7	2x 9.4
190.45	9/1/93 1333	154.66	1535.59			5.67 5.44	5.5x 31.185 9.11/93 9 2992	6.0	2x 12.0
152.60	9/28/93 0850	117.00	2178.87		2560	5.41	6.11x 33.055	7.1	2x 14.2
108.39	12/27/93 1020	72.48	4340.37		3100	6.43	6.11x 39.287	4.2	2.0x 8.4
67.37	2/14/94 1500			9.16/20.6°C					

RCDV2

Initial pH = 8.97
Start = 1402 hrs, 6/29/93
Initial wt = 302.35 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al(ppb) raw	Al(ppb) corr
302.22	7/1/93 1435	266.51	48.55		2650	5.45	29.975	39.9	99.75
265.26	7/21/93 1025	229.76	524.38		8/11/93 2560	5.43	29.865	7.5	15.0
228.05	8/11/93 0930	192.62	1027.46		2680	5.16	31.528	6.4	12.8
190.78	9/1/93 1339	155.29	1535.61			5.66	31.13	6.5	13.0
153.40	9/28/93 0904	117.84	2179.03		2520	5.35	32.69	7.8	15.6
109.51	12/27/93 1030	72.94	4346.43		3140	6.32	38.665	4.4	8.8
67.27	2/14/94 1500			9.18/20.6°					

RCDV3

Initial pH = 8.97
Start = 1405 hrs 6/29/93
Initial wt = 302.61 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
302.47	7/1/93 1445	266.75	48.72		2620	5.33	29.315	32.6	81.5
265.92	7/21/93 1032	230.20	524.5		8/11/93 2540 ~~2690~~	5.34	29.370	4.1	8.2
229.18	8/11/93 0940	193.56	1027.63			5.11	31.22	5.8	11.6
192.25	9/1/93 1346	156.55	1535.73			5.60	30,80	6.4	12.8
155.30	9/28/93 0910	119.56	2179.13		2520	5.35	32.69	8.3	16.6
109.66	2/14/94 1455			9.08/20.6°C					

RCDV4

Initial pH = 8.97
Start = 1408 hrs, 6/25/93
Initial wt = 302.42 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
302.31	7/1/93 1450	266.67	48.7		2630	5.36	29.480	30.6	76.5
265.77	7/21/93 1042	230.08	524.5		8/11/93 2540 ~~2680~~	5.31	29.205	4.4	8.8
229.07	8/11/93 0947	193.40	1027.58			5.14	31.405	4.7	9.4
192.10	9/1/93 1355	156.58	1535.71			5.61	30.855	7.1	14.2
155.13	9/28/93 0916	119.47	2179.06		2520	5.46	33.36	~~7.4~~	14.8
110.41	2/14/94 1500			9.09/20.8°C					

RASH1

Initial pH = 8.97
Start = 1410 hrs, 6/29/93
Initial wt = 304.95 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na ppm	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al ppb raw	Al ppb corr
304.81	7/1/93 1455	269.23	48.75		2620	5.16	28.380	50.0	125.0
268.23	7/21/93 1046	232.77	524.63		8/11/93 ~~2560~~ 2700	5.22	28.710	18.5	37.0
231.64	8/11/93 0955	195.96	1027.75			4.75	29.022	10.0	20.0
194.55	9/1/93 1407	159.09	1535.95			5.25 ~~5.44~~	28.875	15.0	30.0
157.47	9/28/93 0925	121.98	2179.13		2520		29.022	19.7	39.4
114.40	12/27/93 1040	78.36	4340.38		29.60	5.14	31.405	7.7	15.4
73.66	2/14/94 1500			9.14/20.6°C					

RASH2

Initial pH = 8.97
Start = 1413 hrs, 6/29/93
Initial wt = 304.98 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na ppm	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
304.87	7/1/93 1503	269.38	48.83		2640	5.20	28.60	47.2	118.0
268.44	7/21/93 1055	232.83	524.7		8/11/93 ~~2540~~ ~~2740~~	5.14	28.27	17.1	34.2
231.68	8/11/93 1000	196.03	1027.78			4.68	28.595	13.6	27.2
194.58	9/1/93 1414	158.96	1536.01			5.12	28.16	13.9	27.8
157.60	9/28/93 0930	121.88	2179.28		2560	4.73	28.90	16.0	32.0
114.08	12/27/93 1050	80.53	4340.41		2930	5.08	31.039	9.0	18.0
76.71	2/14/94 1505			9.11/20.6°C					

RAS#3

Initial pH = 8.97
Start = 1415 hr, 6/29/93
Initial wt = 305.42 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
305.31	7/1/93 1512	269.84	48.95		2630	5.26	28.93	35.2	88.0
268.84	7/21/93 1005	233.18	524.83		8/11/93 2560 ~~2680~~	5.12	28.16	11.2	22.4
231.95	8/11/93 1005	196.42	1027.83	gp		4.72	28.84	10.9	21.8
194.95	9/1/93 1420	159.51	1536.08			5.19	28.545	11.0	22.0
157.85	9/28/93 0935	122.46	2179.33		2520	4.84	29.57	11.7	23.4
112.84	2/14/94 1459			9.09/20.5C					

RAS#4

Initial pH = 8.97
Start = 1417 hr 6/29/93
Initial wt = 305.41 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
305.30	7/1/93 1517	270.96	49.0		2610	5.25	28.875	28.1	70.25
268.81	7/21/93 1116	233.35	524.98		8/11/93 2560 ~~2680~~	5.14	28.270	10.1	20.2
232.06	8/11/93 1015	196.74	1028.0	gp		4.73	28.90	9.5	19.0
195.22	9/1/93 1425	159.77	1536.17			5.06	27.83	10.1	20.2
158.06	9/28/93 0940	122.44	2179.42		2540	4.75	29.02	12.5	25.0
116.78	2/14/94 1457			9.09/20.5C					

RNS1

Initial pH = 8.97
Start = 1420 hr 6/29/93
Initial wt = 301.30 g

Wt bef	Date/ Time	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
301.18	7/1/93 1523	265.63	49.05		2620	5.51	30.305	71.9	179.75
264.61	7/21/93 1123	229.61	525.05		8/11/93 2540 ~~2660~~	5.29	29.095	80.1 62.5	160.20 125.0 gp
228.45	8/11/93 1023	193.54	1028.05	gp		4.99	30.489	~~69.1~~	~~138.20~~ 8/11/93
192.27	9/1/93 1430	157.06	1536.17			5.39	29.645	61.8	123.6
155.68	9/28/93 0945	120.38	2179.42		2540	5.10	31.161	56.2	112.4
115.42	12/27/93 1055	80.26	4340.5		2820	5.21	31.833	37.0	74.0
77.62	2/14/94 1450			9.10/20.5C					

RNS2

Initial pH = 8.97
Start = 1422 hr 6/29/93
Initial wt = 301.24 g

Wt bef	Time/ Date	Wt aft	Elapsed Time	pH	Na (ppm)	SiO_2 (ppm) raw	SiO_2 (ppm) corr	Al (ppb) raw	Al (ppb) corr
301.14	7/1/93 1527	268.65	49.08		2620	5.50	30.25	73.1	182.75
264.83	7/21/93 1126	229.59	525.06		2570 ~~2690~~ 8/11/93	5.34	29.37	80.3 66.2	160.60 132.4 gp
228.46	8/11/93 1030	193.23	1028.13	gp		5.03	30.73	~~72.8~~	~~145.60~~ 8/11/93
191.94	9/1/93 1434	156.86	1536.2			5.40	29.70	62.5	125.00
155.73	9/28/93 0950	120.35	2179.47		2560	5.11	31.22	54.7	109.4
113.62	2/14/94 1456			9.08/20.5C					

8/31/93 JP

Prepared "Buffer Reagt" for Al analysis.
Dissolved 136 g sodium acetate (lot 905630) in 800 ml H_2O in a 1L volumetric flask. Diluted 5.8 ml acetic acid (lot 921603) to 100 ml with H_2O. Added 40 ml of the acetic acid solution to the sodium acetate solution and diluted to 1L with H_2O. Transferred solution to a 1L PP bottle & labeled "Buffer Reagt".

10/28/93 KM

Prepared powder samples to be sent to Quantachrome for N_2-gas multipoint B.E.T. surface area analysis.

Label	Sample Name	(230/325) 20 12/14/93
SwKT-A	ASH*200/230*UC*WA	
SwKT-B	CAV*100/200*UC*WA*HL*CPT*NaF	
SwKT-C	8006 (NIST Standard)	
SwKT-D	ASH*200/230*UC*WA	
SwKT-E	COV*100/200*UC*WA*HL*CPT*NaF	
SwKT-F	ASH*325/450*UC*WA	
SwKT-G	ASH*230/325*UC*WA	
SwKT-H	9005 (NIST Standard)	
SwKT-I	B-8005B*pH 3.50/7.00*REC	
SwKT-J	B-8006B*pH 3.5/7.00*REC	

Note: Samples SwKT-A to SwKT-H are the same samples sent to Micromeritics on 6/21/91 for surface area analysis (see GC-03-202 and GC-03-216) and which were sent back to SwRI after completion of the analysis. Some grayish discoloration was visible on some samples, probably caused by the technique used by Micromeritics. All the powder samples received from Micromeritics were checked by KM with the stereo binocular scope. All samples looked okay, except Sample I, which showed extreme dark discoloration, possibly contamination w/ liquid Argon gas or some nitrogen gas. Sample I was therefore not sent to Quantachrome.

Samples SwKT-I and SwKT-J were received by JP from the Wasicom synthesis experiments on α-Al_2O_3. These will be used to check changes in the surf. area due to dissolution during the synthesis expts.

12/10/93 RP
The following are the results of surface area analysis done by Quantachrome:

5 Aerial Way, P.O. Box 9011
Syosset, New York 11791-9011
Phone: 516-935-2240
Fax: 516-935-2194
Telex: 510 221 2239

SOUTHWEST RESEARCH INSTITUTE
Mr. Roberto T. Pabalan
Sr. Research Scientist
6220 Culebra Road
Post Office Drawer 28510
San Antonio, Texas 78228-0510

DATE: 11/24/93
OUR REF.NO. 93-1366
YR.P.O.NO. 92276

Dear Mr. Pabalan:

Enclosed are the results obtained on your samples Note: Data and samples will only be kept for 6 months from the above date.

Thank you for submitting your samples to Quantachrome Corporation.

If there are any questions, please do not hesitate to contact us.

Submitted by:
QUANTACHROME CORPORATION

Date: 11/16/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Micropore Version 2.40

Sample ID..... SRWI-A
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen
Cross-Sec Area.. 16.2 A² Corr Factor.. 6.580E-05 Molec Wgt.. 28.0134
Sample Weight... 5.4465 g P/Po Toler... 2 File Name.. AS3B1102.RAW
Analysis Time... 57.2 min Equil Time... 3 Operator... BEM
Outgas Time..... 24.0 hrs Outgas Temp.. 250 °C Station #.. 1
End of Run..... 11-11-93 02:36pm

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
5.5583e-02	0.0222	2.124E+03
1.0802e-01	0.0251	3.867E+03
1.5804e-01	0.0278	5.404E+03
2.0794e-01	0.0294	7.133E+03

Area = 1.056E-01 m²/g
Slope = 3.266E+04
Y - Intercept = 3.080E+02
Correlation Coefficient = 0.9998
C = 1.071E+02

Date: 11/23/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Micropore Version 2.40

Sample ID..... SWRI-B
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen
Cross-Sec Area.. 16.2 A² Corr Factor.. 6.580E-05 Molec Wgt.. 28.0134
Sample Weight... 2.6983 g P/Po Toler... 2 File Name.. AS3B2203.RAW
Analysis Time... 63.6 min Equil Time... 3 Operator... BEM
Outgas Time..... 48.0 hrs Outgas Temp.. 250 °C Station #.. 1
End of Run..... 11-22-93 16:44pm

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
1.0107e-01	2.6934	3.340E+01
1.5229e-01	2.9133	4.934E+01
1.9837e-01	3.0848	6.418E+01
2.4712e-01	3.2637	8.047E+01

Area = 1.079E+01 m²/g
Slope = 3.222E+02
Y - Intercept = 5.551E-01
Correlation Coefficient = 0.9999
C = 5.814E+02

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-C
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen
Cross-Sec Area.. 16.2 A² Corr Factor.. 6.580E-05 Molec Wgt.. 28.0134
Sample Weight... 4.9764 g P/Po Toler... 2 File Name.. A33B1704.RAW
Analysis Time... 54.8 min Equil Time... 3 Operator... BEM
Outgas Time..... 24.0 hrs Outgas Temp.. 250 °C Station #.. 1
End of Run..... Wed Nov 17 13:12:43

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.05320	0.0410	1.095E+03
0.10790	0.0466	2.075E+03
0.15930	0.0504	3.005E+03
0.20830	0.0534	3.946E+03

Area = 1.888E-01 m²/g
Slope = 1.834E+04
Y - Intercept = 1.057E+02
Correlation Coefficient = 0.9999
C = 1.745E+02

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-D
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	A²	Corr Factor.. 6.580E-05	Molec Wgt..	28.0134
Sample Weight...	4.9324	g	P/Po Toler... 2	File Name..	A53B1704.RAW
Analysis Time...	54.5	min	Equil Time... 3	Operator...	BEM
Outgas Time.....	24.0	hrs	Outgas Temp.. 250 °C	Station #..	1

End of Run..... Wed Nov 17 13:13:15

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.05570	0.0229	2.062E+03
0.10830	0.0251	3.864E+03
0.15850	0.0264	5.707E+03

Area = 9.804E-02 m²/g
Slope = 3.545E+04
Y - Intercept = 6.642E+01
Correlation Coefficient = 0.9998
C = 5.347E+02

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-E
Sample Description.....
Comments..... Southwest Research Instite
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	A²	Corr Factor.. 6.580E-05	Molec Wgt..	28.0134
Sample Weight...	3.0305	g	P/Po Toler... 2	File Name..	AS3B2206.RAW
Analysis Time...	75.3	min	Equil Time... 3	Operator...	BEM
Outgas Time.....	48.0	hrs	Outgas Temp.. 250 °C	Station #..	1

End of Run..... 11-22-93 03:07am

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.10000	2.6213	3.391E+01
0.15210	2.8358	5.061E+01
0.19750	2.9998	6.564E+01
0.25580	3.2034	8.585E+01

Area = 1.044E+01 m²/g
Slope = 3.333E+02
Y - Intercept = 2.195E-01
Correlation Coefficient = 0.9998
C = 1.520E+03

Date: 11/16/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-F
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	A²	Corr Factor.. 6.580E-05	Molec Wgt..	28.0134
Sample Weight...	3.8105	g	P/Po Toler... 2	File Name..	AS3B1504.RAW
Analysis Time...	55.0	min	Equil Time... 3	Operator...	BEM
Outgas Time.....	48.0	hrs	Outgas Temp.. 250 °C	Station #..	1

End of Run..... 11-15-93 13:37pm

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.05670	0.0234	2.059E+03
0.10850	0.0277	3.518E+03
0.15850	0.0299	5.034E+03
0.20800	0.0321	6.543E+03

Area = 1.159E-01 m²/g
Slope = 2.970E+04
Y - Intercept = 3.405E+02
Correlation Coefficient = 0.9998
C = 8.823E+01

Date: 11/16/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-G
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	A²	Corr Factor.. 6.580E-05	Molec Wgt..	28.0134
Sample Weight...	5.0051	g	P/Po Toler... 2	File Name..	AS3B1103.RAW
Analysis Time...	273.2	min	Equil Time... 3	Operator...	BEM
Outgas Time.....	24.0	hrs	Outgas Temp.. 250 °C	Station #..	1

End of Run..... 11-11-93 04:36pm

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.05510	0.0277	1.687E+03
0.10860	0.0322	3.031E+03
0.15790	0.0349	4.301E+03
0.20810	0.0370	5.690E+03

Area = 1.322E-01 m²/g
Slope = 2.612E+04
Y - Intercept = 2.187E+02
Correlation Coefficient = 0.9997
C = 1.204E+02

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-H
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	Å²	Corr Factor..	6.580E-05	Molec Wgt.. 28.0134
Sample Weight...	3.8105	g	P/Po Toler...	2	File Name.. A63B1704.RAW
Analysis Time...	57.5	min	Equil Time...	3	Operator... BEM
Outgas Time.....	24.0	hrs	Outgas Temp..	250 °C	Station #.. 1

End of Run..... Wed Nov 17 13:13:33

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.10530	0.3859	2.440E+02
0.15610	0.4247	3.484E+02
0.20640	0.4579	4.544E+02
0.25650	0.4882	5.654E+02

Area = 1.626E+00 m²/g

Slope = 2.123E+03

Y - Intercept = 1.858E+01

Correlation Coefficient = 0.9999

C = 1.153E+02

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-I
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	Å²	Corr Factor..	6.580E-05	Molec Wgt.. 28.0134
Sample Weight...	1.2818	g	P/Po Toler...	2	File Name.. AS3B2301.RAW
Analysis Time...	55.0	min	Equil Time...	3	Operator... BEM
Outgas Time.....	36.0	hrs	Outgas Temp..	250 °C	Station #.. 1

End of Run..... 11-23-93 01:42pm

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.10720	0.4945	1.943E+02
0.15700	0.5535	2.692E+02
0.20690	0.5956	3.505E+02
0.25610	0.6441	4.276E+02

Area = 2.180E+00 m²/g

Slope = 1.573E+03

Y - Intercept = 2.437E+01

Correlation Coefficient = 0.9999

C = 6.557E+01

Date: 11/24/93 Page 1

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Adsorption System Report
6 ANYGAS Version 2.30

Sample ID..... SWRI-J
Sample Description.....
Comments..... Southwest Research Institute
Gas Type..... Nitrogen

Cross-Sec Area..	16.2	Å²	Corr Factor..	6.580E-05	Molec Wgt.. 28.0134
Sample Weight...	1.2016	g	P/Po Toler...	2	File Name.. AS3B2204.RAW
Analysis Time...	45.5	min	Equil Time...	3	Operator... BEM
Outgas Time.....	36.0	hrs	Outgas Temp..	250 °C	Station #.. 1

End of Run.....

MULTI-POINT BET

P/Po	Volume cc/g STP	1/[W((Po/P)-1)]
0.05660	0.0542	8.857E+02
0.10830	0.0645	1.506E+03
0.15840	0.0710	2.120E+03
0.20840	0.0754	2.793E+03

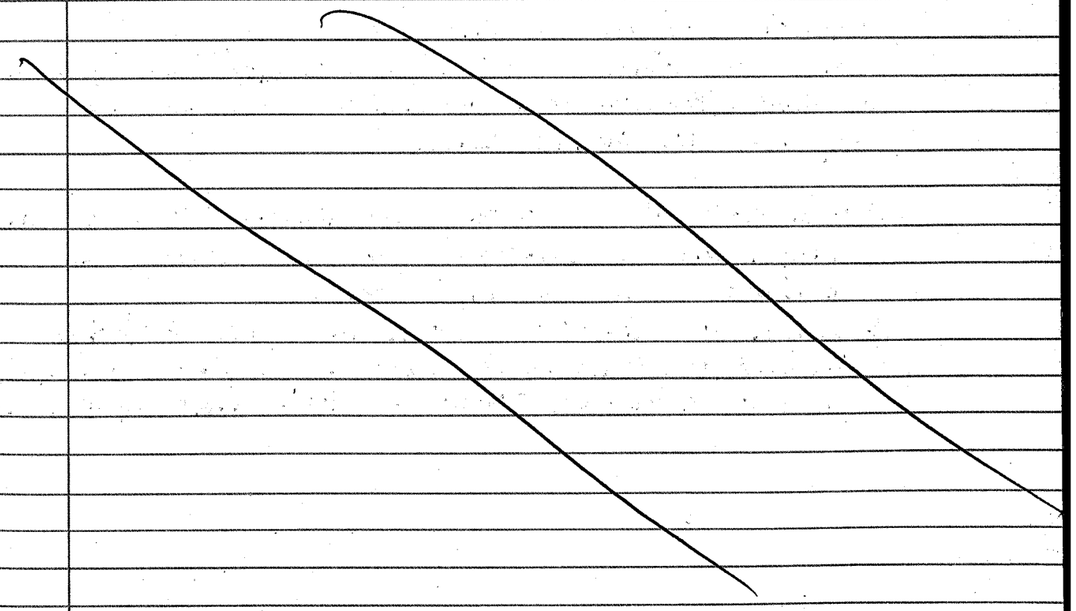
Area = 2.744E-01 m²/g

Slope = 1.253E+04

Y - Intercept = 1.606E+02

Correlation Coefficient = 0.9996

C = 7.902E+01



10 Feb 94
PB

Measurement of structural H_2O in Na-form clinoptilolite.

- Previous measurement of H_2O in Na-form clinoptilolite (CDV) produced values that were in slight disagreement with the theoretically calculated amount of H_2O. One possible reason for the disparity in measured vs. calculated values is that the clinoptilolite structurally exchanges H_2O dependent on degree of saturation. Since the Na-form clinop. used for measurement was stored over saturated NaCl solution (19-21°C) which has an associated % humidity of about 36%, and zeolites are known to exchange H_2O, and because the CDV used for calculations is fully saturated (dissolution expt. pg. 1 of this notebook), the difference in meas. vs. calculated may be the result of differing initial conditions of H_2O content.

- We will attempt to determine H_2O content, starting with zeolite material as close to saturation as possible. Potential problems are that excess water may not be consistently removed and ~~varying results~~ (10 Feb 94) vary the results. And that differential drying of samples during processing may also give varying results.

Two methods of H_2O content determination will be used:
① Samples will be saturated, processed quickly, and measured to provide rapid feedback
② Samples will sit over solutions that give differing % humidities. Measurement should give a plot which can be used to extrapolate water at 100% humidity ⇒ saturation.

10 Feb 94
PB

Procedure:

① 10.03 g of Na-form CDV, previously in atmosphere over sat. NaCl., is mixed with an analcime-clinoptilolite stock solution of 0.1 M NaCl + 0.01M $NaHCO_3$. The mixture is placed in a constant temp shaker bath at 25°C and will be allowed to equilibrate for at least 72 hours.

② About 6 g of Na-form clinop. is transferred to a plastic weigh boat and placed in a ~~dess~~ (10 Feb 94) desiccator over ultra-pure H_2O. Stock Na-form clinop. is stored over fresh saturated NaCl solution (excess NaCl in contact).

16 Feb 94
PB

③ ~ 8 grams of the clinop. equilibrated in NaCl/$NaHCO_3$ solutions is removed by pouring / pipetting into a vacuum filtration apparatus. The vacuum apparatus is a pyrex assembly with a glass frit and a ~~millipore~~ (13) Gelman 0.45 μm filter over a side arm Erlenmeyer flask. Vacuum is applied to the sidearm to draw as much fluid as possible away from the clinoptilolite, which remains on the filter.

④ The clinoptilolite is transferred to a plastic, clean, weigh boat. Not all fluid is removed. The zeolite has the consistency of slightly wet sand. The clinop. is mixed and divided to minimize clumping.

⑤ A portion of the clinop. is transferred to a dry kimwipe (small, folded in half). A second aliquot of clinop. is transferred to a second kimwipe. Weight of kimwipe + clinop is about 2.54 g / 2.55 g respectively. Kimwipe shows that moisture is wicked into the cloth and becomes damp. Kimwipes are carefully folded to prevent loss of zeolite and placed into polycarbonate

16 Feb 94
PD

centrifuge tubes. The tubes have one small kimwipe stuffed into the bottom before the zeolite containing chemwipe is placed into tube. Tubes are capped and placed into centrifuge at ~2800 rpm for 15 min. Combination of 2 tubes / density caused shutdown of centrifuge at speeds much more than 3000 rpm.

⑥ six, pre-cleaned, and dried self sealing quartz crucibles are weighed, zeolite from centrifuge and vacuum filtration only will be added.

Sample #	crucible + clinoptilolite wt (g)	crucible wt (g)	clinop wt (g)
1 V	12.5433	11.5347	1.0086
2 V	12.7338	11.7315	1.0023
3 V	13.7701	12.7668	1.0033
4 V+C	12.0381	11.1012	0.9369
5 V+C	13.6490	12.6609	0.9881
6 V+C	13.4468	12.~~5060~~ 5061 PD 16 Feb 94	0.9407

V = vacuum filtration
V+C = vacuum + centrifuge

Kimwipes from centrifuge samples (combined before addition to crucibles) were noticeably damp. The clinop. dried considerably during weighing. It looked more like that stored over NaCl by the final crucible. In fact, it was dry enough for static to cause some problems.

- zeolite not used for crucibles was placed back into NaCl/$NaHCO_3$ solution.

16 Feb 94
PD

⑦ quartz crucible were placed into preheated convection over at 102 ± 2°C for 2 hours (1430)

⑧ quartz crucibles were removed from over and allowed to cool over Drierite desiccant in vacuum desiccator. removed at 1635, cooled for ~20 min.

⑨ crucibles were reweighed

Sample #	wt after 102°C
1	12.~~8862~~ 2862
2	12.4739
3	13.5249
4	11.9546
5	13.5684
6	13.3723

⑩ crucibles stored overnight in desiccator over drierite (97% $CaSO_4$ + 3% $CoCl_2$)

17 Feb 94
PD

⑪ Muffle furnace preheated to 900°C

⑫ Samples placed into Muffle furnace ~11:45 allow 30 min for reheat to 900

Samples removed from furnace ~ 15:00
⇒ 2.75 at 900°C ± 15°C

After ~~15~~ 10 min cooling outside of furnace, crucibles were transferred to desiccator over drierite. Crucibles still too hot to touch. Crucibles allowed to cool to room temp. 17 Feb 94

17 Feb 94
PD (13) weight of cruciblec + CDV after cooling (1635)

Sample #		wt cruc + CDV (g)
1	6	13.2786
2	5	13.4671
3	4	11.8626
PDY 3		13.4321
5	2	12.3922
6	1	12.2003

17 Feb 94

2/17/94 JP RACDT1 and RACDTIIIA Termination

Reverse experiments RACDTIA and RACDTIIIA were discontinued. Solids (oxalcine) for each sample were filtered and air dried. liquid was filtered into 500 ml plastic bottles labeled as follows:

RACDTIA Solution
RACDTIIIA Solution

Solutions were saved for possible later analysis.

2/18/94 JP Air dried oxalcine powders from experiments RACDTIA and RACDTIIIA were placed in sample vials and labeled as follows:

RACDTIA*REC
RACDTIIIA*REC

2/18/94 gp

Small aliquots (~.1g) of analcime powders used in analcime dissolution of reverse analcime experiments were placed in glass vials to be Taken to Div 06 For auger analysis. (Al/Si ratio)

Vials were labeled as follows and contain either reacted or unreacted analcime as described.

Vial #	Description	
1	ASH * 200/230 * UC * WA	unreacted
2	ASH * 230/325 * UC * WA	unreacted
3	ASH * 325/450 * UC * WA	unreacted
4	ACDT11A	reacted
5	RACDT1A	reacted
6	RACDT111A	reacted

Samples were taken to Jim Spencer in Div 06 for mounting and analysis.

2/21/94 gp

Measure amount of H_2O in RACDT1A + RACDT111A analcime

Objective: determine amount of H_2O in analcime from RACDT1A + RACDT111A experiments.

Method: gravimetric analysis

Equipment: 1) 6 porcelain crucibles
2) Mettler AE240 balance
3) Fisher muffle furnace
4) Drying oven

Procedure:

A) Bring drying oven to 105°C

B) Weigh 6 porcelain crucibles and record weights to place ~ 0.5g RACDT1A or RACDT111A in each crucible + record weights

Weight of qtz crucible #1	= 12.7121 g
+ RACDT1A	= 13.2117 g
Wt of RACDT1A	= 0.4996 g

Weight of qtz crucible #2	= 12.1999 g
+ RACDT1A	= 12.7002 g
Wt of RACDT1A	= 0.5003 g

Weight of qty crucible #3 = 11.6721 g
+RACDT1A = 12.1726 g
Wt of RACDT1A = 0.5005 g

Wt of qty crucible #4 = 12.1112 g
+RACDT11A = 12.6120 g
Wt of RACDT111A = 0.5008 g

Wt of qty crucible #5 = 12.5095 g
+RACDT111A = 13.0089 g
Wt of RACDT111A = 0.4994 g

Wt of qty crucible #6 = 11.0971 g
+RACDT111A = 11.5981 g
Wt of RACDT111A = 0.5010 g

2/21/94
0858 gp C) Place crubles in oven at 105°C for ~2 hrs.

1105 gp D) Remove crubles from drying oven; allow to cool and reweight

Wt of qty crucible #1 = gp 2/21/94 13.2115 g
Previous wt = 13.2115 13.2117 g
Wt absorbed H_2O = .0002 g

Wt of qty crucible #2 = gp 2/21/94 13.7001 g
Previous wt = 13.7001 13.7002 g
Wt absorbed H_2O = .0001 g

Wt of qty crucible #3 = gp 2/21/94 12.1729 g
Previous wt = 12.1729 12.1726 g
Wt absorbed H_2O = .0000 g

Wt of qty crucible #4 = gp 2/21/94 12.6120 g
Previous wt = 12.6120 12.6120 g
Wt absorbed H_2O = .0000 g

Wt of qty crucible #5 = gp 2/21/94 13.0090 g
Previous wt = 13.0090 13.0089 g
Wt absorbed H_2O = .0000 g

Wt of qty crucible #6 = gp 2/21/94 11.5985 g
Previous wt = 11.5985 11.5981 g
Wt absorbed H_2O = .0000 g

1135 gp E) Place crubles in muffle furnace at ~900°C for ~2 hrs.

1630 hrs gp F) Remove crubles, allow to cool, + reweigh.

Wt of qty crucible #1 = 13.1700 g
Previous wt = 13.2115 g
Wt structural H_2O = .0407 g

Wt of qty crucible #2 = gp 2/21/94 12.6589 g
Previous wt = 13.7001 12.7001 g
Wt structural H_2O = .0412 g

Wt of dry crucible #3 = 12.1314 g
Predried Wt = 12.1729 g
Wt structural H_2O = .0415 g

Wt of dry crucible #4 = 12.5706 g
Predried Wt = 12.6120 g
Wt structural H_2O = .0414 g

Wt of dry crucible #5 = 12.9676 g
Predried Wt = 13.0090 g
Wt structural H_2O = .0414 g

Wt of dry crucible #6 = 11.5570 g
Predried Wt = 11.5985 g
Wt structural H_2O = .0415 g

2/22/94 JP

Solids RACDTIA* and RACDTIIIA* were removed from quartz crucibles and placed in glass vials labeled RACDTIA* (heated) and RACDTIIIA* (heated).

6/7/94 RG

1. Removal of Free iron oxide using Nedron silica sand USING CDB (citrate-dithionate-bicarbonate)
2. Digestion of iron in solution
3. Etching of quartz surface using HNO_3 and HCl

Parts 1 + 2:

I. 1st sand used: 60/100 * UC * RC -- 4.9889 grams (used Mettler AE 240)
amount of sodium dithionite added to sand 1: 1.0059 (Mettler AE 240)

II. blank - NO sand used
amount of sodium dithionite added to blank: 1.0015 (Mettler AE 240)

6/8/94 RG

III. 2nd sand used: W510 * 100/200 * UC * RC -- 4.9998g (Mettler AE 240)
amount of $Na_2S_2O_4$ added to sand 2: 1.0012g (Mettler AE 240)

IV. 3rd sand used: W510 * 60/100 * UC * RC * RFe -- 4.9991g (Mettler AE 240
amount of $Na_2S_2O_4$ added to sand 3: ~~4.9991g~~ 0.9999g (Mettler AE 240)

V. 4th sand used: W510 * 100/200 * UC * RC * RFe -- 4.9969g (Mettler AE 240)
amount of $Na_2S_2O_4$ added to sand 4: 1.0046g (Mettler AE 240)

Part 3

I. 1st sand used: 60/100 * UC * RC -- 1.00g (OHAUS)
w/ HCl 6M

II. 2nd sand used: W150 * 60/100 * UC * RC -- 1.00g (OHAUS)
w/HCl 6M

III. 3rd sand used: W150 * 60/100 * UC * RC * RFe -- 1.00g (OHAUS)
w/HCl 6M

IV. 4th sand used: W150 * 100/200 * UC * RC * RFe -- 1.00g (OHAUS)
w/HCl 6M

I. sand 1 (60/100 * UC * RC) used w/ 6m HNO_3 -- 1.0000 (Mettler AE 240)
II. sand 2 (100/200 * W150 * UC * RC) used w/ 6m HNO_3 -- 1.0034 (Mettler AE 240
III. sand 3 (W150 * 60/100 * UC * RC * RFe) used w/ 6m HNO_3 -- 1.0007 (Mettler AE 240)
IV. sand 4 (W150 * 100/200 * UC * RC * RFe) used w/ 6m HNO_3 -- 1.0035 (Mettler AE 240)

R.g 6/9/94

6/9/94 RG. Procedure ~~FRG~~ RG 6/9/94 for Flame-AAS analysis of Fe from surface of Nedron silica

When making the Fe calibrations I used the examples given for 2ppm Fe, 4ppm Fe, and 5ppm Fe. For 0.5ppm Fe and 1.0ppm Fe I used:

0.5ppm Fe: RG 6/9/94
I used 2.5ml of 1000ppm 100ppm diluted to 50ml

1.0ppm Fe: RG 6/9/94 RG 6/9/94
I used 5ml of 1000ppm 1000ppm 100ppm diluted to 50ml

*I have to refilter the 4 various sands and the blank in the removal of free iron oxides from Nedron silica sand using CDB and digestion of iron in solution due to the murkiness of the solutions. I'm not adding anything to them and I'm placing them in PP bottles

*Due to the fact that I did not properly allow the 8 solutions in the "etching of quartz surface using HNO_3 and HCl" to evaporate to near dryness, I am moving them to PP bottles and starting from scratch. They will later be compared to the properly done solutions'

*In the "procedure for Flame-AAS analysis of Fe from surface of Nedron Silica" I used the wrong glassware (beakers instead of volumetric flasks) when making the calibration standards (so they had to be disposed of).

Today I transfered the 8 NON-evaporated solutions to PP bottles. In addition from the murky solutions I filtered the blank and the 4 solutions from the sand into PP bottles

RG 6/10/94

6/10/94 RG Today I repeated "the etching of quartz surface using HNO_3 and HCl"

When recording data or noting the various sands, they will be represented as follows:

Sand 1: $\frac{60}{100}$ * UC * RC
Sand 2: W150 * $\frac{100}{200}$ * UC * RC
Sand 3: W150 * $\frac{60}{100}$ * UC * RC * RFe
Sand 4: W150 * $\frac{100}{200}$ * UC * RC * RFe

In the 1st part of the procedure (where HCl is added to the sand) I used the following amount of each sand using the Mettler AE 240:

Sand 1A: 1.0020 grams
Sand 2A: 1.0024 grams
Sand 3A: 0.9960 grams
Sand 4A: 0.9912 grams

*the A denoting that 6m HCl is being used

When adding the 30ml of HCl to the sand I used a volumetric flask.

I then placed the 4 samples in the 79°C hot water bath five minutes apart (for 30 minutes each)

I then filtered the 4 solutions (using Fisher brand 78 filter paper). I then set them on the hot plate (btwn the 2 and 3 setting) and let them evaporate to near dryness (using the thermodyne NUOVA II)

While 1A-4A were evaporating I went on to the second part of the procedure (where HNO_3 is added to the sand). I used the following amount of each sand using the Mettler AE240:

Sand 1B: 1.0021 grams
Sand 2B: 1.0029 grams
Sand 3B: 1.0014 grams
Sand 4B: 0.9969 grams

*the B denoting that 6m HNO_3 is being used

When adding the 30ml of 6m HNO_3 I used a volumetric flask

RG 6/10/94

6/10/94 RG
- I moved the solutions 1A-4A to the Corning because it heats faster there.
- I then placed solutions 1B-4B in the Fisher water bath at 79°C at 5 minutes apart (for 30 minutes apiece).
- I then filtered ~~1A-4A~~ RG 6/10/94 1B-4B using Fisherbrand P8 filterpaper
- When 1A-4A were near dryness I washed them with 5mL of 1% HNO_3 and then let that ~~those~~ evaporate. I then added 50ml and diluted the various solutions to 100ml (using the 1% HNO_3 for both the 50ml and dilution)
- I then placed solutions 1B-4B on the corning hot plate to evaporate to near dryness
- When 1B-4B were near dryness I washed them with 5mL of 1% HNO_3 and then let that evaporate. I then added ~50mL of 1% HNO_3 to each solution. I then diluted each to a 100mL using a volumetric flask for 100mL.

*I then repeated making calibration standards for the "procedure for Flame-AAS analysis of Fe from surface of Nedron Silica" I used the following amounts of everything when making the various standards:

0.5 ppm Fe:
1ml of 100ppm Fe diluted to 200ml = 0.5ppm Fe

1.0 ppm Fe:
1ml of 100ppm Fe diluted to 100ml = 1.0ppm Fe

2.0ppm Fe:
5ml of 100ppm Fe diluted to 250ml = 2.0 ppm Fe.

4.0ppm Fe:
10ml of 100ppm Fe diluted to 250ml = 4.0 ppm Fe

5.0ppm Fe:
5ml of 100ppm Fe diluted to 100ml = 5.0ppm

- I used the 1% HNO_3 as the dilutant

6/10/94 RG some of the standards have a bit too much dilute, which will be taken care of in the next few days.

The following copies are the two procedures I'm following. The QC-10,061 just represents this book number

QC-10
061

Procedure for
① removal of free iron-oxides from Nedron silica sand using CDB (citrate-dithionate-bicarbonate)
② digestion of iron in solution
③ etching of quartz surface using HNO_3 and Heat.

Objectives: remove free iron-oxides from Nedron sand. Repeat process on pre-processed sand. Characterize residual surface coatings on RFe silica. Compare data from each process step.

Equipment:
- ~~250~~ 100 ml beakers
- temperature controlled water bath
- filter paper / filter assembly
- stirring rods
- analytical balance (AG 240)
- stir/hot plate w/ teflon stir bars
- volumetric flasks (100 ml)
- Fast filter paper (#4)

Reagents:
- nanopure H_2O (nH_2O)
- 0.3M sodium-citrate-dihydrate
- 0.5M sodium bicarbonate
- sodium dithionite
- WSW = 60/100 g 100/200 # HC # RC # RFe
- 6M HCl
- 6M HNO_3, 50% HNO_3, 1% HNO_3 solution
- WSIO = 60/100 g 100/200 # HC

250 ml
~~50~~ ~~100 ml~~
~~50~~ ~~100 ml~~
250 ml

10% (vol)
100 ml

Overview: Add sand and sodium citrate & sodium bicarbonate to beaker. Heat to 75-80°C. Add sodium dithionite, mix. After 15 min remove and wash through filter. Save supernatant + wash.

Procedure:

① Preparation of reagent solutions

a. 0.3 M Na-citrate · $2H_2O$
- mix 88.24 g Na-citrate · $2H_2O$ per 1 liter nH_2O
- you will need about 500 ml of this solution

b. ~~0.5~~ 1.0 M Na-bicarbonate
- mix ~ 84 g per liter
- you will need about 100 ml of $NaHCO_3$ total (5 ml for each 40 ml Na-citrate used)

② Mix buffer-chelator with sand and heat

a. Add 40 ml Na-citrate (0.3 M) and ~~50 m~~ 5 ml $NaHCO_3$ ~~0.5M~~ 1.0M) to a 100 ml beaker.
b. Add 5 g W510 sand to solution (weigh carefully, to nearest 0.001 g)
c. Heat mixture in water bath to 75-80°C (do not exceed 85°C)
d. Process one beaker w/o sand. To be used as blank in Fe analysis.

③ Add sodium dithionite and mix

a. when solution has reached 75-80°C, add 1g of $Na_2S_2O_4$ to the solution. Stir suspension continuously for 1 min. Then stir occasionally for the next 15 min.

④ Remove and decant solution

a. remove beaker from water bath and allow to settle

b. decant the supernatant (quantitatively) by pouring into filter (use a clean beaker to collect supernatant and washings!). Remember to pass blank solution through filter paper as well. Use nH_2O
c. Wash sand and 100 ml beaker thoroughly. Pass all supernatant and washings through filter. Try to accomplish wash with less than 50 ml of nH_2O.

⑤ Acidify and dilute supernatant/wash

a. add 2ml of HNO_3 (50%) to collected wash + supernatant (if volume is greater than 100 ml, add 4 ml 50% HNO_3) Transfer supernatant to 100 ml vol flask and make up to mark with nH_2O. (if >100 ml., use 200 ml vol flask). Discard sand and filter.

⑤ b. label vol. flask W510 # 60/100 # Fe test, etc.

c. set flasks aside for further processing.

⑥ Processing of sand for surface etching

FOR EACH SAND SAMPLE:

a. Add ~~1g~~ 30g of sand to 100 ml beaker (weigh carefully)
b. Add ~~100~~ 30 ml of 6M HCl
c. Heat beaker to 75-80°C for 30 min (perform inside fume hood!)

a. Add ~~1g~~ 30g of sand to 100 ml beaker
b. Add ~~50~~ 30 ml of 6M HNO_3
c. Heat beaker to 75-80°C for 30 min

⑦ Decant and filter supernatant

a. Filter supernatant into #4 filter paper (use 250 ml beaker to catch wash. Wash sand ~~thourro~~ thoroughly w/ nH_2O. (minimize volume of wash)
b. repeat with all sand suspensions (HCl and HNO_3)

⑧ Evaporate wash + supernatant to near dryness, take up w 1% HNO_3. (use about 90 ml)
⑨ transfer solution to 100 ml vol flask and dilute to mark with 1% HNO_3. Label and set vol. flasks aside.

The Second procedure:

Procedure for Flame-AAS analysis of Fe from surface of Nedron silica

Objective: Use Perkin-Elmer AAS to measure concentration of Fe in supernatant solutions obtained from chemical processing of Nedron silica sand. Relative concentration of Fe should give an indication of the amount of Fe present on surfaces. Fe on quartz surfaces may have an impact on the results obtained in sorption experiments between U and SiO_2.

Overview: Several solutions will be aspirated and analyzed using the PE-AAS. Fe concentration is determined by comparing the absorbance of these solutions to a Fe-based calibration curve.

Method: Standard flame-AAS as outlined in PE manual and "standard methods for analysis of water and wastewater", 17th edition.

Equipment:
- 100 ml vol. flasks containing unknown solutions
- polyethylene sample cups
- Perkin-Elmer 3100 AAS
- Fe specific hollow cathode lamp.
- flasks, beakers for dilution/prep of standards

Reagents:
- Fe AAS standard solution (1000 ppm)
- nanopure H_2O
- 1% HNO_3 or 50% HNO_3 as needed

Procedure:

① Prepare standard solutions for calibration of the AAS.

Fe- AAS procedure ②

① Preparation of standard calibration solutions (cont'd)

a. pour a small amount ~ 20 ml of Fe standard solution (1000 ppm) into a clean beaker.

b. Using a 10 ml volumetric pipet and a 100 ml volumetric flask, make a 100 ppm Fe stock solution. Solution may remain in a labeled volumetric flask for a few days. Dilute the 10 ml of 1000 ppm Fe to 100 ml using nH_2O.

c. From the 100 ppm Fe stock solution, prepare several calibration solutions. Each calibration solution should be 10 ml or greater in volume. An exception is the 5.0 ppm cal. solution, which should be at least 50 ml in volume.

make cal. standards of 0.5, 1.0, 2.0, 4.0, and 5.0 ppm

Example:

10 ml of 100 ppm Fe diluted to 25 ml = 4 ppm Fe

$\frac{(10)(100)}{250} = 4$ $\quad \frac{1(100)}{100} = 1$ $\quad \frac{1(100)}{200} = 0.5$

10 ml of 100 ppm diluted to 50 ml = 2 ppm Fe

$\frac{(5)(100)}{250} = 2$

5 ml of 100 ppm Fe diluted to 100 ml = 5 ppm Fe

$\frac{(5)(100)}{100} = 5$

③

① c. cont'd

each calibration standard should be diluted so that the matrix is 1% HNO_3. This can be accomplished by adding 2 ml of 50% HNO_3 for each 100 ml of solution, or by making all dilutions with pre-prepared 1% HNO_3. Most likely, the approach using 1% HNO_3 as the diluent will be easier.

d. Prepare sample cups for cal. stds, cal blank and unknowns. Label Sample cups.

- calibration blank: add ~ 50 ml of 1% HNO_3 to a 100 ml beaker.
- add ~ 20 ml of 5.0 ppm std to a sample cup.
- add ~ 10 ml of each cal. std to sample cup
- add ~ 10 ml of each unknown to sample cup

② Set up AAS for Fe determination
- optimize w/ 5 ppm Fe std
- use 248.3 nm wavelength and 0.2 slit.
- lean, blue flame
- prepare machine in accordance with operating manual
- use 5 std, linear calibration, manual mode.

③ Analyze unknowns and record data.

PM RG
6/14/94

Today I am doing the Flame-AAS analysis from the surface of Nedron silica. The first thing I did was find the absorbance of the 0.5, 1.0, 2.0, 4.0, and 5.0 ppm Fe using the Perkin Elmer 3000 (with a 7mA lamp current, 2.0 integration and 5 replications). (my blank was 1% HNO_3)

concentration:	0.5	1.0	2.0	4.0	5.0
absorbance	.016	.033	.066	.128	.162
	.017	.034	.069	.129	.163
	.016	.030	.067	.130	.161
	.014	.032	.064	.130	.160
	.014	.033	.066	.133	.164

I have to do it again b/c 5.0 ppm should always be in the .199-.215 range

conc:	0.5	1.0	2.0	4.0	5.0
absorb:	0.016	0.031	0.070	0.140	0.178
	0.016	0.032	0.069	0.145	0.180
	0.014	0.034	0.070	0.144	0.178
	0.015	0.034	0.070	0.146	0.179
	0.015	0.033	0.070	0.143	0.179

The following chart is the absorbance readings from the iron samples and blank taken in the procedure: removal of free iron oxides from Nedron silica using citrate dithionate - bicarbonate

sand:	1	2	3	4	blank
absorbance	0.064	0.160	0.016	0.019	0.012
	0.067	0.158	0.014	0.019	0.014
	0.066	0.158	0.011	0.020	0.011
	0.066	0.156	0.016	0.018	0.016
	0.065	0.156	0.014	0.018	0.009

PM 6/14/94

~~6/13/94~~ 6/14/94 Rg

The following table uses the samples from the quartz etching. The sands 1-4 represent are on page 53 of this notebook
A represents the use of 6M HCl
B represents the use of 6M HNO_3 ~~6/13/94 Rg~~ 6/14/94 Rg
This table is for the original 1A-4A ~~&~~ 1B-4B that I did not evaporate enough:

sand absorbance ↓

1A old	2A old	3A old	4A old	1B old	old 2B	3B old	4B old
0.548				030			
0.549				029			
0.552				029			
0.557				028			
0.552				031			

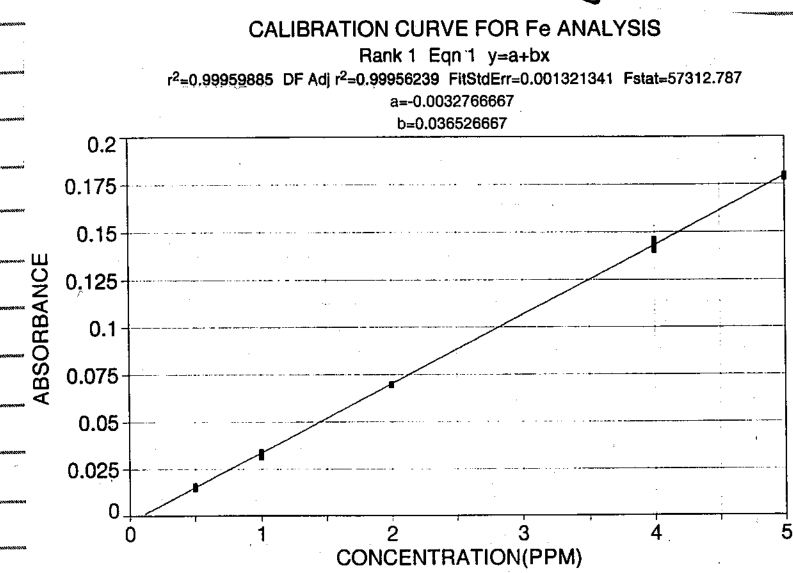
The following table uses the samples from the quartz etching that I did evaporate enough.
The sands 1-4 represent are on page 53 of this notebook
A represents the use of HCl
B represents the use of HNO_3

I'm disregarding this b/c I recorded it before the AAS stabilized

1A	2A	3A	4A	1B	2B	3B	4B
0.053	0.061	0.030	0.026	0.012	(0.021)	0.000	0.009
0.054	0.062	0.033	0.027	0.010	0.028	0.001	0.008
0.055	0.061	0.029	0.025	0.013	0.030	0.003	0.011
0.054	0.061	0.027	0.026	0.011	0.028	0.000	0.008
0.052	0.059	0.030	0.023	0.010	0.026	0.000	0.010
					0.032		

The following were the conditions of the Perkin-Elmer 3000 AAS I used:
Current: 7 mA
Int. time: 2 seconds
reps: 5
wavelength: 248.3 nm
slit: 0.2
Flame: lean, blue, air-acetylene
I used 5 ppm Fe to optimize the flame
I used manual mode.

6/14/94 Rg calibration curve for Fe analysis:



Rank 1 Eqn 1 y=a+bx

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9995988544	0.9995623866	0.0013213410	57312.786835

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.00327667	0.000464040	-7.06117734	-0.00423688	-0.00231645
b	0.036526667	0.000152575	239.4008915	0.036210951	0.036842383

Date	Time	File Source
Jun 14, 1994	10:24:50 AM	c:\excel\wfean.xls

It is apparent from this that the general form of the equation for points on the line is $y = \sout{0.0328}$ −0.00328 (6/14/94) + 0.0365x

(The reason one table is not complete on page 60 is b/c the information from two runs proves that the information is out of range and invaluable.

RG 6/14/94

6/14/94 Rg

Calibration standards	
Conc:	Abs:
0.5	0.016
0.5	0.016
0.5	0.014
0.5	0.015
0.5	0.015
1	0.031
1	0.032
1	0.034
1	0.034
1	0.033
2	0.07
2	0.069
2	0.07
2	0.07
2	0.07
4	0.14
4	0.145
4	0.144
4	0.146
4	0.143
5	0.178
5	0.18
5	0.178
5	0.179
5	0.179

Equation to find concentration(x) or absorbance(y)
Y equals -0.00328 +0.0365X

Absorbance reading from CDB sand:

Sand1	Sand2	Sand3	Sand4	Blank
0.064	0.16	0.016	0.019	0.012
0.067	0.158	0.014	0.019	0.014
0.066	0.158	0.011	0.02	0.011
0.066	0.156	0.016	0.018	0.01
0.065	0.156	0.014	0.018	0.009
Average:	Average:	Average:	Average:	Average:
0.0656	0.1576	0.0142	0.0188	0.0112

Corrected averages(ave of sand-ave blank)

0.0544	0.1464	0.003	0.0076

Concentration of Fe in CDB solution as ppm:

Sand1:	Sand2:	Sand3:	Sand4:	Blank:
1.580274	4.100822	0.172055	0.298082	0.396712

Number of grams used of each sand:

Sand1:	Sand2:	Sand3:	Sand4:
4 .9889 g	4.9998g	4.9991g	4.9969g

Concentration of Fe in 1 gram CDB sand as ppm:

Sand1	Sand2	Sand3	Sand4
31.6758	82.01972	3.441715	5.965342

The absorbance readings of the sand from the "quartz etching"
The sands which 1-4 represent are on page 53 of notebook GC-10
A represents the use of 6M HCl
B represents the use of 6M HNO3

1A	2A	3A	4A	1B	2B	3B	4B
0.053	0.061	0.03	0.026	0.012	0.032	0	0.009
0.054	0.062	0.033	0.027	0.01	0.028	0.001	0.008
0.055	0.061	0.029	0.025	0.013	0.03	0.003	0.011
0.054	0.061	0.027	0.026	0.011	0.028	0	0.008
0.052	0.059	0.03	0.023	0.01	0.026	0	0.01
Average:	Average:	Average:	Average:	Average:	Average:	Average:	Average:
0.0536	0.0608	0.0298	0.0254	0.0112	0.0288	0.0008	0.0092

Concentration of Fe in solution:

1.558356	1.755616	0.906301	0.785753	0.396712	0.878904	0.111781	0.341918

Amount of each sand used in procedure:

1A	2A	3A	4A	1B	2B	3B	4B
1.002	1.0024	0.996	0.9912	1.0021	1.0029	1.0014	0.9969

Concentration of Fe in one gram of each sand:

1A	2A	3A	4A	1B	2B	3B	4B
155.5246	175.1413	90.99411	79.27294	39.5881	87.63627	11.16245	34.2981

The formula I used was(a64*100)/1.002

6/14/94 Rg Explanation of spread sheet data:
① On the left side of page one I listed the results of the calibration standards on the AAS.
② I came up with the equation y= -.00328+.0365x by taking the a (-.00328) + B (.0365) values that the computer program Table Curve gave me when I was using it to find the calibration curve (the curve and the Table Curve's a + b are located on the graph on page 61 of this notebook).
③ I then listed the absorbance reading from CDB sand that the flame AAS gave me.
④ I added the 5 absorbance readings and divided by 5 to find the average.
⑤ I then subtracted the average of the blank from the other averages to compensate for the amount there 6/14/94 Rg matrix absorbed.
⑥ I then found the concentration of Fe in CDB solution as ppm by plugging the corrected averages into the y= -.00328 + 0.0365x formula
⑦ To find the concentration of Fe in 1 gram of CDB sand I took the concentration of Fe in CDB solution multiplied it by its volume/weight (100ml or 100g). This gave me the number of micrograms so I then divided by the amount of sand I had used to come up with the ppm/g

Page 2:
6/15/94 Rg ① The numbers listed are the absorbance reading the AAS gave me (the 5 numbers listed below each sand number).
② I then computed the average absorbance reading by adding the five absorbance numbers and dividing by five
③ I then calculated the concentration of Fe in solution by plugging the average absorbance of each sand solution into the y= -0.00328 + 0.0365x equation.

6/15/94 RG

④ To find the concentration of Fe in one gram of sand, I took the amount of concentration Fe in solution and multiplied it by the weight of the solution (100g). I then divided by the amount of sand used. This is demonstrated by the equation (A64*100)/1.002 where A64 = 1.553356 and the answer to this expression is 155.5246.

Procedure: Find the absorbance/concentration of Fe in a "blank" solution of 6M HCl.

Steps:

① I heated about 30 ml of 6M HCl for 30 minutes in a 80°C water bath.

② I then filtered it with Fisherbrand P8 filter brand, trying to minimize the amount of NH_4O I used to wash.

③ I then evaporated it to near dryness.

④ I then transfered it to a 100ml volumetric flask using a 1% HNO_3 matrix

For calibration standards I used a 2.0 ppm Fe and 5.0 ppm. I made them as follows:

2.0 ppm Fe

5 ml of 100 ppm Fe diluted to 250ml = 2.0 ppm

5.0 ppm Fe

5ml of 100 ppm Fe diluted to 100ml = 5.0 ppm

R.G 6/15/94

6/15/94 RG I then calibrated the AAS using 2.0 ppm Fe and 5.0 ppm Fe and got the following:

2.0	5.0
.071	.199
.071	.199
.072	.198
.074	.195
.072	.199

I then ran the HCl and got the following:

6M HCl
.002
.001
.002
.0
.002

The following were the conditions of the Perkin Elmer 3000 AAS I used:

current: 7 mA

int. time: 2 sec

reps: 5

wavelength: 248.3

slit: 0.2

flame: lean, blue, air-acetylene

~~to~~ 6/15/94 RG

I used 5ppm Fe to ~~optimize~~ 6/15/94 RG optimize the flame manual mode

6/15/94 RG

6/15/94 RG

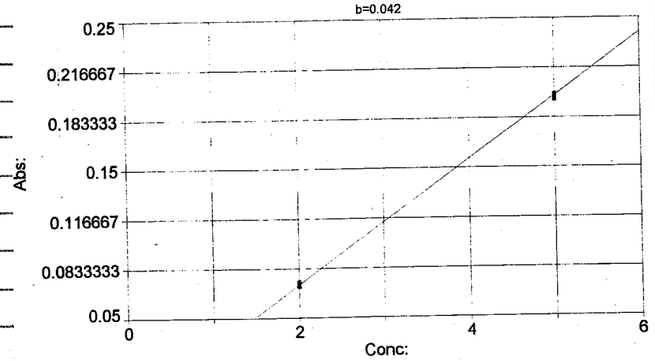
Calibration Curve: Fe Analysis of HCl

Rank 10 Eqn 1 y=a+bx

r^2=0.999546691 DF Adj r^2=0.999417174 FitStdErr=0.0015 Fstat=17640

a=-0.012

b=0.042



Rank 10 Eqn 1 y=a+bx

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9995466908	0.9994171739	0.0015000000	17640.000000

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	-0.01200000	0.001204159	-9.96545758	-0.01478649	-0.00921351
b	0.042000000	0.000316228	132.8156617	0.041268232	0.042731768

Date	Time	File Source
Jun 14, 1994	1:53:15 PM	c:\excel\feanhcl.xls

FEANHCL.XLS

Conc:	Abs:		Equation to find concentration(x) or absorbance(y)	
2 ppm	0.071		y=-0.012+0.042x	
2 ppm	0.071			
2 ppm	0.072		Absorbance reading from 6M HCl	
2 ppm	0.072		0.002	
2 ppm	0.074		0.001	
5 ppm	0.199		0.002	
5 ppm	0.199		0	
5 ppm	0.198		0.002	
5 ppm	0.195		Average Absorption:	0.00175
5 ppm	0.199		Concentration:	0.327381

6/15/94 RG When I did the calibration curve: Fe analysis of HCl I used Value Acrue.

When I did the spreadsheet, I first recorded the concentration and absorbance of my calibration standards ~~RG~~ 6/15/94 RG (2.0 ppm and 5.0 ppm) I then wrote down the y=-0.012 + 0.042x using the a (-0.012) and b (0.042) that Value Acrue gave me. I then wrote down the five absorbance readings the AAS gave me and averaged them. I then plugged the average absorption into the y=-0.012 + 0.042x equation to calculate the concentration.

6/17/94 RG

6/17/94 RG

I have begun working with the zeta Potentiae Analyzer. I have begun by trying to find the cell constant of 0.01 m KCl. The temperature is 24.8°C
My measured resistance (R_c) is 748.
The specific conductance is (κKCl) 0.001326
The specific resistance (R_s) is 753.925
Since $K_c = R_c \kappa KCl$
or $\frac{R_c}{R_s}$

$K_c = 748(0.001326) = .9918$
$K_c = 748/753.925 = .9921$

I am going to repeat this procedure so I can see if I can get closer to the 0.971 value Paul Bertotti calculated doing the same thing.
temperature of KCl: 23.3 ~~6/17/94 RG~~ 6/17/94 RG
measured resistance (R_c): ~~938.5~~ 716
specific conductance: 731.52
specific resistance: 0.001367
Since $K_c = R_c \kappa KCl$
or $\frac{R_c}{R_s}$
6/17/94 RG
$K_c = 731.52(0.001367) = 716(0.001367) = 0.9788$
$K_c = 731.52/753.925$ 6/17/94 RG $716/731.52 = 0.9788$

The above 2 calculations were done w/ Probe one
I then did the test with Probe 2:
Temperature KCl: 22.9
R_c KCl: 610
R_s KCl: 737.37
κKCl: 0.001356
$K_c = 610/737.37 = .8273$
$610(0.001356) = .8272$
6/27/94 RG explanation of this begins on page 73

6/21/94 gp

Termination of remaining clinoptilolite-analcime experiments.

ACDTIB - membrane containing the clinoptilolite was broken and clinoptilolite + analcime were mixed.
Solution was filtered into a PP bottle (250ml) and labelled "ACDTIB Solution".
Solids were washed with matrix solution, air dried and placed in a glass vial labeled "ACDTIB* solids."

ACDTIIB - Solution was filtered into 250ml PP bottle + labeled "ACDTIIB solution"
Analcime solid was washed with matrix solution, air dried, and placed in glass vial labeled "ACDTIIB*Analcime"
Clinoptilolite was removed from membrane, washed with matrix solution, air dried, + placed in glass vial labeled "ACDTIIB*Clinoptilolite"

ACDTIIIB - Solution filtered into 250 ml PP bottle & labeled "ACDTIIIB Solution"
Analcime solid was washed with D.I. water, air dried + placed in glass vial labeled "ACDTIIIB*Analcime".
Clinoptilolite was removed from membrane, washed with D.I

water, air dried, and placed in glass vial labeled "ACDTIIIB* Clinoptilolite".

ASEA1 - Solution was filtered into 125ml PP bottle + labeled ASEA1 "Solution". Analcime was washed with matrix solution, air dried, and placed in glass vial labeled "ASEA1* Analcime".

ASEA2 - Solute was filtered into 125ml PP bottle + labeled "ASEA2 Solute". Analcime was washed with DI water, air dried, + placed in glass vial labeled "ASEA2*Analcime".

ASEA3 - Solute was filtered into 125ml PP bottle + labeled "ASEA3 Solute". Analcime was washed with matrix solute, air dried + placed in glass vial labelled "ASEA3*Analcime".

CDUSE3 - No solution remaing. Solid was placed in glass vial labeled "CDUSE3*Clinoptilolite".

CDUSE4 - No solution remaing. Solid was placed in glass vial + labeled "CDUSE4*Clinoptilolite"

RCDU1 - Only a few ml of solute remained and was not saved. Solid was filtered, washed with matrix solute, air dried, and placed in glass vial labeled "RCDU1*Clinoptilolite".

RCDU2 - Only a few ml of solute remained & was not saved. Solid was filtered, washed with DI water, air dried, + placed in glass vial labeled "RCDU2*Clinoptilolite".

RCDU3 - Solute was filtered into a 60ml PP bottle + labeled "RCDU3 Solute". Solid was washed with matrix solute, air dried, + placed in glass vial labeled "RCDU3*Clinoptilolite".

RCDU4 - Solute was filtered into a 60ml PP bottle + labeled "RCDU4 Solute". Solid was washed with DI water, air dried, + placed in glass vial labeled "RCDU4*Clinoptilolite".

RASH1 - Only a few ml of solute remained & was not saved. Solid was filtered, washed with matrix solute, air dried, + placed in glass vial labelled "RASH1* Analcime".

RASH2 - Only a few ml of solute remained + was not saved. Solid was filtered washed with DI water air dried, & placed in glass vial labeled "RASH2*Analcime".

RASH3 - Solute was filtered into 60 ml PP bottle labeled "RASH3 Solute". Solid was washed with matg solute, air dried, & placed in glass vial labeled "RASH3*Analcime".

RASH4 - Solute was filtered into 60 ml PP bottle labeled "RASH4 Solute". Solid was washed with matg solute, air dried, & placed in glass vial labeled "RASH4*Analcime".

RNS1 and RNS2 - solutes were discarded.

6/27/94 RG "The following is an explanation of the work I've done with the Zeta Potential Analyzer:

Measurement of the Electrophoretic Mobility of Wedron Silica

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

DATE WRITTEN: June 20, 1994
DATE REVISED: N/A

CONDITIONS:

1.) W510*60/100*UC*RC*RFe, crushed in Spex ball mill
2.) 0.1 m $NaNO_3$ matrix
3.) pH range 1-4
4.) initial solution volume = 120 ml, initial quartz powder mass = 30 g

OBJECTIVES:

- To measure the electrophoretic mobility of quartz over the pH range of 1 to 4. The electrophoretic mobility can be used to determine the pH of zero charge.
- To further characterize Wedron silica sand used in sorption experiments.

EQUIPMENT:

Orion pH/mV/ISE/°C meter
Combination pH electrode
ATC probe
Analytical balances (Mettler 4600 and 240AE)
Micromeritics zeta potential analyzer w/conductivity probe
Magnetic stir plate w/Teflon stir bars
Filter funnel and collection apparatus
250 ml beakers
Oxford macropipettors, 5 and 10 ml

SUPPLIES:

pH buffer solutions (1, 2, 4, and 7)
Wedron silica sand (W510*60/100*UC*RC*RFe)
ultrapure water
2L 0.01 m $NaNO_3$ stock solution
2L 0.1 m $NaNO_3$ stock solution
Pasteur pipets w/dispensing bulb
1L 1.0 N HNO_3 solution
Filter paper (Fisher P8 or equivalent)
0.01 M KCl solution
Oxford macropipettor tips

6/27/94 RG

PROCEDURE:

Special considerations:

- Clean rotating reservoir and sample cell thoroughly after each sample run. Residue on apparatus electrodes and in reservoir chamber must be minimized.
- All measurements (pH and conductivity/resistance) should be made while the slurry is stirred. Use stir plate and stir bar.

1.) Stock solution preparation

2000 ml 0.1 m $NaNO_3$ stock solution (16.999 g in 2000 g H_2O)
2000 ml 0.01 m $NaNO_3$ stock solution (1.6999 g in 2000 g H_2O)
0.1 N HNO_3 (63.01 g HNO_3 per liter H_2O)

2.) Wedron silica preparation

a.) Fill the Spex ball mill chamber 1/3 to 1/2 full with W510*60/100*UC*RC*RFe sand.
b.) Grind, using tungsten carbide components, for about 15 minutes until sand is powdered.

3.) Determine conductivity cell constant

a.) Using 0.01N KCl, measure the conductivity cell constant.
b.) Follow the procedure detailed in the Micromeritics manual p. 12-13.
c.) Determine the resistance of the KCl solution; measure the temperature of solution; and calculate the cell constant using the equation given on p. 13.
d.) Record cell constant value for later use.

*Note: Steps 4-8 will be repeated as necessary to analyze several silica slurries at the pH values of 4, 3.5, 3, 2.75, 2.5, 2.25, 2.1, 2, 1.9, 1.75, 1.5, 1 (total of 12 solutions).

4.) Prepare silica slurry

a.) Into a tared 250 ml beaker, weigh 30.00 g of powdered Wedron silica.
b.) Add 120 g of 0.1 m $NaNO_3$.
c.) Mix solution thoroughly using a Teflon stir bar and magnetic stir plate.

5.) Adjust pH of the silica slurry

a.) Calibrate the pH meter and probe over the pH range of 1 to 7.
b.) Measure the pH of the silica slurry. Record pH and temperature.
c.) Add 1.0 N HNO_3 dropwise using a Pasteur pipet to achieve the desired pH value (see list above for value).

6.) Determine the electrophoretic mobility of the slurry. Follow the detailed procedure as listed in the Micromeritics manual.

a.) Measure slurry resistance. Record.
b.) Pre-set current (2000 microamps).
c.) Select test (+ or -). If gassing occurs within the sample cell, test polarity should be reversed.
d.) Add slurry to sample cell. Use Oxford macropipet. Weigh cell and record weight.
e.) Add slurry to sample reservoir. Close shutter; connect sample cell and reservoir.
f.) Place reservoir into rotation device, open shutter and start rotation. rotate for about 2 min.
g.) Set test timer for 5 min. Start test.
h.) When test is complete, stop rotation, remove sample cell and re-weigh. Record weight.

7.) Calculate electrophoretic mobility for slurry.

8.) Clean sample cell and reservoir, and prepare next slurry. (Go to step 4).

9.) Record all measurements and calculations on the worksheets provided. Adjust slurry density (and volume as necessary) to compensate for addition of acid.

10.) After completion of each analysis, clean the sample cell and reservoir. Wash with ultrapure H_2O. Remove all silica from cell electrodes. Filter the used silica slurry. Discard the solid and paper into solid waste.

6/27/94 RG Before I explain the procedure, I will explain how to use the Zeta Potential Analyzer:

Rudayna's guide to the Zeta Potential Analyzer

① When you begin everyday you must find the resistance of 0.1N KCl, which will be used as the conductivity cell constant. To do this, stick the probe in the 0.1N KCl so that the two electrodes at the base of the probe are completely submerged. Then turn on the machine MAKING SURE that the function switch is set on resistance, the meter multiplier on x100, the resistance multiplier to 10^4, and the resistance potentiometer (the knob that says resistance) is turned fully clockwise. Now turn the machine on. Turn the resistance knob counterclockwise until the needle deflects to a minimum value (this should occur in quick, jerky motions). If this occurs when the resistance knob is less than 100 move down one level on the resistance multiplier and repeat method until the minimum deflection is obtained. Then find the temperature of the KCl (you will use this to find the conductivity cell constant). Find the specific resistance (R_s) and specific conductance (λ) according the temperature (these numbers are constant depending on temperature). Then use the following equation to calculate the conductivity cell constant:

conductivity cell constant = measured resistance • λ_{KCl}
= measured resistance / R_s

RG 6/27/94

6/27/94 Rg

④ Find the resistance of your sample solution by following the directions outlined in step one of Rudayna's guide to the zeta potential analyzer.
③ Now turn the function switch to current preset. Turn the current knob until the desired current is obtained. Keep in mind that the current value is obtained by multiplying the meter indication by the setting of the meter multiplier.
④ Now turn the function switch to + or - test. You will usually use + test, but if gassing occurs during rotation (next step) move to - test.
⑤ Now fill the sample cell w/ solution and weigh (w/cap), then fill the sample reservoir 4/5 full. Now close shutter door, remove cap from sample cell, and connect cell and reservoir. Put apparatus in rotation device and rotate for 2 minutes, being alert for gassing. Then rotate for the needed time.
⑥ After rotation is complete, remove all from reservoir and cap. Then rewigh. Then carefully clean.

6/27/94 Rg
Important ~~Also~~ notes:
① clean and dry conductivity probe with NH_2O after each use
② the shutter door falls off very easily so make sure to press it on securely after each use

6/27/94 Rg

6/27/94 Rg

Rudayna's guide to the pH probe
This is what I did when following step 5 of the procedure.
① I pressed calibrate and entered 4 as the number of buffers used.
② I methodically put the pH probe in the 4 buffers (1, 2, 4, 7) entering the correct pH value and making sure to wash the probe with NH_2O and dry it between readings.
③ The machine then prints out the results if the slope is NOT between 95-105%, ~~105%~~ 6/27/94 Rg repeat.

When I did the test the following unusual things happened:
Runs 3+7 occured w/ the shutter door falling off and had to be repeated.
Run 4 went for 4 minutes with the shutter closed then 8 with it open.
after Run 7 I changed the current to 0.005 and the time of test to 480 seconds, I forgot about the lengthening of the test in Run 8 and let it run for 5 minutes.

On the following pages are the runs I did and the pH readout for each run:

6/27/94 Rg

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 1

Sample Identification Wedron silica pH=4.02 Date 6/20/94
By RG

Slurry Preparation 30.01g silica w/ 140.92g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stir plate

Slurry Resistance 218 ohms
Concentration 17.56% wt % solids
Temperature 22 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9762 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9978 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.002 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.6710 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.4897 g
Weight Change . . . . . ΔW = 0.1813 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0743

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = -0.0123 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^5)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

6/27/94 RG

BRATION RUN1
PH
H =1.00
4mV 21.9C
H =2.00
3mV 21.9C
H =4.00
mV 21.9C
H =7.00
7mV 21.9C

97.8%
7.000

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN2

Sample Identification Wedron Silica pH=3.53 Date 6/21/94
By RG

Slurry Preparation 29.96g Silica w/ 120.01g 0.1m $NaNO_3$ mixed thoroughly using a TEFLON stir bar and a magnetic stir plate

Slurry Resistance 147 ohms
Concentration 19.98% wt % solids
Temperature 21.5 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9713 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9979 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.002 amperes
Time of Test, t 306 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.3865 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.2743 g
Weight Change . . . . . ΔW = 0.1122 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = ~~0.0859~~ 6/21/94 RG 0.08615

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = -0.00951 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^5)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

BRATION RUN2
PH
H =1.00
1mV 21.5C
H =2.00
2mV 21.5C
H =4.00
mV 21.5C
H =7.00
4mV 21.5C

=97.8%
=7.000

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN3

Sample Identification Nedron silica pH=3.06 Date 6/21/94 By RG

Slurry Preparation 30.01g silica w/123.89g 0.1m $NaNO_3$ mixed thoroughly with a TEFLON stir bar and a magnetic stir plate

Slurry Resistance 200 ohms
Concentration 19.50% wt % solids
Temperature 21.5 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9723 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9979 g/cc
Viscosity, η poise
Dielectric Constant, D
Current, I 0.002 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.4879 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.5176 g
Weight Change . . . . . ΔW = -0.0299 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0836

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.0019 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN3
pH=3.06
.5mV 21.5C
pH =2.00
.8mV 21.6C
pH =4.00
4mV 21.5C
pH =7.00
.4mV 21.5C
=97.6%

The shutter door fell off during this run.

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN4

Sample Identification Nedron silica pH= 3.01 Date 6/21/94 By RG

Slurry Preparation 30.01g silica w/119.99g 0.1m $NaNO_3$ mixed thoroughly using a TEFLON stir bar and magnetic stir plate

Slurry Resistance 113 ohms
Concentration 20.01 wt % solids
Temperature 22 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9723 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9979 g/cc
Viscosity, η - poise
Dielectric Constant, D -
Current, I 0.002 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.8669 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.5815 g
Weight Change . . . . . ΔW = 0.2854 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0864

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = -0.0314 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN4
pH =1.00
.7mV 22.0
pH =2.00
.9mV 21.9
pH =4.00
1mV 21.9C
pH =7.00
1.0mV 21.9
=97.2%
=7.000

This ran 4 minutes with the shutter door open and 5 with it closed

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 5

Sample Identification Wedron silica pH=2.77 Date 6/21/94 By RG

Slurry Preparation 30.00g silica w/130.07g 0.1mNaNO$_3$ mixed thoroughly using a Teflon stir bar and magnetic stir plate

Slurry Resistance 119 ohms
Concentration 19.99% wt % solids
Temperature 22.1 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Liquid Density, ρ_l 0.9977 g/cc
Viscosity, η poise
Dielectric Constant, D
Conductivity Cell Constant 0.9713 cm^{-1}
Function Switch Set to + Test
Current, I 0.002 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.5644 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.5741 g
Weight Change . . . . . ΔW = -0.0097 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0863

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.0010 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

micromeritics®

Form 120/42701/00

RUN5
pH =1.00
.9mV 22.3C
pH =2.00
.1mV 22.3C
pH =4.00
7mV 22.3C
pH =7.00
.2mV 22.3C

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN6

Sample Identification Wedron silica pH=2.53 Date 6/22/94 By RG

Slurry Preparation 30.00g silica w/130.16g 0.1m NaNO$_3$ mixed thoroughly using a Teflon stir bar and a magnetic stirplate

Slurry Resistance 169 ohms
Concentration 19.98% wt % solids
Temperature 22.1 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Liquid Density, ρ_l 0.9977 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Conductivity Cell Constant 0.9713 cm^{-1}
Function Switch Set to + Test
Current, I 0.002 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.4967 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.6601 g
Weight Change . . . . . ΔW = -0.1634 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0850

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.0132 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN6
pH =1.00
1.3mV 25.0C
pH =2.00
3.5mV 22.0C
pH =4.00
.6mV 22.0C
pH =7.00
1.7mV 22.0C

=97.5%
)=7.000
00 06-21-9

6/27/94RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 7

Sample Identification Wedron Silica pH=2.27 Date 6/22/94 By RG

Slurry Preparation 29.98 g silica w/ 120.19g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stirring plate

Slurry Resistance 158 ohms
Concentration 19.87% wt % solids
Temperature 22.1 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9742 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9977 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.002 amperes
Time of Test, t 306 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.4913 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.3351 g
Weight Change . . . . . ΔW = 0.1562 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.085608

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = -0.01241 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN7
CALIBRATION
1-PH
pH =1.00
1.9mV 22.1C
pH =2.00
5.2mV 22.1C
pH =4.00
.5mV 22.1C
pH =7.00
.9mV 22.1C

The shutter door fell off during this run.

6/27/94RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 8

Sample Identification Wedron Silica pH=2.250 Date 6/27/94 By RG

Slurry Preparation 30.01g silica w/ 122.73 g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stir plate

Slurry Resistance 163 ohms
Concentration 19.65 wt % solids
Temperature 23.0 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 6/27/94 .9755 ~~0.997~~ cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9975 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.005 amperes
Time of Test, t 300 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.3294 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.3238 g
Weight Change . . . . . ΔW = 0.0056 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.084295

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = -0.000175 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN8
pH =2.000
5.0mV 22.0C
pH =4.000
.1mV 22.0C
pH =7.000
5.2mV 22.0C
P=97.8%
U=7.000
106 01-01-90

6-27-94 RG
(The date was WRONG on the PH-probe)

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN9

Sample Identification Wedron Silica pH=2.102 Date 6/27/94 By RG

Slurry Preparation 29.99g silica w/120.03g 0.1mNaNO$_3$ mixed thoroughly USING a TeFlon stir bar and magnetic stirplate

Slurry Resistance 119 ohms
Concentration 19.99% wt % solids
Temperature 23.1 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9755 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9975 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.005 amperes
Time of Test, t 420 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.1542 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.2024 g
Weight Change . . . . . ΔW = -0.0482 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.085961

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.00145 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN9

M =4.000
2mV 22.0C
H =2.000
7mV 22.0C
H =4.000
mV 22.0C
H =7.000
9mV 22.0C

=97.8%
=7.000
26 06-27-94

6/27/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN10

Sample Identification Wedron Silica pH=2.025 Date 6/27/94 By RG

Slurry Preparation 29.99g silica w/119.98g 0.1mNaNO$_3$ mixed thoroughly USING a TeFlon stir bar and magnetic stirplate

Slurry Resistance 92 ohms
Concentration 20.00% wt % solids
Temperature 23.1 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Conductivity Cell Constant 0.9755 cm^{-1}
Function Switch Set to + Test
Liquid Density, ρ_l 0.9975 g/cc
Viscosity, η — poise
Dielectric Constant, D —
Current, I 0.005 amperes
Time of Test, t 420 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.2390 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.1352 g
Weight Change . . . . . ΔW = 0.1038 g

$$\phi = \frac{(\text{weight fraction solids} \div \rho_p)}{(\text{weight fraction solids} \div \rho_p) + (\text{weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.08601

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.00319 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (-).

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Form 120/42701/00

RUN10

pH =10.000
1.7mV 21.9C
pH =2.000
5.8mV 21.9C
pH =4.000
.3mV 21.9C
pH =7.000
4.5mV 21.9C

6/28/94 RG

Before I proceed with pasting in the raw data let me explain how I obtained all my information and explain how I did step two of the procedure (Wedron silica preparation).

Rudayna's guide to 8ex Vale mill chamber:

① Do steps 1-5 when changing type of materials to be grinded.
② Run granite through grinder for 15 minutes.
③ Using NH_2O, thoroughly wash down the two balls and the chamber which they go in.
④ Then spray chamber and balls with acetone to let them dry faster.
⑤ Let dry.
⑥ Fill chamber until the two balls are just barely covered with sample. close. 6/27/94
⑦ screw chamber in machine, clooch close hatch, set number of minutes to be grinded and wait

Explanation of raw data:

① Using the Mettler 4600, I tared a 250 ml beaker and Teflon stir bar. I then added ≈30.00g of the powdered Wedron silica sand and tared again. I then added ≈120g 0.1m $NaNO_3$ to the powdered and quickly moved the slurry onto a magnetic stir bar [SLURRY PREPARATION]
② I then calculated the pH by using a correctly calibrated (see page 77 of this notebook) pH probe and adding 1.0N HNO_3 drop by drop until the desired pH is obtained. It is imperative that the slurry be stirring properly on a stir plate while its pH is being calculated. [pH calculation]
③ Find the cell conductivity constant, slurry resistance, and current by following Rudayna's guide to the Zeta Potential Analyzer on page 75 of this notebook

6/28/94 RG

④ Before transfering the slurry into the cell or reservoir, find the temperature of the slurry using the temperature probe. The temperature will correspond with a constant liquid density.
⑤ Look up the sample density of whatever your working with.
⑥ When your finished rotating carefully weigh the cell.
⑦ ΔW equals the final weight of the sample cell - initial weight of the sample cell. 6/28/94 RG (⑥) (⑤)
⑧ Next, find the volume fraction of solids using the following equation: 6/28/94 RG

6/28/94 RG $V_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I}$

6/28/94 RG $\phi\ V_\varepsilon = \frac{\text{Weight Fraction Solids} \div \text{Sample density}}{(\text{Weight Fraction solids} \div \text{sample density}) + (\text{Weight Fraction liquid} \div \text{liquid density})}$

⑨ Now, find the electrophoretic mobility (V_ε) using the following equation:

$$V_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)(\rho_p - \rho_l)}$$

where t = time of test
I = current
ρ_p = sample density
ρ_l = liquid density

6/28/94 RG

6/28/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 11

Sample Identification Nedron silica pH= 1.9000 Date 6/28/94 By RG

Slurry Preparation 30.01g silica w/ 120.00g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stir plate

Slurry Resistance 157 ohms
Concentration 20.01% wt % solids
Temperature 21.9 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Liquid Density, ρ_l 0.9978 g/cc
Viscosity, η — poise
Dielectric Constant, D —

Conductivity Cell Constant 0.9781 cm^{-1}
Function Switch Set to + Test
Current, I 0.005 amperes
Time of Test, t 420 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.4370 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.3641 g
Weight Change . . . . . ΔW = 0.0729 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.0861

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = −0.0017 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (−).

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Form 120/42701/00

RUN 11
CALIBRATION
PH1-PH
1 pH =1.000
170.9mV 21.9C
2 pH =2.000
115.2mV 21.9C
3 pH =4.000
38.7mV 21.9C
4 pH =7.000
-74.1mV 21.9C

SLP=97.7%
ISO=7.000
09:50 06-28-94

6/29/94 RG

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 12

Sample Identification Nedron silica pH= 1.72 Date 6/28/94 By RG

Slurry Preparation 30g silica w/ 120.09g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stir plate

Slurry Resistance 68 ohms
Concentration 19.99% wt % solids
Temperature 23.2 °C
Sample Density, ρ_p . . . . . 2.65 g/cc
Liquid Density, ρ_l 0.9975 g/cc
Viscosity, η _____ poise
Dielectric Constant, D —

Conductivity Cell Constant ____ cm^{-1}
Function Switch Set to + Test
Current, I 0.005 amperes
Time of Test, t 420 seconds

Final Weight of Cell, Sample and Filling Stem . . . . . 35.4209 g
Initial Weight of Cell, Sample and Filling Stem . . . . . 35.4764 g
Weight Change . . . . . ΔW = −0.0555 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids . . . . . ϕ = 0.8596

$$v_\varepsilon = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)\ (\rho_p - \rho_l)}$$

Electrophoretic Mobility . . . . . v_ε = 0.002928 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_\varepsilon \cdot \eta}{D} = \frac{(1.131\times10^6)v_\varepsilon \cdot \eta}{D} = 1.131\times10^6 x \quad x$$

Zeta Potential . . . . . ζ = _____ volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (−).

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Form 120/42701/00

P1 PH =1.000
270.7mV 22.1C
P2 PH =2.000
215.4mV 22.1C
P3 PH =4.000
98.4mV 22.1C
P4 PH =7.000
-74.4mV 22.1C

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 13

Sample Identification Nedron Silica — Date 6/28/94
pH= 1.502 — By RG
Slurry Preparation 30.00g Silica w/ 120.17g 0.1m $NaNO_3$ mixed thoroughly Teflon stir bar and magnetic stir plate

Slurry Resistance 56 ohms — Liquid Density, ρ_l 0.9970 g/cc
Concentration 19.98% wt % solids — Viscosity, η poise
Temperature 25.2 °C — Dielectric Constant, D
Sample Density, ρ_p 2.65 g/cc
Conductivity Cell Constant 0.978A cm^{-1} — Current, I 0.005 amperes
Function Switch Set to + Test — Time of Test, t 420 seconds
Final Weight of Cell, Sample and Filling Stem 35.4995 g
Initial Weight of Cell, Sample and Filling Stem 35.3557 g
Weight Change ΔW = 0.1438 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids ϕ = 0.085872

$$v_e = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)(\rho_p - \rho_l)}$$

Electrophoretic Mobility v_e = −0.00922 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_e \cdot \eta}{D} = \frac{(1.131\times10^4)v_e \cdot \eta}{D} = 1.131\times10^4 x \quad x$$

Zeta Potential ζ = volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (−).

micromeritics® Form 120/42701/00

ELECTROPHORETIC MOBILITY AND ZETA POTENTIAL DATA SHEET
for
ZETA POTENTIAL ANALYZER

RUN 14

Sample Identification Nedron Silica — Date 6/28/94
pH= 1.650 — By RG
Slurry Preparation 30.00g Silica w/ 120.15g 0.1m $NaNO_3$ mixed thoroughly using a Teflon stir bar and magnetic stirring plate

Slurry Resistance 36 ohms — Liquid Density, ρ_l 0.9976 g/cc
Concentration 19.98% wt % solids — Viscosity, η poise
Temperature 22.9 °C — Dielectric Constant, D
Sample Density, ρ_p 2.65 g/cc
Conductivity Cell Constant 0.978A cm^{-1} — Current, I 0.005 amperes
Function Switch Set to + Test — Time of Test, t 420 seconds
Final Weight of Cell, Sample and Filling Stem 35.6283 g
Initial Weight of Cell, Sample and Filling Stem 35.3421 g
Weight Change ΔW = 0.2862 g

$$\phi = \frac{\text{(weight fraction solids} \div \rho_p)}{\text{(weight fraction solids} \div \rho_p) + \text{(weight fraction liquid} \div \rho_l)}$$

Volume Fraction of Solids ϕ = 0.0884 0.08592

$$v_e = \frac{\Delta W \cdot \text{conductivity cell constant}}{\text{slurry resistance} \cdot t \cdot I \cdot \phi(1-\phi)(\rho_p - \rho_l)}$$

Electrophoretic Mobility v_e = −0.03981 cm/sec per volt/cm

$$\zeta = \frac{(36\times10^4)\pi \cdot v_e \cdot \eta}{D} = \frac{(1.131\times10^4)v_e \cdot \eta}{D} = 1.131\times10^4 x \quad x$$

Zeta Potential ζ = volt(s)

NOTE: When the cell electrode is positive (+) and material migrates into the cell, the particles are negatively charged and, by convention, the electrophoretic mobility is negative (−).

micromeritics® Form 120/42701/00

pH =1.000
1.9mV 22.7C
pH =2.000
.9mV 22.7C
pH =4.000
1mV 22.8C
pH =7.000
.5mV 22.8C

=98.6%
=7.000

7/28/94 The following is a crude outline of what I did the past three weeks:

From July 1 - July 18 I did heavy liquid separation of Jw150*VCKRC*RFe sand using sodium polytungstate. This involved pouring the sodium polytungstate in a funnel w/ tubing w/ clamp apparatus. I would then pour the sand in and stir. The unwanted materials would sink near the bottom whereas the wanted quartz floated to the top. I would then drain out the unwanted material and restir. After I got out as much as I could, I would filter the sand out, wash it clean with water and put it in the oven to dry. All the sand had to have this done to them three times.

After that was finished I began learning how to use the titrator in experimentation. I discovered the DG-111-SC pH probe was unusable so I ordered a new one from Fisher.

Since then I have been working on cleaning clay. I did half the clay I used one way and half the other way as to determine which method was more effective. The first process involved me making up two beakers of 50g of clay in 500mL of 3M NaCl. I stirred them for 2 hours, let them settle, decanted the clear 3M NaCl, refilled them with 3M NaCl, and started over with the stirring process. I did this four times. 7/29/94 RG

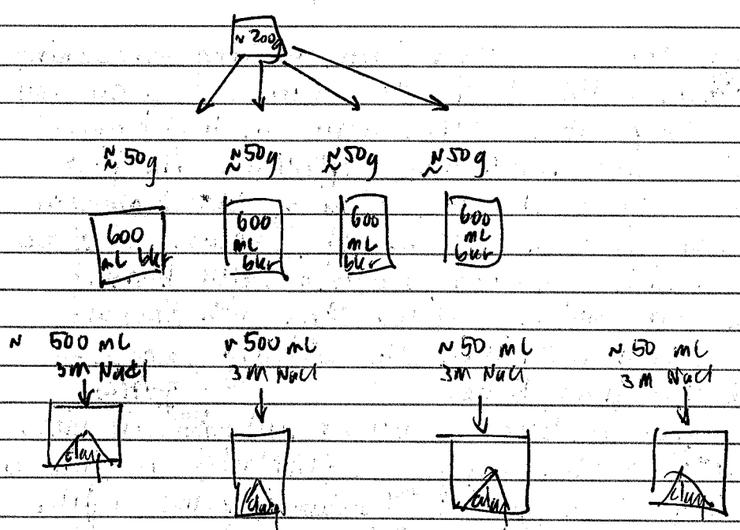
The other method involved putting 50g of clay (mixed 7/29/94 thru 7/29/94) with about 50mL 3M NaCl) in dialysis tubing then putting 7/29/94 submerging the clay filled tubing in 3M NaCl. This 3M NaCl had to be changed one in the morning when I arrived and in the afternoon when I left. 7/29/94

1 Aug 94 PD

Continued preparation of Na-form "chet0" montmorillonite started by R. Ghabbash on 7/24/94.

The montmorillonite clay ~~~500~~ 8/1/94 PD 200 g aliquot has been subdivided into two aliquots of about 100 g each. The 100 g portions were divided into 50 g portions and placed into clean 600 mL beakers. To two 50 g aliquots, about 500 mL of 3m NaCl was added. The other two 50 g portions were mixed with about 50 mL of 3m NaCl and made into a slurry. An oxford macro-pipettor was used to transfer the slurry of clay/NaCl into Spectra Por 4 dialysis membranes. A comparison of Na-exchange and size fraction recovery will be made between the clay suspended in 3m NaCl solution and the clay injected into membranes prior to exchange with NaCl solution. Details of the procedure follow:

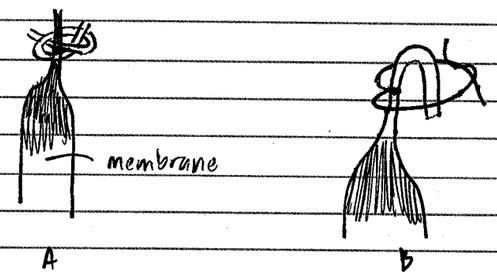
A. Subdivision of Montmorillonite



1 Aug 94 PD

A stir bar (teflon coated) was added to the 600 mL beakers containing 500 mL NaCl solution. The 500 ml solutions were placed on stir plates (29 Jul 94) and mixed at speeds to suspend all clay. On the morning of 1 Aug 94, I removed the beakers from the stir plate, allowed the clay to flocculate and settle, decanted the used NaCl solution and replaced it with fresh 3m NaCl solution. (~500 mL) the mixture was suspended by using stir plate and stir bar and allowed to sit overnight.

The clay slurries (~50mL NaCl) were transferred to cellulose dialysis membranes. About 8 ~~an~~ PD 8/8/94 lengths of membranes ~~s~~ PD, each about 10 inches in length, were cut. 8/1/94 The membrane material was soaked in deionized H_2O until it was pliable. Each membrane was then ~~throu~~ PD thoroughly rinsed with deionized H_2O. One 8/1/94 end of each membrane was tied into a knot, thus sealing off that end. Clay slurry was then transferred into the membrane using an Oxford macropipettor (5 ml). When each membrane was filled (about 1-2" from top), the end was tied using nylon string as shown below:



Once both ends were secure, the membrane was immersed in a 3m NaCl solution (contained in a 1 L beaker).

8/16/94 gp XRD analysis of analcime -

Obj - xrd analcime (characterization)

Method - x-ray diffraction

Equipment - Siemens D500 (Div 06)

Procedure -

① Sample ASH*450/635*uc*WA was mixed with a small amount of Al powder as an internal reference.

② Powder was placed in an aluminum sample holder & taken to Div 06 for analysis on the Siemens D500

③ After analysis powder was saved. Placed in plastic vial & labeled "ASH*450/635*XRD1."

Results are kept in 3-ring binder entitled "Analcime/Clinoptilolite Geochemistry Experiments".

Copies of some results are shown on next two pages.

Jade: Peak Listing — Wed Aug 17 1994 @11:09am

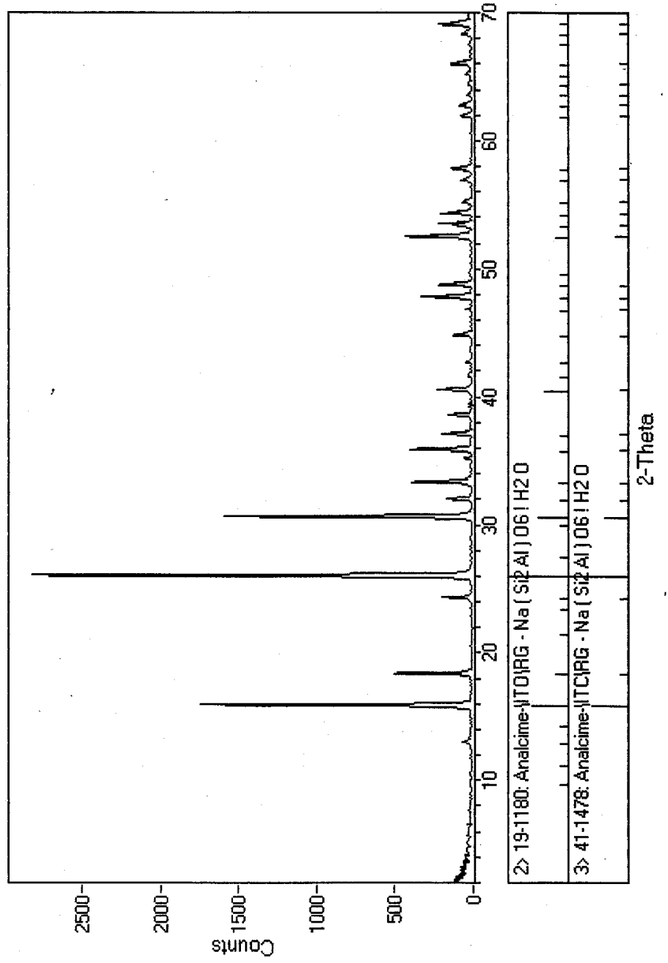
File: ASH635.MDI> 8\17\94 ASH*450/635*XRDI POWDER

Scan Parameters: Radiation = CU_1.540598; Scan Range = 2 - 70; Step Size = .02; Count Time = 1 sec.

Search Parameters: Filter length(pts) = 25; Noise level(sigmas) = 4.0; Intensity cutoff(%) = 2.5-100; 2-Theta Zero (degs) = 0

	Peak-Position 2Theta	Peak-Position d	Centroid-Position 2Theta	Centroid-Position d	Peak & Area are without Bkgrd Bkgrd	Peak	I%	Area	I%	FWHM*
1:	12.794	6.9136	12.800	6.9103	11	55	2.0	634	2.3	0.207
2:	15.704	5.6386	15.692	5.6427	11	1732	61.9	14887	53.2	0.155
3:	18.173	4.8777	18.170	4.8785	10	498	17.8	3716	13.3	0.134
4:	24.154	3.6817	24.156	3.6814	22	178	6.4	1353	4.8	0.137
5:	25.878	3.4401	25.860	3.4425	29	2796	100.0	27975	100.0	0.180
6:	30.494	2.9291	30.471	2.9313	12	1585	56.7	14222	50.8	0.162
7:	31.842	2.8081	31.856	2.8069	13	154	5.5	1462	5.2	0.171
8:	33.168	2.6988	33.197	2.6965	12	377	13.5	3813	13.6	0.182
9:	35.054	2.5578	35.057	2.5576	13	51	1.8	169	0.6	0.060
10:	35.755	2.5092	35.766	2.5085	13	390	13.9	4027	14.4	0.186
11:	36.960	2.4302	36.978	2.4291	12	193	6.9	1745	6.2	0.163
12:	38.483	2.3374	38.470	2.3382	13	147	5.3	1544	5.5	0.189
13:	40.433	2.2291	40.453	2.2280	12	215	7.7	2182	7.8	0.183
14:	41.531	2.1726	41.537	2.1723	12	36	1.3	307	1.1	0.153
15:	42.605	2.1203	42.611	2.1201	11	36	1.3	231	0.8	0.115
16:	44.727	2.0246	44.739	2.0240	10	118	4.2	1324	4.7	0.202
17:	47.755	1.9030	47.749	1.9032	11	320	11.4	3710	13.3	0.209
18:	48.682	1.8689	48.716	1.8677	10	212	7.6	2379	8.5	0.202
19:	52.434	1.7436	52.464	1.7427	10	428	15.3	4905	17.5	0.206
20:	53.455	1.7127	53.427	1.7136	11	212	7.6	2133	7.6	0.181
21:	54.222	1.6903	54.251	1.6895	12	197	7.0	1850	6.6	0.169
22:	55.101	1.6654	55.113	1.6651	14	60	2.1	499	1.8	0.150
23:	56.832	1.6187	56.843	1.6184	10	71	2.5	583	2.1	0.148
24:	57.771	1.5946	57.771	1.5946	9	134	4.8	1713	6.1	0.230
25:	61.888	1.4980	61.896	1.4979	12	73	2.6	587	2.1	0.145
26:	62.710	1.4804	62.740	1.4798	9	81	2.9	1068	3.8	0.237
27:	64.355	1.4464	64.355	1.4465	12	33	1.2	243	0.9	0.133
28:	65.138	1.4309	65.154	1.4306	10	41	1.5	516	1.8	0.227
29:	65.960	1.4151	65.984	1.4146	10	131	4.7	1941	6.9	0.267
30:	67.465	1.3871	67.465	1.3871	10	26	0.9	145	0.5	0.100
31:	68.328	1.3717	68.336	1.3716	13	58	2.1	776	2.8	0.241
32:	69.052	1.3591	69.082	1.3586	16	203	7.3	2437	8.7	0.216

* Intensity values are based on total raw counts.



Pages 1 through 98 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Felix
SwRI - QA
10/14/94

9 Dec 94
PB

Scoping experiment to investigate the influence of added carbonate on the sorption behavior of Np.

Background and procedure are posted below and on following pages. If successful, this experiment will show sorption at pH 10, 11, a that is different that the results of expts Np1, Np2, Np3, and NpC (Notebook GC-012).

8 Dec 94
PB

Scoping experiment to determine the influence of added carbonate on the sorption behavior of Np.

Purpose: To determine if addition of NaOH instead of $NaHCO_3$ and exclusion of an infinite reservoir of $CO_2(g)$ (by capping experimental containers tightly) will impact the observed sorption behavior of ^{237}Np. Our preliminary experiments indicate that a desorption edge develops near pH 9 for both Np-qtz and Np-clinop systems in $NaNO_3$-$NaHCO_3$ matrix. Other published studies of Np-237 sorption (under similar conditions) do not show this desorption edge. It is suspected by me that the presence of significant carbonate in solution has biased our results.

Overview: Solutions of pH 8, 9, 10, and 11 will be made by adding NaOH to our 1×10^{-6} m Np stock solution. After pH adjustment and addition of 0.1 g of Na-form clinoptilolite, the solution containers will be capped ~~tightly~~ tightly and placed in a water-shaker bath at 25°C. After ~ 2 weeks, the solutions will be removed, sampled for pH and Np concentration, and processed for desorption if results indicate that a significant reduction in [Np] has occurred.

9 Dec 94
PB

8 Dec 94
PB

(2)

Procedure (and Equipment):

A. Equipment needed
1. PC bottles (4) 30 mL
2. 1×10^{-6} Np stock solution ~ 101 mL
3. Na-form clinoptilolite 100/200 mesh ~ 0.4 g
4. Eppendorf pipettor w/ tips (various sizes)
5. Orion pH meter w/ probe
6. Analytical balances (AE 240, PM 4600)
7. water-shaker bath with temp. control
8. PP cent. tubes (if desorp. required)
9. fresh NaOH solution (at least 1 M concentration)
10. 7 mL LSA vials
11. LSC cocktail and LS analyzer
12. 0.02 M HNO_3 and 0.1 M HNO_3 for neutralization of experimental solutions as necessary.

B. Procedure
1. Mix and sample 1×10^{-6} m Np stock solution for determination of initial Np concentration/activity.
   - remove two 1 mL aliquots of stock solution and transfer to pre-weighed LSA vials. Add 0.25 ml 0.02 M HNO_3 and 5 mL scintillation cocktail. Analyze for Np.

9 Dec 94
PB

8 Dec 94 PB ③

B. Procedure (cont'd)

2. Transfer ~~100~~ 25 g of Np stock solution into each of 4 30 mL polycarbonate bottles.
   - weigh bottles
   - add 25 g to each (prelim. weight using PM 4600) actual wt measured by AE 240
   - record total weight

3. Add 0.1 g Na-form clinoptilolite to each PC bottle

4. using NaOH solution, adjust pH of clinopt Np... solution to pH 8, 9, 10, and 11 respectively.
   - use 0.1 M NaOH if possible to minimize the "microprecipitation" of Np hydroxide
   - after pH is adjusted cap the bottle immediately and re-weigh.

5. Place exp. containers in water-shaker bath @ 25°C and allow solution/solid to react for ~ 10-14 days.

6. After reaction period, remove containers from water bath, dry and reweigh.

7. Sample experimental solutions for pH and [Np].
   For each exp. solution:
   - open container, withdraw one, 1 mL aliquot and transfer to preweighed and labeled LSA vial.
   - measure pH
   - withdraw a second, 1 mL aliquot & transfer to LSA vial.

8 Dec 94 PB ④

B. Procedure (cont'd)

8. Recap all experimental containers as soon as sampling is complete.

9. Analyze LSA vials, plot ΔNp conc. vs. pH.

9 Dec 94 PB

Following the procedure posted on the previous pages...

- swirled the 1×10^{-6} m Np stock solution for ~ 30 secs to ensure adequate mixing. Transferred ~ 100 g to Teflon FEP beaker.

- removed 2 1 mL aliquots of the stock solution and discharged them into 7 mL LSA vials. The LSA vials had 0.25 mL of 0.02 M HNO_3 added to acidify the contents.

TABLE 1 - INITIAL SAN SAMPLING

vial	vial wt + 0.02 M HNO_3	vial wt + Np soln.	Np soln. sample wt.
NpOH i a	7.6179	8.6250	1.0071
NpOH i b	7.5398	8.5457	1.0059

TABLE 2 - CONTAINER WEIGHTS

exp soln	wt cont. (PB weighed 12/8/94) after add soln.	wt cont. (PB 12/8/94) after add clinop.	wt cont after pH adjust.	final Meas. pH	(°C) Temp
NpOH - 8	12.9343	38.0772	38.1794	38.2063	9.34	18.0
NpOH - 9	12.9775	38.1779	38.2826	38.2601	8.13	18.1
NpOH - 10	12.9482	38.0342	38.1362	38.2895	10.49	18.1
NpOH - 11	12.9959	38.0649	38.1689	38.3324	10.56	18.1

- made 0.1 M NaOH by weighing 0.2167 grams NaOH (lot #: 930429 C Fisher) and diluting to 50 mL in clean volumetric flask. ⇒ 0.1094 molar.

- weighed exp containers (1 oz polycarbonate bottles) and recorded weights in table 2 above.

- transferred ~ 120 g of Np stock solution to a 150 mL FEP beaker. Then transferred ~ 25 g of solution from beaker to each exp. container.

9 Dec 94
PD

- each container was capped and re-weighed. weights were recorded in Table 2.

- approximately 0.1 g of Na-form clinoptilolite was weighed out on weigh paper and transferred to each exp. container. After addition of clinoptilolite, each container was re-weighed. The new weight was recorded in Table 2.

- An Orion 920 pH meter was calibrated using a Ross combination pH electrode. A two point calibration was performed using buffers of pH 7 and 10.

- Using a pasteur pipet, 0.1 M NaOH was transferred dropwise into each exp. container while measuring pH with the probe. One large drop changed pH from ~7.4 to ~9.4. more or less NaOH was added to adjust pH within the desired range. Final pH values for each solution are recorded in Table 2. Note that exp. soln. labeled pH 8 is actually adjusted to pH 9.34 and the container labeled 9 is actually 8.13. Also note the weight change for each solution. Container NpOH-9 has a final weight lower than the weight prior to addition of NaOH because the amount of NaOH added was less than the amount of solution lost during pH measurement. Final container weights are recorded in Table 2.

- Each solution container was capped tightly and placed on a gyratory shaker at 120 rpm. Solutions will be allowed to react/equilibrate for about 2 weeks.

3 Jan 95 — 1/4/95
PD

Np sorption solutions will be sampled for [Np] and pH. An aliquot (0.5 mL) (in duplicate) will be withdrawn from each container, then the pH will be measured. IF pH measurements are okay (at least one/two above 9) the solutions will be prepared for desorption. Desorption will be carried out in polypropylene test tubes in a manner similar to that used for previous sorption experiments.

Sampling Tube

vial	vial wt. + 0.5 ml HNO_3	vial + sample	sample weight	vial wt + HNO_3	vial + sample	sample weight
NpOH 8 a	7.7961	8.2957	0.4996	NpOH 8a	7.8292	8.3289	0.4997
NpOH 8 b	7.8477	8.3521	0.5044	NpOH 8b	7.8339	8.3370	0.5031
NpOH 9 a	7.8250	8.3233	0.4983	NpOH 9a	7.8264	8.3242	0.4978
NpOH 9 b	7.7662	8.2690	0.5028	NpOH 9b	7.8829	8.3851	0.5022

container weight summary

exp soln	wt before Sample	wt after Sample	before pH	after pH	pH
NpOH 8	37.9776	36.9731	—	36.9531	8.63
NpOH 9	38.0277	37.0261	—	37.0172	7.74
NpOH 10	38.0482	37.0449	—	37.0321	9.95
NpOH 11	38.0990	37.0990	—	37.0808	10.10

3 Jan 95 PD

desorption

test tube	Initial wt	wt + clinop + solution	wt + acid added
NpOH 8D	13.9470	20.7200	23.7065
NpOH 9D	13.6446	37.1639	40.1482
NpOH 10D	13.7030	36.4401	39.4183
NpOH 11D	13.5983	37.0741	40.0530

exp soln	wt after transfer	wt. after acid added	
NpOH 8	30.1416	33.1525	
NpOH 9	13.4954	18.4229	
NpOH 10	14.2925	19.2188	
NpOH 11	13.6026	18.5259	

pH meter (orion 920A) was calibrated using pH buffers of 7, 9, and 10. Resulting slope for calibration was 99.9%.

pH values are recorded in container weight summary table.

After sampling NpOH solutions for Np concentration and measuring pH, the solutions were prepared for desorption of the clinoptilolite. 50 mL polypropylene test tubes were pre weighed and labeled NpOH 8D, etc.

Transfer of clinoptilolite from the 30 mL PC containers is difficult due to the geometry of the container and eppendorf pipettor w/tip. As a result, clinoptilolite was transferred to test tube for the NpOH 8 solution only. PD 3/3/95

3 Jan 95 PD

For solutions NpOH 9, 10, and 11, the exp solution was decanted into the test tube while the clinoptilolite remained in the original exp. container. Since little Np sorption occurs on the PC containers, desorption of clinop. in the original containers will likely be little affected. Approximately 5 mL of 0.1 M HNO_3 was added to the clinop. containing solutions while ~ 3 mL of HNO_3 (0.1M) was added to the decanted or clinop. free solutions.

4 Jan 95 PD

LSA vials, including NpOH i-a and NpOH i-b vials, were placed in LSC to be counted. PROTOCOL 27 (10^{-6} M Np A/B) was selected for the counting routine.

Desorption solutions were placed on a gyratory shaker at ~120 rpm. They will be sampled in about 10 days

of note may be the fact that pH 10 solutions were taken up readily by the liquid scintillation cocktail. This is opposite the difficulty seen when trying to mix solutions at pH 10 when adjusted with $NaHCO_3$. The difference in behavior is likely due to the lower ionic strength (and less CO_3 content) associated with the NaOH adjusted solutions as compared to the $NaHCO_3$ adjusted solutions. => First indication that adjustment with NaOH may be preferred over $NaHCO_3$ (at least in the case of Np sorption experiments).

6 Jan 95
PB

Results of LSA of NpOH initial and sorption phase experimental samples are posted below. Preliminary inspection of the data reveals that sorption continued to increase with increasing pH, and the max magnitude of sorption was greater than previously seen in Np-clinop. expt using $NaHCO_3$ as a pH adjustment tool. => An experiment will be designed and run in polypropylene tubes at m/v of 25 mL and 0.5g clinop (m/v = 20 g/L). pH will be adjusted using NaOH, containers will be sealed and thermostatted in water (to maintain temp and minimize CO_2 (g) diffusion into the solutions. Desorption results of NpOH scoping expt will be posted when available (~8 days)

06 Jan 95 00:54 ALPHA/BETA - 1.02 Page #1
Protocol #:27 Np-237 10(-6) User : Mike Almendarez

Time: 999.00
Data Mode: Alpha/Beta Nuclide: NP/PA Discriminator: 126
Background Subtract: 1st Vial

	LL	UL	LCR	2S%	BKG
Beta A:	0.0 -	350	0	0.1	20.65
Beta B:	150 -	350	0	0.5	2.00
Alpha:	150 -	350	0	3.0	0.19

Quench Indicator: SIS
3% 2s alpha region
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

	S#	TIME	CPMA	CPMB	CPMa	a:2S%	SIS	FLAG
BLANK	1	999.00	20.65	2.00	0.189	14.548	133.14	B
ApOH-a	2	13.59	287.44	74.46	326.890	3.002	303.93	
2-b	3	13.72	276.14	70.96	323.863	3.001	308.66	
8a	4	28.64	53.68	13.96	154.978	3.004	298.06	
8b	5	28.27	51.97	14.45	157.080	3.003	308.06	
9a	6	28.31	28.80	6.66	156.787	3.004	278.54	
9b	7	27.13	25.02	6.59	163.652	3.003	294.31	
10a	8	29.39	24.84	6.41	151.053	3.004	293.75	
10b	9	29.52	25.38	7.76	150.353	3.004	319.76	
11a	10	29.40	28.02	7.05	151.001	3.004	297.98	
11b	11	29.97	26.93	7.31	148.092	3.004	310.14	

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 06-Jan-95 05:22
C14 Eff (0-156 keV) = 97.10 %
C14 CHI SQUARE IPA DATA PROCESSED - 06-Jan-95 05:32
C14 Chi Square = 22.30
H3 IPA DATA PROCESSED - 06-Jan-95 05:34
H3 Eff (0-18.6 keV) = 67.95 %
H3 CHI SQUARE IPA DATA PROCESSED - 06-Jan-95 05:44
H3 Chi Square = 27.74
BKG IPA DATA PROCESSED - 06-Jan-95 06:45
Bkg (0-18.6 keV) = 17.98 cpm
Bkg (0-156 keV) = 26.15 cpm
C14 E^2/B (1-156 keV) = 465.38
H3 E^2/B (1-18.6 keV) = 251.84

3/2/95 JP

Auger analysis of analcime -

The following sample powders were delivered to Jim Spencer in Div 06 for analysis by auger spectrometry

label	Description
A	Portion of analcime recovered from experiment ASEA1
B	Portion of analcime recovered from ACDTIIB
C	Portion of analcime recovered from RACDT1A
D	Portion of analcime recovered from RASH1
E	Portion of ASH*200/230*UC*WA (unreacted analcime)
F	Alpha-alumina 8007
G	Weldron quartz sand. 60/100 *UC* RC* RFe *HL
H	Calcite from Nopal I - crystals

4/24/95 PD

Analysis of Np solutions using VIS-NIR spectrophotometry.

Purpose: As an alternative to organic solvent extraction techniques to determine the oxidation state of Np in 1×10^{-6} and 1×10^{-5} m Np solutions, VIS-NIR spec. can be used to determine the presence of peaks for NpO_2^+ and $NpO_2CO_3^-$ in solutions, thus confirming not only the oxidation state (+5) but also the speciation (presence of NpO_2^+, etc.).

The molar absorptivity coefficient for the NpO_2^+ species has been given as $\epsilon = 398\ M^{-1}cm^{-1}$ (Hagan et al. 1966). The peak is located at 981 nm (NIR). As CO_3^{2-} concentration grows an $NpO_2CO_3^-$ peak appears at 991 nm. Peaks have been observed in perchloric and nitric solutions.

By quantifying NpO_2^+, we not only determine its presence and $NpO_2CO_3^-$ but also gain insight into reactions that may occur at the surface of minerals during sorption processes.

On 18 Apr 95, I took a 1×10^{-6} m and a 1×10^{-5} m Np solution (in 0.1 m $NaNO_3$ matrix) along with a 0.1 m $NaNO_3$ blank to B. Herrera (Div 01). ~3 mL of each solution was contained in matched optical glass cuvettes having a path length of 10 mm (1 cm).

The UV-VIS-NIR spectrophotometer is a dual beam instrument (Perkin-Elmer lambda 9) and can accept up to 1cm cells in its present configuration. Since I had only one cell with the matrix, the machine blank correction was run referenced to air followed by analyses of the 1×10^{-5} m Np solution

Hagan P.G. and Cleveland J.M. (1966) The absorption spectra of neptunium ions in perchloric acid solution. J. Inorg Nucl. chem (28) 2905-2906.

24 Apr 95 PD

The resulting spectra were very complex and were not as expected. Examples of the spectra are pasted below and on the following pages (dated 18 apr 95).

The machine was not setup correctly for the way in which the blank - samples were analyzed (I believe). Therefore, on 20 Apr 95, I reanalyzed a new 1×10^{-5} m Np sample along with 2 blank (0.1 m $NaNO_3$) cuvettes.

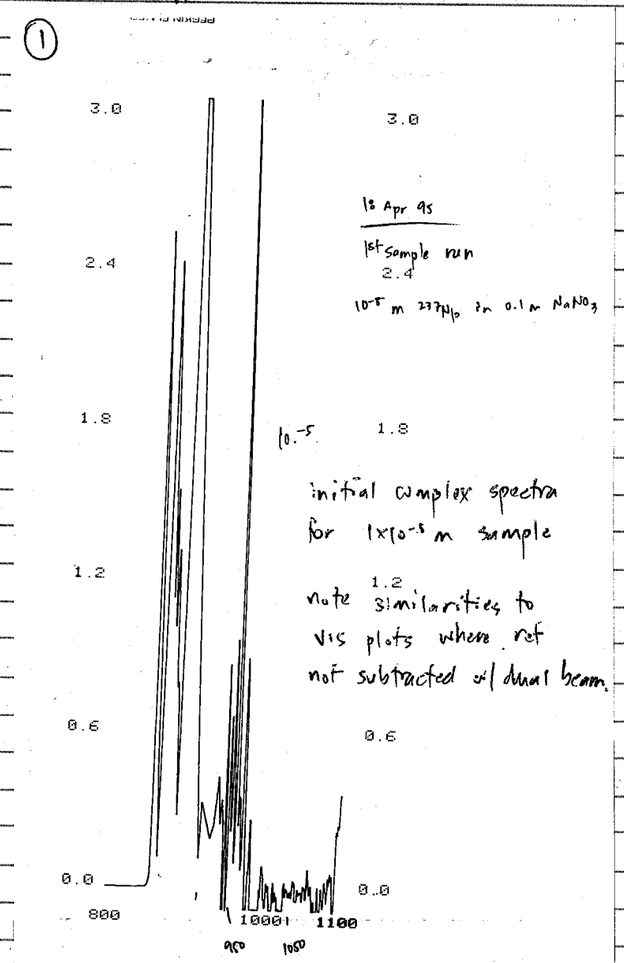
After establishing blank correction with 0.1 m $NaNO_3$ in both the sample and reference beams (see plot of blank correction only 20 Apr 95) the 1×10^{-5} m Np sample was run vs. 0.1 m $NaNO_3$. The results indicated a small (non-distinct) peak was present at ~980 nm but its intensity was only 0.005 - 0.006 (most of the background baseline was 0.001 - 0.002) My calculated $A = \epsilon M L = 398\ M^{-1}cm^{-1}(1 \times 10^{-5}\ M)(1\ cm)$ $= 0.04$ was not correct in fact $(398)(1 \times 10^{-5}\ M)(1) = 0.004$ I had used a path length of 10 (10 mm) but ϵ is in $M^{-1}cm^{-1}$ =) L = 'cm not 10 cm.' =) the A of 0.003 - 0.005 relative to noise was in excellent agreement with expected results but was too low to use for quantitation or qualitation of NpO_2^+ presence. Review of the 18 Apr 95 data shows presence ? of a similarly small peak for 1×10^{-5} m sample. The 1×10^{-6} sample was not run due to low Abs on 1×10^{-5} m samples.

A plot of 0.1 m $NaNO_3$ matrix vs. air shows the intense H_2O absorption in the range of 950-1050. ⇒ very important to have accurate 4/24/95 accurate subtraction of reference blank. I will try to make a 1×10^{-4} m Np solution out of the residual Np solutions remaining from spike #30. If successful I will run that solution in the next week or two. If not enough Np remains, I will make a new 1×10^{-4} m Np solution from another Np spike.

24 Apr 95
PO

selected figures from VIS-NIR spectrophotometry of Np solutions.

①

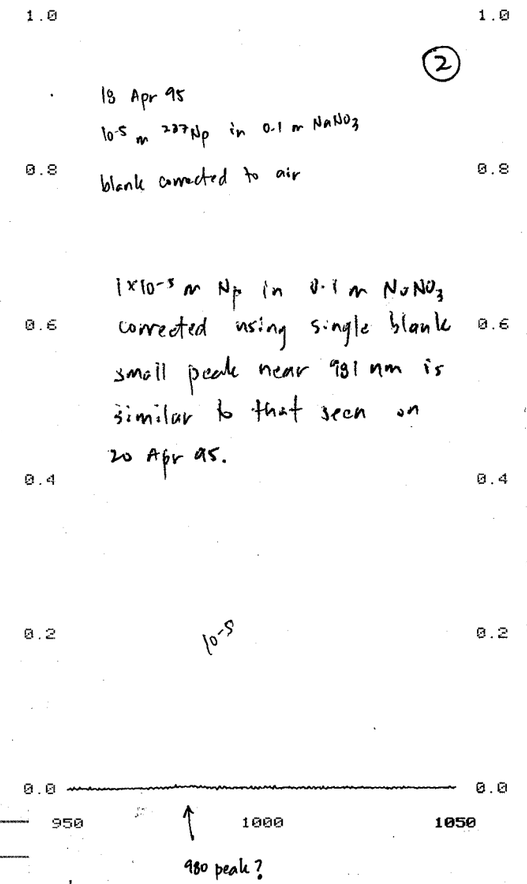


① not sure what spectrum shows, may have been ref vs. air note similarities to vis plots

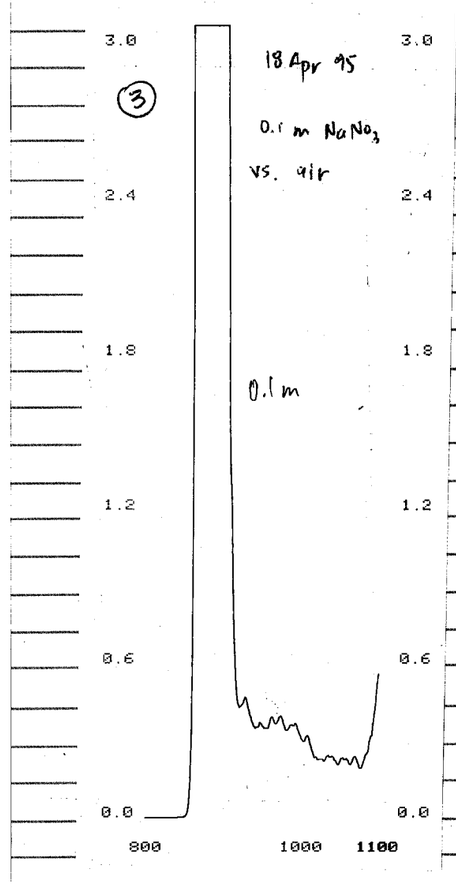
24 Apr 95
PO

Np NIR plots (cont'd)

②



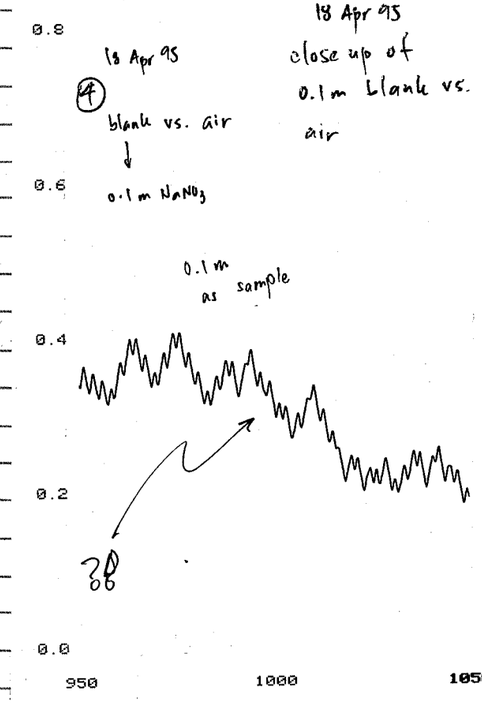
③



② plot from 950 – 1050 nm Compare "peak" @ 981 nm to "peak" in ⑦. ~~Recall PO 24 Apr 95~~ Recall predicted $A = \epsilon C L = 398\ cm^{-1} M^{-1} (1\ cm)(1 \times 10^{-5} M) = 0.004$

③ Ref. solution vs air. large peak ~ 900 does not appear on other plots except "close up of" this in Fig ⑤.

24 Apr 95
Pa

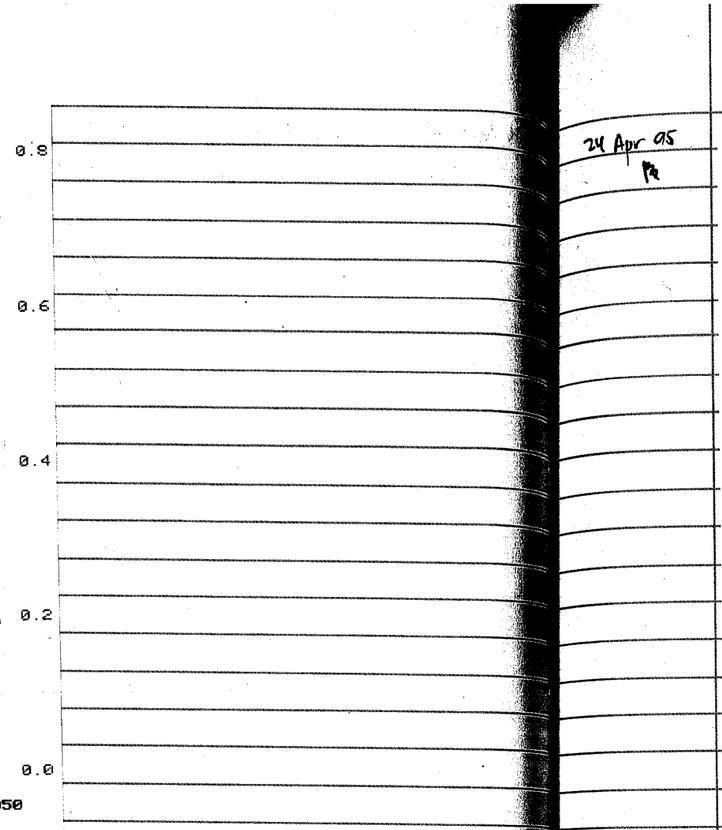


18 Apr 95

④ blank vs. air
↓
0.1 m $NaNO_3$

0.1 m as sample

18 Apr 95
close up of 0.1 m blank vs. air



24 Apr 95
Pa

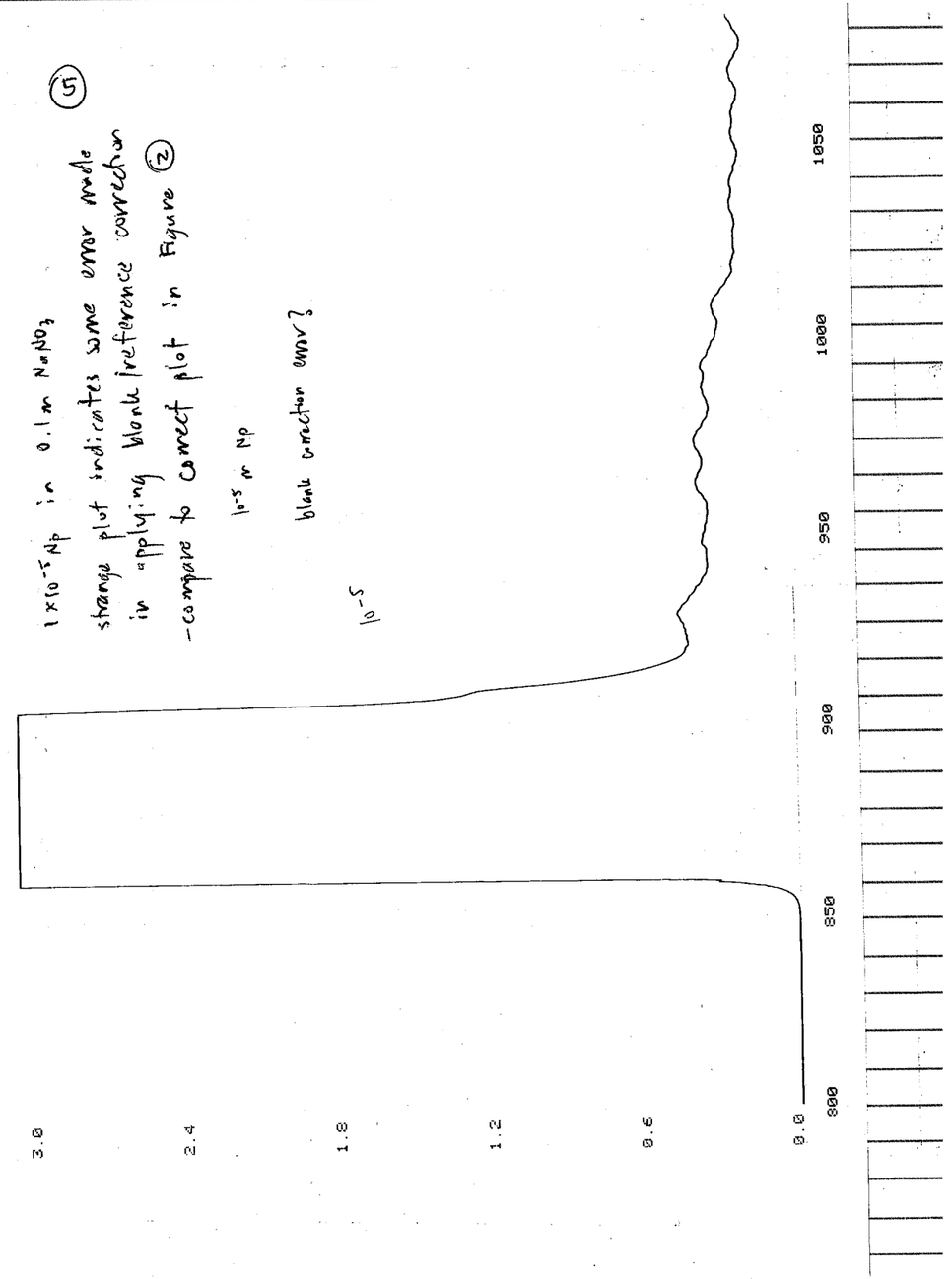
④ plot of ③ from 950 - 1050 w/ expanded vertical scale.

⑤ 1×10^{-5} Np in 0.1 m $NaNO_3$

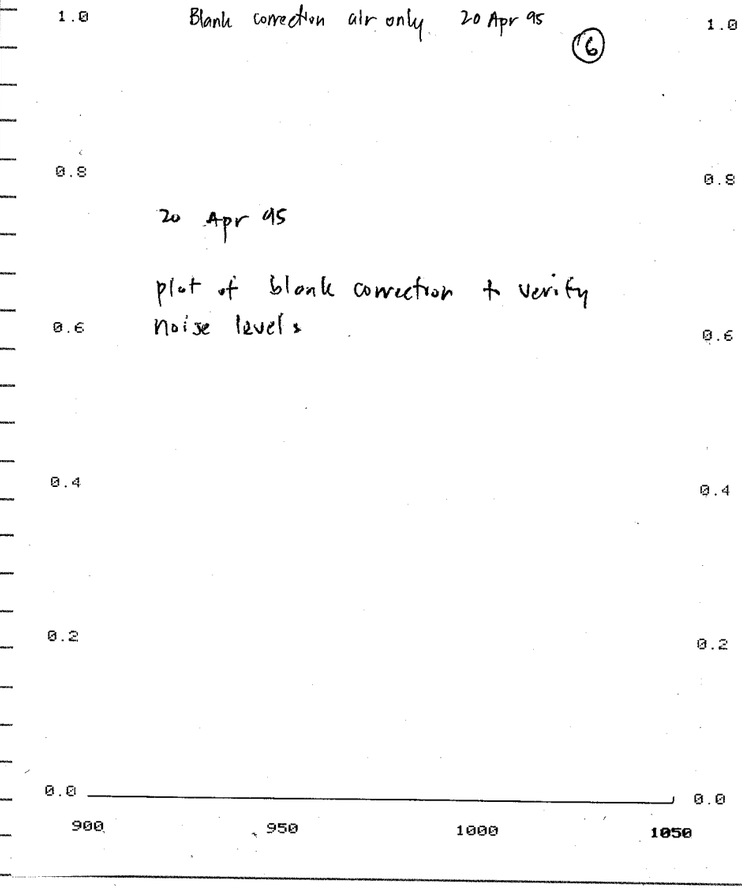
strange plot indicates some error made in applying blank/reference correction
- compare to correct plot in Figure ②

10^{-5} m Np

blank correction error?



24 Apr 95
PB



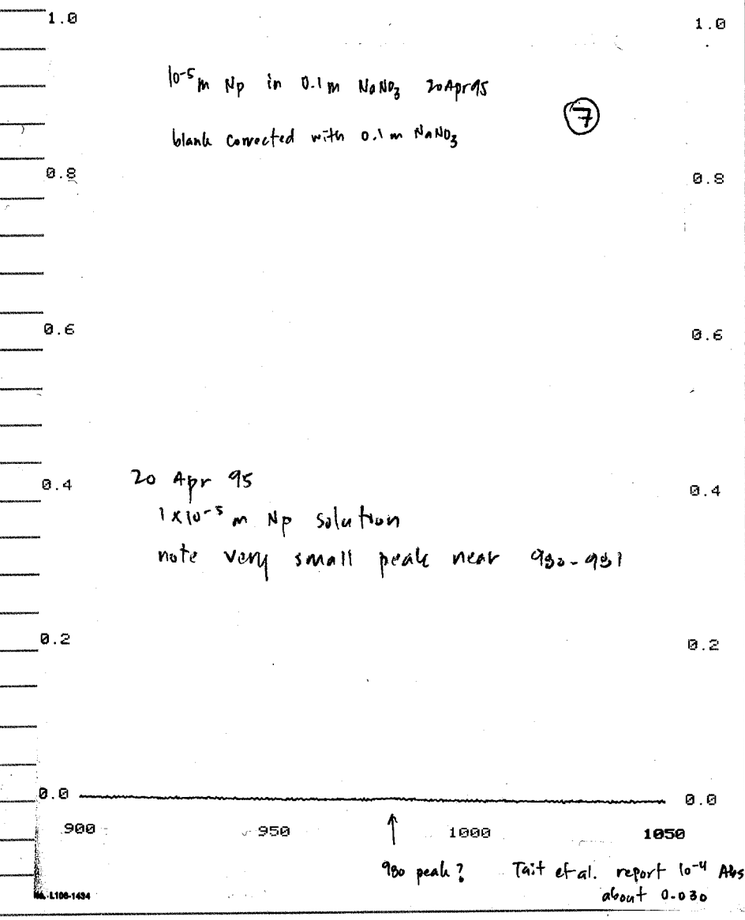
Blank correction air only 20 Apr 95 (6)

20 Apr 95

plot of blank correction to verify noise levels

(6) blank correction plot

24 Apr 95
PB



10^{-5} m Np in 0.1 m $NaNO_3$ 20Apr95 (7)

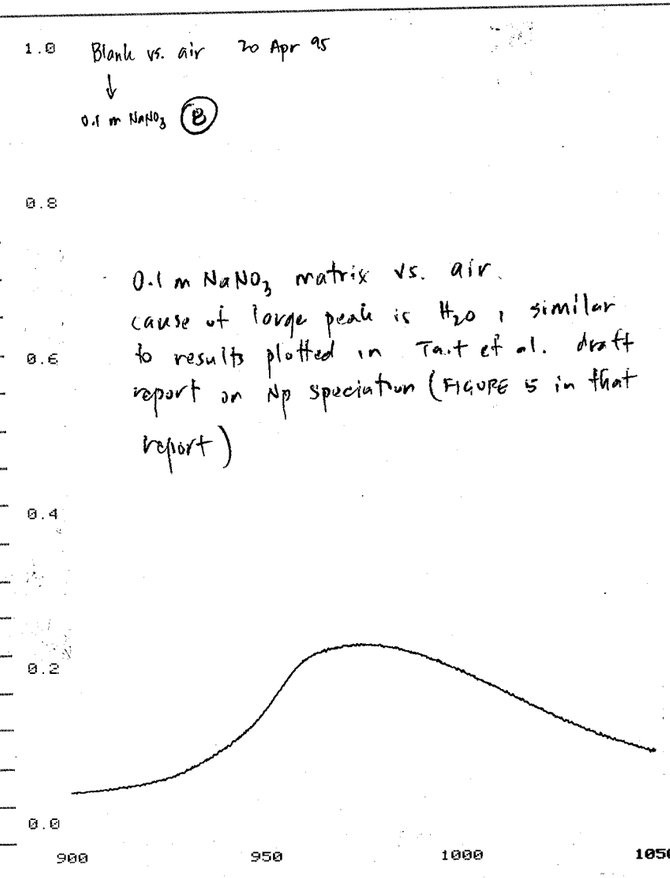
blank corrected with 0.1 m $NaNO_3$

20 Apr 95
1×10^{-5} m Np solution
note very small peak near 980-981

(7) 981 nm peak (NpO_2^+) is present but small Abs from Tait and subsequent calculations indicate that the Abs is correct for 1×10^{-5} m solution compare to (2)

24 Apr 95
PJD

Blank vs. air 20 Apr 95
↓
0.1 m $NaNO_3$ (B)



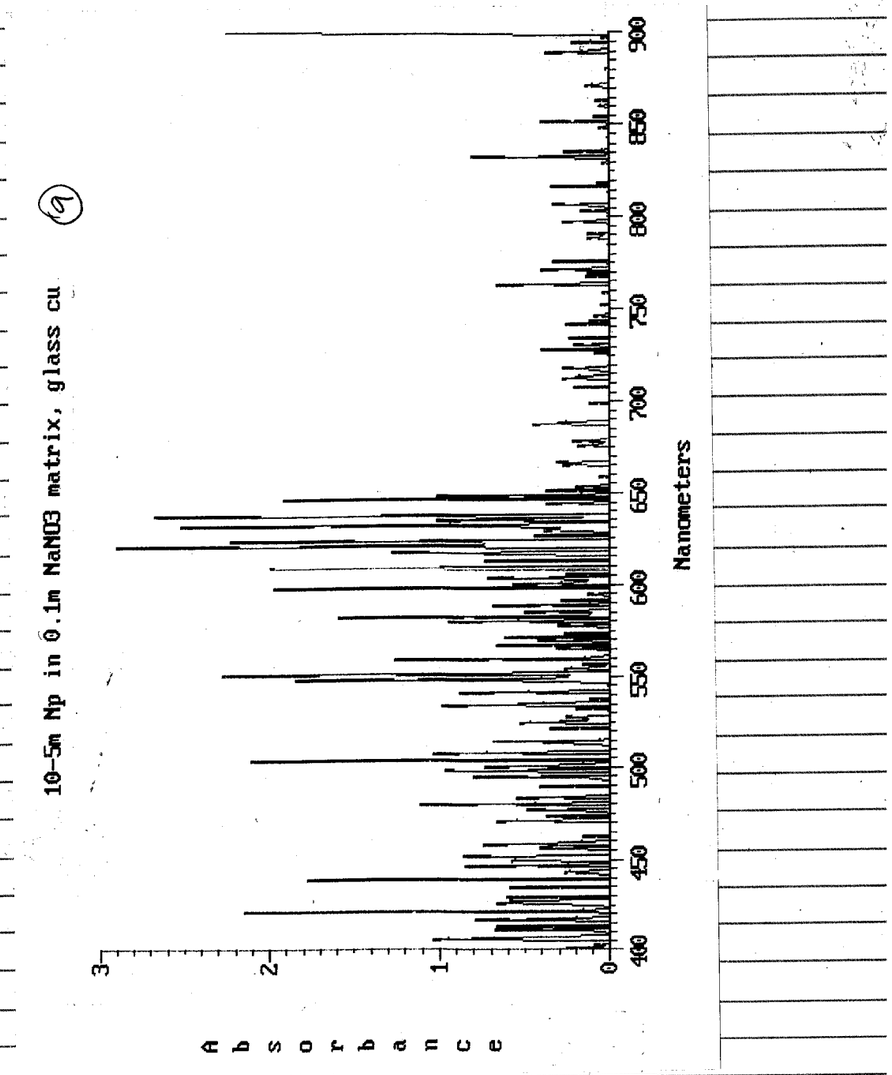
0.1 m $NaNO_3$ matrix vs. air.
cause of large peak is H_2O, similar to results plotted in Tait et al. draft report on Np speciation (FIGURE 5 in that report)

(B) spectrum of 0.1 m $NaNO_3$ referenced to air. Shape and magnitude of peak are as expected and compare well with data from Tait et al. draft report.

24 Apr 95
PJD

VIS Spectra of 1×10^{-5} m Np solution

- spectra taken on MR 101 using 0.1m $NaNO_3$ as a blank and 1×10^{-5} m Np as sample show complex behavior that is not useful. Compare with (1) from NIR plots.



25 Apr 95
PD

Scoping experiments to determine method / effectiveness of separately counting U and Np in solution.

Purpose: U and Np are critical radionuclides (along with Pu) because of their high solubility and low exposure limits. All seem to be very transportable, but all show indications that they will sorb to minerals given the appropriate conditions. Experiments to date have investigated RN sorption as functions of pH, concentration, etc. but ~~not~~ PD yes/no only using single RN's. Under actual repository conditions, one would expect several RN's to be in solution at once. A likely question is how will the sorption of one RN (say for instance U) impact the sorption of another. Especially if the concentration of a nuclide (element) is much greater than another. Because U is much more soluble than Np (or Pu) it might be interesting to conduct sorption expts. in which U was present in large quantities and in the same solution as Np (or Pu). The sorption of Np (or Pu) with and without U could be compared to evaluate what impact, if any, the presence of U had on Np (or Pu) sorption. Analysis of U would also be preferable so that effects on U sorption could be quantified. A problem arises in that our present counting methods, LSA, do not resolve between alpha emitters. Since all nuclides used here for U/Np and Pu emit α's primarily we can not count solutions in the LSA and resolve the individual elements. Alpha spec is available but interference and sample prep time could pose problems. γ-spec is available but suffers from interference and low efficiency. Therefore we will investigate several options for running these competitive sorption experiments by investigating different chemical and counting separation techniques.

25 Apr 95
PD

Nuclides: several are available and on hand

U	Np	Pu
238-U	237-Np	238 Pu
232-U		239 Pu
233-U		241 Pu

other nuclides for U and Np are available, but not readily.

For now, I will concentrate on expts that will include U and Np.

Several papers by C. Sill discuss the separation and precipitation of actinides for α-spec analysis. The precipitation occurs as the actinides are entrained in a $BaSO_4$ ppt which is separated by centrifugation and filtering. All nuclides ppt except for U which can also be seperated by changing oxidation states. The method(s) are describe as having excellent (~98-99%) recovery and are quantitative as long as the ~~concentration~~ PD 4/25/95 quantity of actinides is below 1 mg total.
(Sill says 100 μg)

The are several variations to the Basoy technique most of which arise because of differing initial sample conditions. A modification used in α-spec course claims to scavenge actinides from water samples. One potential problem is that the procedure is written for a large sample (~100 mL) and I would like to use about 1 mL samples

Since ^{233}U spikes are fairly clean, ~~it would be~~ PD 4/25/95 and ^{233}U is easily counted in the LSA, it would be preferable to use ^{233}U and ^{237}Np in the competitive sorption experiment. The γ- signal from U-233 is ~~non-ortho~~ PD 4/25/95 usable and ^{237}Np and 233-U α's interfere. To count w/ LSA, Np must be separated from U in solution. The Basoy method looks promising, this will be evaluated first.

26 Apr 95
PD

Separation of actinides by $BaSO_4$ precipitation.

Procedure: (copy of lab from α-spec course, modifications are noted as I proceed w/ process)

SCOPE

This exercise will illustrate how many actinide elements may be separated from impurities by coprecipitation with barium sulfate. A $BaSO_4$ precipitate will quantitatively carry all elements (except uranium) from radium through californium when present in quantities less than about 1 mg. Our exercise will scavenge actinides from a sample of tap water we have contaminated with known amounts of ^{241}Am and ^{239}Pu. The technique, however, is also applicable to solid samples such as soils or filters which have been processed through a dissolution procedure (as in exercise #5).

MATERIALS/SUPPLIES REQUIRED

400 ml glass beaker

Watch glass

Hot plate

Concentrated H_2SO_4

Barium chloride solution (1.779 g $BaCl_2 \cdot 2H_2O$ per 100 ml H_2O) containing 10 mg Ba/ml

Centrifuge and 50 ml glass centrifuge tubes

Radioactive tracers

PROCEDURE

1. A water sample (~100 ml) which is contaminated with some actinide elements will be provided. Transfer sample to a 400 ml glass beaker and add approximately 3.5 ml concentrated H_2SO_4 and appropriate tracers.

2. Precipitate barium sulfate by dropwise addition of 1 ml of a barium chloride dihydrate solution which contains about 10 mg Ba per ml (17.79 g $BaCl_2 \cdot H_2O$/liter). Solution turns cloudy.

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3. Cover beaker with a watch glass and heat the water sample on a hot plate until fumes of H_2SO_4 are observed. All the water has now evaporated and the remaining acid solution should be clear, i.e., the $BaSO_4$ has redissolved.

4. Take beaker off hot plate and cool. Rotate the acid around the bottom of the beaker to pick up any elements adhering to the glass.

5. Transfer to centrifuge tube using ~20 ml H_2O. The solution will turn cloudy again as soon as the water is added showing that the $BaSO_4$ has reprecipitated.

6. Centrifuge at ~2000 rpm for ~5 minutes. Wash precipitate with 2 or 3 small portions of water.

   The $BaSO_4$ precipitate may either be mounted directly onto a filter or other substrate for gross alpha or gamma counting or processed further for source preparation for alpha spectrometry -- we will save this precipitate to use for source preparation (see exercise #10).

Because I intend to use smaller solution volumes and different RN's, the materials list will be modified.

Materials / Supplies
- 50/100 mL glass beakers
- Np (1x10-6 M) and U (500 ppb) stock solutions ✓ PD 4/26/95
- watch glass
- $BaCl_2$ solution (1.779 g $BaCl_2 \cdot 2H_2O$ in 100 mL dH_2O)
- centrifuge tubes (50 mL polypropylene)
- Eppendorf pipettors w/ tips
- 7 mL LSA vials w/ cocktail (Ultima Gold)
- concentrated H_2SO_4
- hot plate
- 21K centrifuge

Procedure:

3 beakers will be processed: 1) with U only 2) with Np only and 3) U + Np in solution.

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A. - For beaker ① ~ 1 mL U stock solution was transferred to beaker using eppendorf pipettor, the stock solution was diluted with ~ 10 mL dH_2O.

- 1 mL of conc. H_2SO_4 was added to the beaker
- dropwise, ~ 1 mL of $BaCl_2$ solution was added
  the mixture became cloudy
- the beaker was covered w/ watch glass and placed on a hot plate

B. - For beakers ② and ③ ~ 1 mL of Np stock solution, and ~ 0.5 mL U + ~ 0.5 mL Np solutions were added, respectively.

- $BaSO_4$ and H_2SO_4 were added as done with beaker ①
- beakers were covered and placed on hot plate

① blkr + U 30.1844 g
wt blkr 29.1910 g
wt U 0.9934 g

② blkr + Np 29.6177 g
wt blkr 28.7880 g
wt Np 0.9097 g

~~③ blkr + Np + U~~
wt blkr PD 4/26/95

③ blkr + U 29.3663 g
wt blkr 28.8715 g
wt U 0.4948 g

blkr + U + Np 29.8628 g
blkr + U 29.3663 g
wt Np 0.4965 g

C. Solution in all beakers evaporated until fumes of H_2SO_4
- Solutions became clear as $BaSO_4$ dissolved.

D. beakers allowed to cool, remaining acid in beaker swirled to pick up any material on glass.

E. Solution transferred to centrifuge tubes by washing with ~ 10 mL of dH_2O, solution turned cloudy

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PD

F. centrifuge tubes placed into 21K and rotated at 2000 rpm for 5 minutes, blank tube containing dH_2O used for balance.

- ppt. spread over bottom and side of tube in thin layer (recall 21K has an angled rotor, permanent fixed angle) some evidence of thin $BaSO_4$ film on surface of water / H_2SO_4 mix.

- resuspended and recentrifuged tubes @ 5000 rpm for 5 min, some improvement with film, but ppt did not form pellet, instead, as before, ~~it formed~~ PD 4/26/95 as it formed thin coating on tube bottom and side.

G. decanting any supernatant that would not be contaminated with $BaSO_4$ sloughing off tube would be difficult. Satisfied with removal of $BaSO_4$ from solution in tube, I sampled the solution for analysis by withdrawing 1 mL or 0.5 mL aliquots.

SAMPLE VIAL	wt. vial	wt. vial + sample	wt. sample
U a	7.2856	8.3366	1.0510
U b	7.4044	7.9307	0.5263
Np a	7.3211	8.3663	1.0452
Np b	7.3391	7.8664	0.5273
U+Np a	7.2976	8.3532	1.0556
U+Np b	7.3106	7.8401	0.5295

background for LSA 100 - 350 keV = 3

Sample vial	cpm	cpm/g
U a	~~179~~ PD 4/26/95 347	~~168.4~~ PD 4/26/95 330
U b	PD 4/26/95 ~~347~~ 177	~~168.4~~ PD 4/26/95 336
Np a	PD 4/26/95 ~~4.6~~ 5.9	5.6
Np b	PD 4/26/95 ~~5.9~~ 4.6	8.7
U+Np a	PD 4/26/95 85	160.5
U+Np b	164	155.4

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Comparison of cpm for samples ~~shows~~ reveals that the BaSO4 technique is reproduceable for the U bearing solutions and seems to be so for the Np solutions, although more error exists. Not all of the Np is removed (Background is 2.8 - 3.1 cpm, so 2-3 counts are contributed by Np) Counts for the U and U+Np solutions are very similar. These results can't be quantified primarily due to 2 reasons: a) dilution effects are difficult to consider, I have no means of measuring dilution of each sample to correct cpm. b.) samples of original stock solutions were counted but weight of sample was not recorded properly hence, no comparisons can be made to estimate what counts should be. Nevertheless, useful info was gained in that

1.) centrifuge tubes (PP - 50 mL) were inadequate especially when used w/ ZnK, Another tube type or/and centrifuge must be considered.
2.) the BaSO4 film was a problem more tests need to be done to evaluate this
3.) some means of calculation of dilution must be found
4.) the evaporation step took several hours (too long for the method to be useful for a high number of samples).
5.) First estimates seem to show that not all Np is removed nor does all U remain in solution following the BaSO4 ppt. step. This problem must be addressed

LSA Count results

Protocol #:19 U_NP separation

Time: 120.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	100	0	0.0	0.00
Region B:	100 -	350	4	2.0	0.00
Region C:	0.0 -	2000	0	0.0	0.00

Quench Indicator: SIS
cpm from 100-350 keV to check U-Np separation
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

	S#	TIME	CPMA	CPMB	B:2S%	CPMC	SIS
Ub	1	56.55	18.373	176.835	2.00	200.973	593.47
Ua	2	28.83	19.424	347.069	2.00	372.216	552.63
Npb	3	120.00	18.800	4.567	8.54	29.117	171.11
Npa	4	120.00	19.175	5.908	7.51	30.692	192.76
Npa+Ub	5	117.63	18.499	85.012	2.00	109.147	541.49
Np+U b	6	60.92	19.567	164.166	2.00	189.281	525.92
Ui	7	1.85	58.378	5427.027	2.00	5516.216	664.12
Np2	8	48.44	68.642	206.441	2.00	280.842	555.78

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PD

Continuation of U/Np separation using BaSO4 ppt. scavenging

Procedure: A similar expt. will be run using modified quantities of U/Np stock solutions and different centrifuge tubes and centrifuge

cent. tubes = ground glass 10 ml tubes w/ conical tip, graduated
centrifuge = 4-6 place medical centrifuge used for separation of w/Th organic phases

A. Into several 25 PP 50 mL glass beakers, distribute varying quantities of U or Np stock solutions

B. Add 5 mL dH_2O and 1 mL conc H_2SO_4
↳ added quantity to make up to 5 mL volume

C. Heat beakers on hot plate, add 1mL $BaCl_2$ solution dropwise.

D. Bring up heat to remove water and dissolve BaSO4

E. transfer to centrifuge tubes w/ ~ 10 mL dH_2O, reppt. BaSO4

F. centrifuge at setting #6 for 10 min

G. dilute to known graduation on tube, sample for LSA.

Beaker	wt. blr	nuclide	blr+samp.	dH_2O added	H_2SO_4 added	$BaCl_2$ added	wt samp	volume tube
(1)	13.6020	U	18.6287	0	1	1	5.0267	9.8
(2)	13.6839	U	16.1256	2.5	1	1	2.4417	9.75
(3)	13.6261	Np	14.6326	4	1	1	1.0065	9.8
(4)	13.8208	Np	18.7839	0	1	1	4.9631	9.8
(5)	13.6808	Np	16.1200	2.5	1	1	2.4392	10.25
(6)	13.9094	U	14.9230	4	1	1	1.0136	10.2

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PD

Sample vials	wt vial	vial + samp	sample wt.
1a	7.3109	8.4047	1.0938
1b	7.3354	8.4256	1.0902
2a	7.3544	8.4398	1.0854
2b	7.3131	8.4094	1.0963
3a	7.3232	8.4162	1.0930
3b	7.3151	8.4202	1.1051
4a	7.3490	8.4490	1.1000
4b	7.3046	8.4060	1.1014
5a	7.2923	8.3862	1.0939
5b	7.3561	8.4580	1.1019
6a	7.2778	8.3736	1.0958
6b	7.2027	8.3835	1.1008
7a	7.3436	8.3471	1.0035
7b	7.2989	8.3032	1.0043
8a	7.3426	8.3456	1.0030
8b	7.2949	8.2962	1.0013

U stock

Np stock

Protocol #:19 U_NP separation User : Paul Bertetti

Time: 120.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	100	0	0.0	0.00
Region B:	100 -	350	4	2.0	0.00
Region C:	0.0 -	2000	0	0.0	0.00

Quench Indicator: SIS
cpm from 100-350 keV to check U-Np separation
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

	S#	TIME	CPMA	CPMB	B:2S%	CPMC	SIS	
1a	1	1.84	51.630	5455.978	2.00	5511.413	513.54	
1b	2	1.87	46.524	5376.471	1.99	5429.946	512.90	
2a	3	3.92	28.061	2554.082	2.00	2588.520	512.74	
2b	4	3.86	31.088	2593.005	2.00	2629.793	511.49	
3a	5	0.50	38.000	2.000	200.0	52.000	71.904	← LOW COUNT REJECT
3b	6	120.00	19.475	7.100	6.85	31.958	203.32	
4a	7	120.00	19.350	7.600	6.62	33.050	212.42	
4b	8	120.00	19.075	7.508	6.66	32.425	216.16	
5a	9	120.00	19.300	6.992	6.90	31.967	205.54	
5b	10	120.00	20.250	7.067	6.87	32.942	197.61	
6a	11	9.49	41.096	1053.846	2.00	1098.630	497.82	
6b	12	9.17	24.537	1090.840	2.00	1120.938	506.37	

27 Apr 95
PD

Protocol #:21 U_NP separation User : Paul Bertetti

Time: 120.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	100	0	0.0	0.00
Region B:	100 -	350	4	2.0	0.00
Region C:	0.0 -	2000	0	0.0	0.00

Quench Indicator: SIS
cpm from 100-350 keV to check U-Np separation
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

	S#	TIME	CPMA	CPMB	B:2S%	CPMC	SIS
7a	1	0.92	245.652	10913.04	2.00	11200.00	598.83
7b	2	0.93	106.452	10755.91	2.00	10905.38	602.82
8a	3	26.96	83.346	371.105	2.00	460.534	553.23
8b	4	26.64	78.228	375.375	2.00	459.722	560.92

Protocol #: 9 A/B count routine User : Paul Bertetti

Time: 200.00
Data Mode: Alpha/Beta Nuclide: NP/PA Discriminator: 126
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Beta A:	0.0 -	350	0	2.0	0.00
Beta B:	100 -	350	0	2.0	0.00
Alpha:	100 -	350	0	2.0	0.00

Quench Indicator: SIS
standard a/b sample count protocol
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

	S#	TIME	CPMA	A:2S%	CPMB	CPMa	a:2S%	SIS	FLAG
7a	1	0.93	179.57	15.48	76.34	10784.9	1.997	285.75	
7b	2	0.92	168.48	16.06	70.65	10944.6	1.993	280.65	
8a	3	29.91	121.26	3.32	41.83	334.336	2.000	241.05	
8b	4	30.58	121.22	3.28	43.03	327.142	2.000	251.67	

Counting results of samples are listed in the printouts on this and the preceding page. A cpm protocol was used to check for any activity in the Np solutions and to quantify activity in the U solutions. An α/β routine was run on the stock solution samples (7 and 8) (Protocol #9) to see if the activity of the uranium samples would be affected. Comparison of the results from samples 7a and 7b in the printouts above shows no difference in the counting results for U in 100-350 keV range when cpm or α/β was used.

27 Apr 95
PB

Protocol #: 9 A/B count routine User : Paul Bertetti

Time: 30.00
Data Mode: Alpha/Beta Nuclide: NP/PA Discriminator: 126
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Beta A:	0.0 -	350	0	2.0	0.00
Beta B:	100 -	350	0	2.0	0.00
Alpha:	100 -	350	0	2.0	0.00

Quench Indicator: SIS
standard a/b sample count protocol
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

S#	TIME	CPMA	A:2S%	CPMB	CPMa	a:2S%	SIS	FLAG
1	1.98	340.40	7.70	312.12	5051.52	2.000	469.17	
2	2.01	464.68	6.54	369.65	4986.07	1.998	411.21	
3	4.27	207.49	6.72	176.58	2344.03	1.999	442.85	
4	4.20	221.43	6.56	161.19	2385.24	1.998	384.35	
5	30.00	21.83	7.81	3.77	3.267	20.203	157.66	
6	30.00	22.93	7.62	3.60	3.233	20.307	147.23	
7	30.00	25.50	7.23	3.90	3.500	19.518	138.84	
8	30.00	30.73	6.59	4.07	3.667	19.069	122.41	
9	30.00	24.87	7.32	3.90	3.400	19.803	140.62	
10	30.00	23.77	7.49	3.33	3.133	20.628	130.49	
11	10.17	97.44	6.35	73.45	984.169	1.999	394.11	
12	9.92	111.59	6.01	67.64	1008.37	2.000	318.14	

Protocol #:19 U_NP separation User : Paul Bertetti

Time: 30.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	100	0	0.0	0.00
Region B:	100 -	350	4	2.0	0.00
Region C:	0.0 -	2000	0	0.0	0.00

Quench Indicator: SIS
cpm from 100-350 keV to check U-Np separation
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

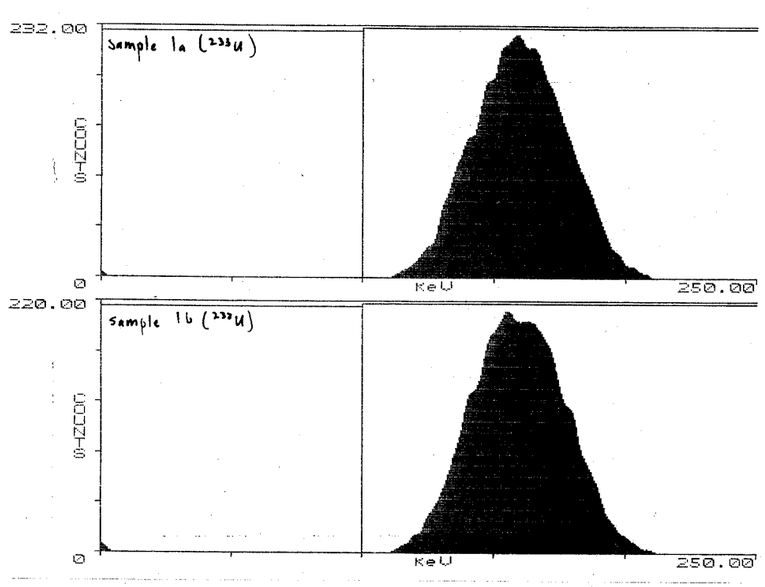
S#	TIME	CPMA	CPMB	B:2S%	CPMC	SIS
1	1.81	45.304	5551.381	2.00	5600.000	520.29
2	1.84	42.391	5442.935	2.00	5490.217	517.31
3	3.94	45.431	2541.878	2.00	2593.655	514.06
4	3.86	61.399	2591.969	2.00	2659.326	510.76
5	30.00	23.100	7.100	13.70	35.633	181.00
6	30.00	20.833	8.133	12.80	35.967	212.41
7	30.00	26.133	7.400	13.42	39.167	170.37
8	30.00	20.833	7.233	13.58	34.033	203.39
9	30.00	25.933	8.000	12.91	40.167	184.44
10	30.00	20.333	6.867	13.93	33.533	196.20
11	9.38	23.348	1066.738	2.00	1095.842	507.28
12	9.33	39.335	1071.811	2.00	1117.578	500.41

27 Apr 95
PB

Counting samples with an a/β protocol (#9) although allows quantitation of Np activity, so it would be beneficial if all samples could be counted using the same protocol. Unfortunately, the U samples did not seem to exhibit the same behavior when counted. Compare counts attributed to β for samples vs. counts attributed to β for stock solutions. Therefore to count U samples, cpm should be used, to count Np samples a/β should be used

The first bit of information that can be obtained from the LSA printouts is that not all of the Np is removed from solution. Use of the glass centrifuge tubes was more successful in that no film was observed and the Baso4 formed a "pellet" at the tube bottom. Assisting was the medical centrifuge which rotated the tubes @ horizontal rather than a fixed angle as in the 21K. But the decantation of all solution without inclusion of some ppt. would not be possible without filtration. Nevertheless the count rate in the 100-350 keV region exceeds the ~~background~~ PB 4/27/95 background by a large margin (background ≤ 0.5 cpm in a/β mode) Considering dilution effects, the signal would seem to be about 10% of the Np. However, the amount of Np as measured by the LSA does not vary with the amount of Np solution originally added. This is contrary to the behavior of the U as indicated by cpm. It is possible that a contaminant nuclide (U?) may be present in the Np solution, but the peak of counts in 100-350 region do not correlate with the 233 peak (compare the spectra on the following pages)

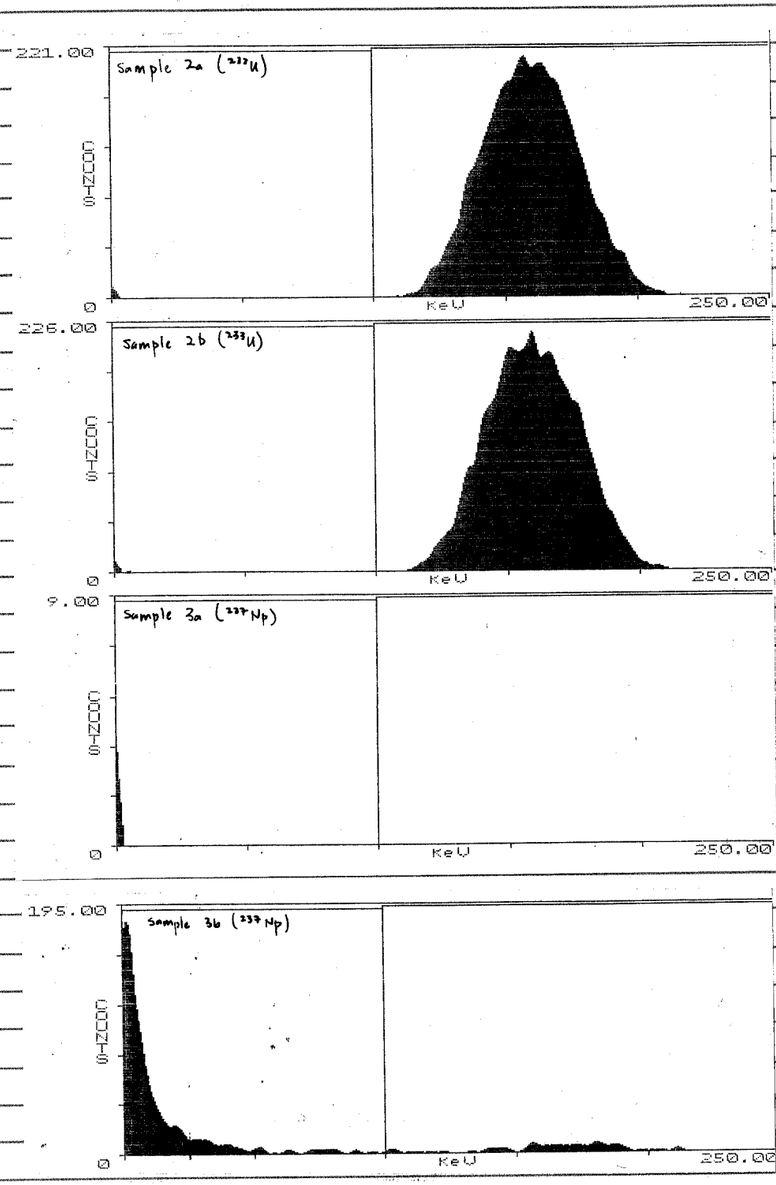
27 Apr 95
PD



LSA spectra from analysis of U and Np samples following separation/isolation via Ba Soy (s) scavenging

spectra are for analysis using protocol #4 on page 130 of this notebook. Note that the Np samples show a strong β signal (presumably due to presence of Pa-233 due to in-growth or not separated) low peak height of Np samples (100-350 keV) has max that is slightly shifted to the right of U sample peak in same region ∴ may not be due to U contaminant (at least U-233) Shape of peak shows the activity is not simply an extension of the β activity seen at low energies.

27 Apr 95
PD



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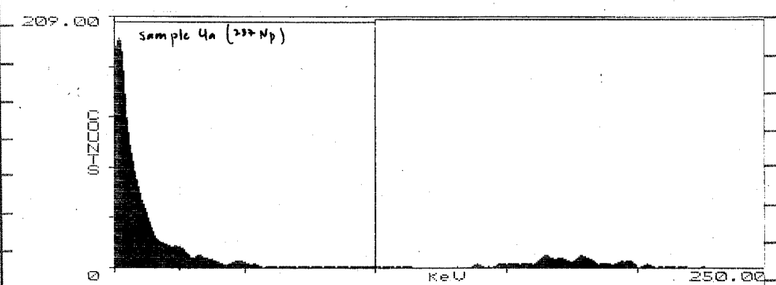

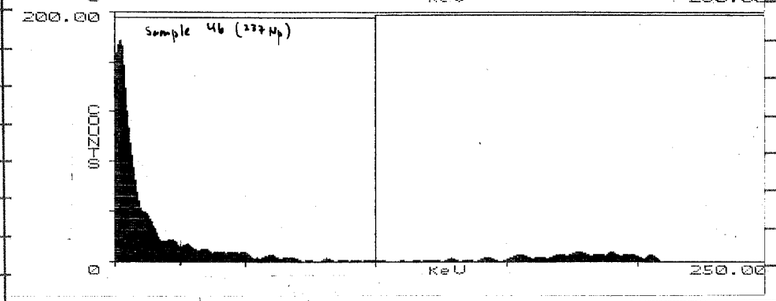

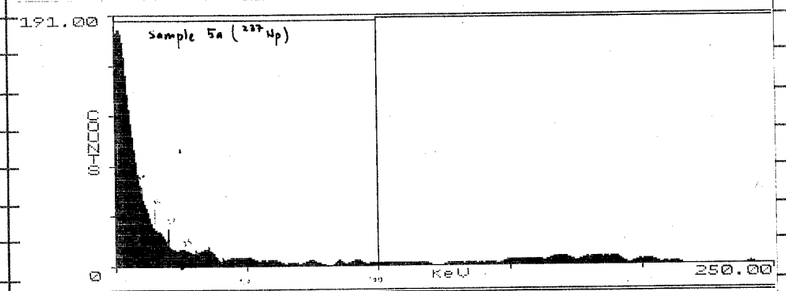

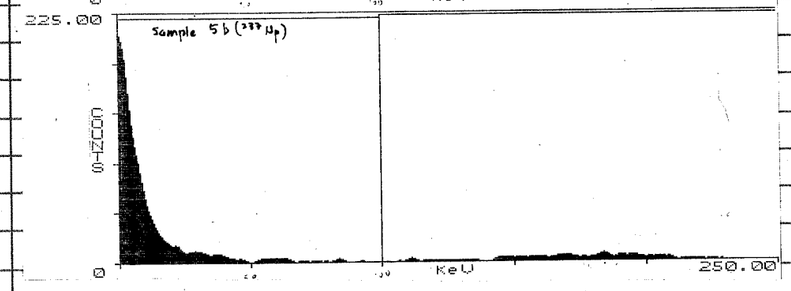

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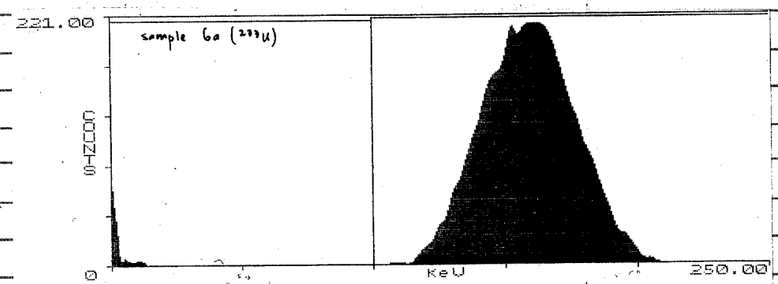

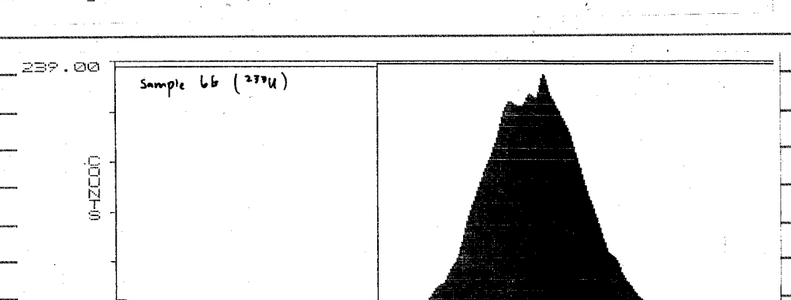

The BaSO4(s) separation efficiency can be calculated by correcting for dilution, sample mass and initial stock solution concentration. Using the constants and conversion factors:

$\lambda_{U-233} = 8.2781 \times 10^{-12}\ m^{-1}$

$\lambda_{Np-237} = 6.1583 \times 10^{-13}\ m^{-1}$

$N_A = 6.022\,1367 \times 10^{23}$ atoms / mole

^{237}Np : 237.048168 g/mole

^{233}U : 233.039629 g/mole

and given the count rates from the USA

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Solution	avg. cpm per gram	cpm/g	Corrected cpm/g (for dilution)
U-stock (233)	10,834	NOT USED PB 4/27/95	

sample	cpm	mass corrected cpm	avg cpm/g	dilution corrected cpm/g
1a	5551.4	5075	5034	9814 ($*9.8/5.0267$)
1b	5442.9	4993	—	
2a	2541.9	2342	2353	9396
2b	2591.9	2364	—	
3a	3.3	3.0	3.0	29.2
3b	3.2	2.9	—	
4a	3.5	3.2	3.3	6.5
4b	3.7	3.4	—	
5a	3.4	3.1	3.0	12.6
5b	3.1	2.8	—	
6a	1066.7	973	974	9821
6b	1071.8	974	—	
7a	10913	10875	10793	10793
7b	10755.9	10710	—	
8a	334.3	333	330	330
8b	327.1	327	—	

Sample	% Recovery
1	91
2	87
3	9
4	2
5	4
6	91

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It appears as though about 10% of the uranium was lost from solution during the separation process. The varying amount of Np recovery is due to the consistent number of measured counts regardless of the original amount Np added. This implies that the counts are due to some source other than Np but also not dependent on Np (for example, if due to Pa-233, one would assume the amount of Pa-233 would also vary with amount Np added). The Np counts may also indicate that some Baso$_4$ (s) may be entrained with the sample's withdrawal. However, one would expect the concentration of Np in the Baso$_4$ (s) to also be somewhat dependent on the original amount of added Np. It is possible that a 'residual' amount of Np remains in each case. Based on the count rate the residual would be

$$\frac{3 \text{ cpm/g}}{6.1583 \times 10^{-13}\ \text{m}^{-1}} = 4.9 \times 10^{12} \text{ atoms/g}$$

$$\left(4.9 \times 10^{12}\right)\left(\frac{\text{mole}}{6.022137 \times 10^{23}}\right)\left(\frac{237.0483168\ \text{g}}{\text{mole}}\right) = 1.9 \times 10^{-6}\ \text{g/g}$$

corrected for dilution (about 10mL)

$\Rightarrow$ 1.9×10^{-8} g Np ~~in 1g standard solution~~ PB: 4/27/95 ~~corrected for dilution~~

Since the method is supposed to handle ~ 1mg of total actinide (or conservatively 100 µg), there is at least a 100 times excess of Baso$_4$ for the amount of Np present. One complicating factor may be that the precip. of Baso$_4$ is too "concentrated", and particles have formed in manner such that not all of the Np is entrained.

The loss of U is large (should be 1% or less). If the reason for lack of Np scavenging given above is true, it would conflict with the behavior of U somewhat. Another explanation is that the U is somehow not oxidized sufficiently to prevent some U(4+) from ppt. with the Baso$_4$ (s). This seems unlikely, but an oxidant

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might be added to provide "insurance" that the U is fully oxidized to (+6).

The dilution factor is not exact because of difficulties in reading the markings on the graduated centrifuge tube. However, the impact is minimal. For example if instead of 9.8 mL a value of 10 mL is used for solution #1, the calculated recovery only changes by ~1% to 92%. Values for dilution are accurate to at least 0.2mL.

An additional problem arises in the recovery of Np. The $BaSO_4$(s) may be dissolved in EDTA, but it is not clear that such a high ionic strength solution will "dissolve" in the LSA cocktail. Success may require Ultima Gold AB or some ionic strength tolerant cocktail.

The high number of Pa ? counts relative to the Np present may indicate that Pa is also retained in solution. An increase in low kev counts (with time) for the Np solutions also points toward the presence of Np in solution (Pa-233 in-growth)

To summarize:

Problems

- about 10% of U lost, regardless of initial quantity added.
- about 2×10^{-9} g Np remain in solution regardless of quantity added.
- still difficult to correct for dilution
- can't separate liquid ppt without filtering
- can't count total then subtract U cpm to get Np because of Pa interference and loss of U cpm in α/β mode.

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Alternatives:

- try variation in $H_2SO_4 - BaCl_2$ ratio
- try $Ba(NO_3)_2$ instead of $BaCl_2 \cdot 2H_2O$
- use oxidant to stabilize U(6+)
- dissolve ppt. after partial decantation and compare recovery of Np

Other Items:

- count U and Np using Ultima-Gold and Ultima-Gold AB/B cocktails to check efficiencies, etc.
- count Np in U-238 solution (should not require separation, no/little signal from U-238).
- polarography could be used for U-238 if necessary (should have little impact from Np if $[Np] = 10^{-6}$ m and $[U] = 10^{-4}$ or 10^{-5} m. 2×10^{-5} m U = 5000 ppb or 5 ppm

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A 1.97×10^{-4} Np solution made from residual spike #30 that was not used in Np/Pa LSA optimization counting was made. Initial characterization of this solution is given on pp. 267-271 of notebook 031. About 0.169 μCi of Np were recovered to make this solution. Since the original spike was about 0.1999 μCi/g, I can estimate about 0.85 g of spike were recovered. When combined with the unused portion of the spike used to make the 1×10^{-5} m Np solution 30A, the total is about 3.89 g of spike $\Rightarrow$ ~1.5 g of spike #30 remains on columns or has been disposed of in the sanitary sewer.

The 1.97×10^{-4} solution will be used to investigate the presence of an NpO_2^+ peak at 981 nm. Based on an $\epsilon = 398\ m^{-1}cm^{-1}$, the solution should give an Abs = 0.08 relative to a 0.02 m HNO_3 matrix.

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The approximate pH and speciation of the 1.97×10^{-4} Np solution can be calculated with EQ3NR.

I will assume 0.02 m HNO_3 matrix and a Np concentration of 46.6 ppm (or 1.97×10^{-4} m) in equilibrium with atmospheric CO_2

NO_3^- = 0.02 moles/L ≈ 0.02 moles/kg

Based on these assumptions, the calculated pH is: 1.75

and NpO_2^+ represents 98% of species (NpO_2^{++} 1.37%) (98.63%)

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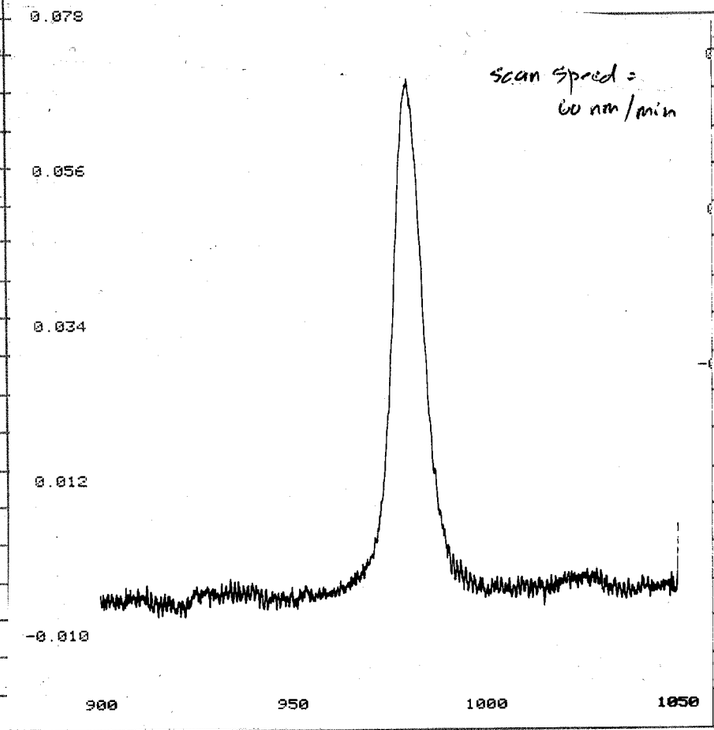
transferred ≈ 2.5 mL of $^{\sim}2 \times 10^{-4}$ M Np solution into a quartz cuvette. Prepared another qtz cuvette as a blank with ≈ 2.5 mL of 0.02 M HNO_3. (two blanks were prepared, each w/ ≈ 2.5 mL)

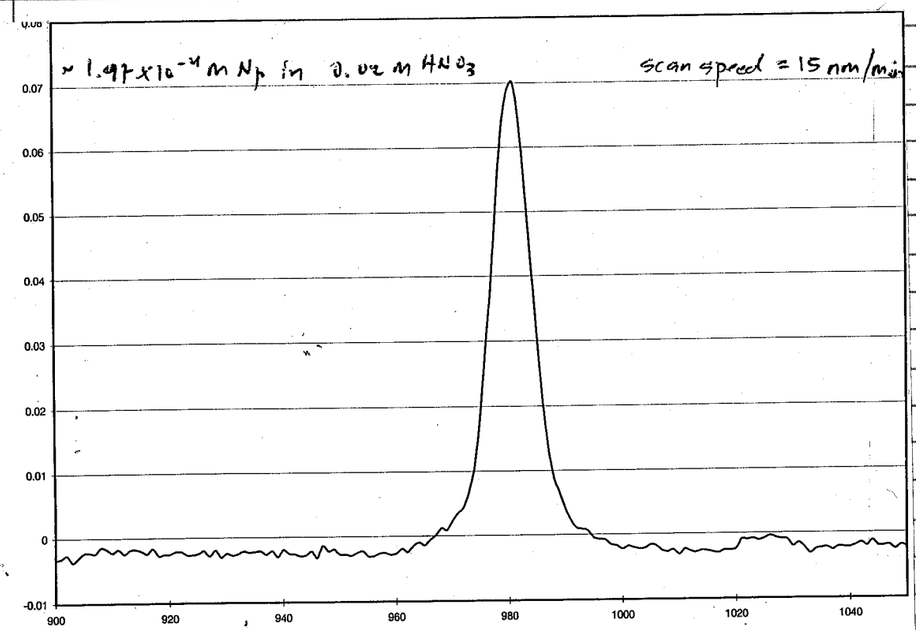
$\sim 2 \times 10^{-4}$ M Np soln was measured using the Lambda 9 NIR @ DIV 01 at a scan speed of 15 nm/min from 900-1050 nm. Hard copy output of the scan and digitized data were not available because of printer problems. Data was transposed from CRT display (150 pts in 1 nm increments.) A second reference and sample scan were run @ 60 nm/min to produce the strip chart plot shown on next page. The digitized data @ 15 nm/min ~~min~~ Np 13Jul95 were imported into Excel and plotted in the fig at bottom of next page.

max abs @ ≈ 981 nm = 0.072 using ε = 398 $M^{-1} cm^{-1}$

$A = \varepsilon L C$

$$C = \frac{A}{\varepsilon L} = \frac{0.072}{398\ M^{-1}cm^{-1}\ (1\ cm)} = 1.81 \times 10^{-4}\ M$$





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Tait et. al. ~~later~~ Pb measured an absorbance of
13 Jul 95
N 0.034 for 1.0×10^{-4} M Np in 0.1 M KNO_3. => Results are very compatible with ours. The next step will be to modify the pH of the 10^{-4} M solution to promote formation of $NpO_2CO_3^-$. However, it may be more important to simply verify oxidation state of more dilute solutions via solvent extraction as the relationship between NpO_2^+ and $NpO_2CO_3^-$ is well established and determination of formation/stability constants is well beyond the scope of this study.

The results to date indicate that Np ~~remains in~~ Lap is July 95 exists in the (+5) state and remains in that state for the stock solutions and their immediate precursors used in our experiments.

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Several mineral powder samples were sent to Div 01 on 13 Jul 95 by J. Prikryl for ICP and total U analysis (U for selected samples only). Included in the samples were aliquots of SAz-1 Ca and Na forms and several reference mineral powders. Samples presented were as listed below:

	Label	Sample	Content
	20A	uran* syn	synthesized uranophane
*	20B	RGM-1	rhyolite
	20C	uran* syn #3	synthesized uranophane
	20D	uran* syn	synthesized uranophane
*	20E	STM-1	nepheline syenite
*	20F	NBS 278	obsidian rock
	20G	uran* syn* 3	synthesized uranophane
*	20H	Na-SAz-1	Na form SAz-1, by P. Mueller
*	20I	cheto	raw SAz-1, Ca form
*	20J	SAz-Na	Na-form SAz-1, freeze dried
*	20K	cheto	raw SAz-1, Ca form
*	20L	SAz-Na	Na-form SAz-1, freeze dried
*	20M	SCo-1	Cody shale
*	20N	SCo-1	Cody shale

All but the uranophane samples are of interest here. Samples were delivered to Div 01 as powdered solids. Analysis on dissolved samples was done by ICP.

Several samples of varying SiO_2 content were provided to test capabilities of the analysis. Except for the Na-form SAz-1 all are standard materials with certified well established chemistry.

Results reported for the (*) samples are given on the following pages.

RGM-1 Rhyolite

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Sample ID	Analysis	Sample Result
20B	Si as SiO_2	74.7%
	Al as Al_2O_3	12.6%
	Fe as Fe_2O_3	1.72%
	Mg as MgO	0.251%
	Ca as CaO	1.06%
	Na as Na_2O	4.36%
	K as K_2O	4.58%
	Ti as TiO_2	0.248%
	LOI (Loss @ 1000°C)	1.11%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	868	4
Beryllium	2	2
Boron	<20	20
Cadmium	<4	4
Chromium	12	4
Cobalt	<10	10
Copper	16	4
Lead	25	10
Lithium	64	4
Manganese	282	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	126	76
Selenium	<10	10
Silver	<10	10
Strontium	106	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	32	20

Element	BHVO-1	MAG-1	QLO-1	RGM-1	SCO-1	SDC-1	SGR-1	STM-1
As (ppb)	5-332 (5)	6-112 (3)	4.5-160 (3)	109±9 (3)	19-126 (2)	3-300 (6)	19 (1)	8-395 (5)
Al (%)	7.30±0.12 (8)	8.65±0.15 (6)	8.60±0.08 (6)	7.29±0.09 (6)	7.21±0.20 (7)	8.50±0.20 (6)	3.53±0.23 (5)	9.79±0.12 (5)
As	1.5 (1)	6 (1)	< 5	2.9 (1)	11-54 (2)	< 5	70 (2)	< 5
Au (ppb)	1.5±0.3 (3)	2.5 (2)	1.3 (2)	0.33 (2)	2.4 (2)	1.5 (2)	10 (2)	0.40 (2)
B	2.3 (1)	138±15 (3)	38±4	29±2 (3)	68±4 (4)	10-30 (3)	42±15 (4)	< 10
Ba	142±18 (12)	490±35 (8)	1400±20 (7)	820±40 (8)	580±100 (11)	620±60 (8)	320±20 (9)	590±20 (7)
Be	0.90 (1)	2.84±0.13 (3)	1.3±0.2 (4)	2.5±0.4 (4)	1.7±0.2 (3)	3.0±0.6 (4)	0.88 (2)	8.9±0.2 (3)
Bi (ppb)	15±4 (3)	360 (2)	65 (2)	280 (2)	190 (1)	277 (2)	1030 (1)	110-250 (2)
C	120±60 (4)	24400±1000 (3)	29±2 (3)	35±10 (5)	8500-1500 (4)	270±120 (3)	2.85 (2)	80±20 (3)
Ca (%)	8.18±0.12 (7)	1.04±0.20 (5)	2.27±0.06 (5)	8100±600 (7)	1.87±0.14 (6)	1.02±0.03 (6)	6.12±0.23 (3)	0.79±0.06 (5)
Cd (ppb)	120 (1)	454 (1)	< 10000	< 0 (1)	150 (1)	110 (1)	1030 (1)	< 1500
Ce	41±4 (6)	94±7 (5)	57±3 (4)	53±6 (5)	62±6 (6)	154±12 (5)	42±10 (5)	3-530 (7)
Cl	92±4 (4)	31200±400 (5)	218±14 (6)	490±40 (6)	48±14 (4)	33±10 (5)	29±13 (3)	440±30 (6)
Co	45±1 (6)	20±2 (7)	2.2±0.4 (6)	2.0±0.3 (6)	10±1 (9)	18±2 (6)	12±1 (6)	0.3-11 (6)
Cr	300±30 (7)	105±13 (5)	1.6-10 (4)	2.5±0.4 (4)	62±5 (6)	49±7 (4)	32±12 (4)	2.6±1.1 (5)
Cs	0.086 (2)	8.3±0.5 (4)	1.8±0.2 (4)	9.9±0.4 (4)	2.5±0.5 (4)	3.83±0.02 (4)	5.2±0.4 (4)	1.52±0.08 (4)
Cu	137±6 (5)	30±3 (6)	29±3 (6)	10.7±0.6 (6)	30±2 (8)	30±2 (6)	66±4 (7)	4±2 (9)
Dy	4.8±0.2 (3)	3.2	< 3.2	4.3 (1)	3.8 (2)	< 3.2	< 3.2	7.8 (1)
Er	2.0±0.3 (3)	< 2.2	< 2.2	< 2.2	2.5 (1)	< 2.2	< 2.2	4.4 (1)
Eu	2.0±0.4 (8)	1.6±0.2 (5)	1.55±0.12 (4)	0.77±0.12 (4)	1.2±0.2 (5)	1.8±0.2 (5)	0.60±0.10 (4)	3.6±0.4 (6)
F	380 (1)	970 (2)	275±22 (3)	360±40 (3)	750-1500 (3)	600 (2)	1070 (1)	930±60 (3)
Fe (%)	8.49±0.18 (10)	4.86±0.26 (7)	3.02±0.09 (8)	1.31±0.03 (10)	3.59±0.09 (6)	4.91±0.12 (8)	2.07±0.14 (7)	3.64±0.14 (9)
Ga	22±3 (3)	21.4±0.4 (3)	18±1 (3)	14±2 (3)	12±2 (4)	25±5 (3)	11±2 (3)	37±1 (3)
Gd	7±2 (7)	7±2 (4)	5.2±1.4 (3)	3.1 (1)	4.1 (4)	7.7-9.9 (3)	1.7-5±2 (2)	12±3 (3)
Ge	1.6 (2)	< 1	1.3 (2)	1.26 (2)	< 1.0	1.54 (2)	< 1.0	1.38 (2)
H	190 (1)	1200-7800 (2)	540 (1)	840 (1)	4460 (1)	2060 (1)	30100 (1)	-
Hf	4.2±0.2 (7)	3.6±0.7 (5)	4.4±0.2 (4)	6.1±0.5	4.26±0.10 (4)	8.1±0.1 (4)	1.35±0.04 (5)	27±1 (5)
Hg (ppb)	4.0 (1)	-	-	-	-	-	-	-
Ho	0.94 (2)	< 1.0	< 1.0	< 1.0	0.93 (1)	< 1.0	< 1.0	2.1 (2)
In	< 4.6	< 4.6	< 4.6	< 4.6	< 4.6	< 4.6	< 4.6	0.087 (1)
Ir	0.00044 (1)	< 6.8	< 6.8	< 6.8	< 6.8	< 6.8	< 6.8	< 6.8
K (%)	4600±700 (12)	3.0±0.2 (6)	2.98±0.09 (8)	3.59±0.08 (9)	2.28±0.08 (6)	2.72±0.07 (8)	1.43±0.13 (5)	3.57±0.06 (6)
La	16.7±0.8 (3)	46 (2)	31 (2)	25 (2)	35±10 (4)	42-150 (3)	19-32 (2)	170±30 (4)
Li	4.5 (2)	76±4 (3)	24 (2)	51±9 (4)	43 (2)	34 (2)	127 (2)	29±7 (4)
Lu (ppb)	320 (2)	400 (1)	420 (1)	420 (1)	370 (2)	490 (1)	200 (1)	660 (2)
Mg (%)	4.30±0.12 (8)	1.86±0.12 (5)	0.61±0.04 (5)	0.170±0.012 (7)	1.61±0.14 (5)	1.01±0.06 (6)	2.80±0.22 (4)	0.055±0.010 (5)
Mn	1270±40 (8)	810±160 (6)	720±70	282±17 (8)	420±40 (8)	900±60 (7)	290±30 (7)	1720±140 (6)
Mo	0.95±0.05 (5)	1.9 (2)	2.6±0.4	2.3±0.6 (6)	1.3±0.1 (3)	0.7-1.9 (2)	35.6±0.5 (4)	5.3±0.4 (5)
N	-	800 (1)	-	-	-	-	-	-
Na (%)	1.64±0.06 (8)	2.77±0.19 (6)	3.09±0.10 (7)	3.00±0.10 (10)	0.64±0.05 (6)	1.50±0.06 (8)	2.09±0.14 (4)	6.61±0.14 (7)
Nb	19±2 (10)	9.5±1.4 (4)	11±2 (6)	9±2 (7)	9±1 (4)	18±3 (8)	5.27±0.06 (3)	270±20 (4)
Nd	24±6 (6)	44±3 (4)	27±7 (5)	18.2±0.8 (4)	26±2 (6)	41±6 (5)	14±3 (4)	82±5 (4)
Ni	117±18 (9)	54±8 (8)	2±1 (4)	1-14 (5)	28±2 (7)	41±10 (7)	32±3 (7)	2.1±0.6 (4)
O (%)	-	46.1 (2)	47.8 (2)	49.2 (2)	50.8 (2)	-	-	-
Os	< 22	< 22	< 22	< 22	< 22	< 22	< 22	< 22
P	1220±130 (6)	760±65 (3)	1100±80 (4)	205±6 (4)	1000±200 (4)	800±200 (6)	1330±140 (3)	710±20 (5)
Pb	4±2 (3)	25±4	21±1 (3)	22±1 (3)	29±2 (4)	24 (2)	38±5 (3)	17±1 (3)
Pd (ppb)	3.5 (1)	< 220	< 220	< 220	220	30,000 (1)	< 220	< 220

data for STM-1
RGM-1 and SCO-1
from Gladney & Goode, 1981

STM-1 Nepheline Syenite

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Sample ID	Analysis	Sample Result
20E	Si as SiO_2	67.2%
	Al as Al_2O_3	18.7%
	Fe as Fe_2O_3	5.35%
	Ca as CaO	1.11%
	Na as Na_2O	10.6%
	K as K_2O	5.07%
	Ti as TiO_2	0.135%
	Mn as MnO	0.248%
	P as P_2O_5	0.195%
	LOI (Loss @ 1000°C)	1.70%
	TOTAL	110%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	598	4
Beryllium	9	2
Boron	<20	20
Cadmium	<4	4
Chromium	11	4
Cobalt	<10	10
Copper	4	4
Lead	16	10
Lithium	38	4
Magnesium	588	19
Molybdenum	<4	4
Nickel	<12	12
Selenium	<10	10
Silver	<10	10
Strontium	706	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	260	20

Element	BHVO-1	MAG-1	QLO-1	RGM-1	SCO-1	SDC-1	SGR-1	STM-1
Pr	5.6 (2)	7.72 (1)	6.1 (1)	4.1 (1)	5.43 (1)	8.73 (1)	6.86 (1)	19.4 (1)
Pt (ppb)	< 10	< 6800	< 6800	< 6800	< 6800	< 6800	< 6800	< 6600
Rb	10±2 (4)	152±3 (5)	77±9 (5)	156±4 (5)	118±9 (5)	128±7 (5)	85±7 (5)	120±6
Re	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Rh	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0	< 1.0
Ru	< 0.46	< 0.46	< 0.46	< 0.46	< 0.46	< 0.46	< 0.46	< 4.6
S	101 (1)	4600±600 (3)	13 (1)	75 (2)	600-1200 (3)	580 (2)	17500 (2)	22 (1)
Sb	0.17±0.01 (4)	0.91±0.09 (5)	2.1±0.4 (6)	1.29±0.14 (5)	2.51±0.06 (6)	540±10 (4)	3.3±0.4 (6)	1.65±0.03
Sc	30±2 (7)	17±2 (7)	10±1 (7)	5.0±0.8 (7)	10.6±0.9 (6)	17±2 (7)	4.7±0.6 (4)	0.678±0.00
Se (ppb)	10-140 (3)	1150±160 (3)	10 (2)	8 (2)	850 (2)	36 (2)	3400±300 (4)	9-30 (2)
Si (%)	23.3±0.4 (7)	23.4±0.5 (5)	30.6±0.5 (6)	34.0±0.6 (5)	29.2±0.4 (4)	30.5±0.6 (5)	13.1±0.1 (3)	27.8±0.4
Sm	6.1±0.7 (7)	7.8±0.9 (4)	4.9±0.5 (4)	4.3±0.4 (4)	5.3±0.4 (4)	8±2 (5)	2.7±0.3 (4)	15±3 (6)
Sn	2.15 (1)	5.0 (2)	2.3-4.2 (2)	3.9 (2)	4.1 (2)	3.0 (2)	1.58 (1)	10±3 (3)
Sr	440±70 (9)	156±19 (10)	348±19 (8)	114±13 (9)	188±26 (11)	200±20 (9)	390±60 (9)	730±30 (7)
Ta	1.1±0.2 (6)	1.0±0.2 (6)	0.79±0.13 (5)	0.97±0.11 (5)	0.91±0.08 (4)	1.2±0.2 (5)	0.53±0.03 (3)	19±2 (5)
Tb	1.0±0.2 (6)	1.02±0.05 (4)	0.82±0.08 (4)	0.74 (2)	0.75±0.04 (4)	1.26±0.08 (3)	0.37±0.04 (4)	1.7±0.3 (4)
Te (ppb)	6.3 (1)	< 320,000	< 5	< 5	< 320,000	5.5 (1)	< 320,000	< 5
Th	1.1±0.2 (7)	12.8±0.5 (5)	4.6±1.1 (6)	16±2 (5)	10.1±0.7 (5)	12.0±0.4 (4)	4.90±0.02 (4)	33±5 (4)
Ti	16000±500 (7)	4300±200 (4)	3700±170 (6)	1600±200 (6)	4000±700 (7)	6000±250 (6)	1700±300 (5)	910±60 (4)
Tl (ppb)	49 (1)	790 (1)	230 (1)	1070 (1)	790 (1)	800 (1)	340 (1)	300 (1)
Tm (ppb)	310±40 (4)	440 (2)	390 (2)	370 (2)	350 (2)	720 (2)	180 (2)	< 1000
U	0.42±0.06 (7)	2.85±0.02 (4)	1.97±0.03 (4)	5.84±0.07 (4)	3.09±0.14 (4)	3.02±0.10 (4)	5.57±0.15 (4)	9.07±0.07 (4)
V	314±12 (5)	142±3 (5)	60±5 (4)	14±1 (4)	122±16 (8)	110±30 (5)	128±8 (6)	2.0 (1)
W	0.28 (2)	1.67 (1)	0.61±0.03 (3)	1.6±0.1 (4)	1.61 (1)	0.84 (2)	2.64 (1)	3.9±0.2 (4)
Y	28±2 (4)	25-57 (2)	28 (2)	27 (2)	26 (2)	44-70 (2)	12.7 (1)	53 (2)
Yb	2.1±0.5 (7)	3.0±0.3 (5)	2.8±0.7 (5)	2.9±0.2 (4)	2.6±0.3 (6)	5±1 (5)	1.1±0.2 (3)	4.4±0.4 (4)
Zn	102±7 (8)	126±18 (10)	61±4 (7)	32±7 (10)	106±9 (9)	102±8 (8)	80±9 (8)	230±30 (12)
Zr	180±30 (5)	130±10 (4)	190±20 (4)	210±10 (3)	160±24 (6)	270±30 (3)	56±5 (4)	1260±80 (3)

Table 3. Major and minor element oxide concentrations in eight new USGS standard rocks (%)

Element	BHVO-1	MAG-1	QLO-1	RGM-1	SCO-1	SDC-1	SGR-1	STM-1
SiO_2	49.9±0.8 (7)	50.0±1.1 (5)	65.5±1.2 (6)	72.7±1.3 (5)	62.6±0.8 (4)	65.3±1.4 (5)	28.1±0.3 (3)	59.6±0.4
Al_2O_3	13.8±0.2 (8)	16.4±0.3 (6)	16.2±0.2 (6)	13.8±0.2 (6)	13.6±0.4 (7)	16.1±0.4 (6)	6.68±0.43 (5)	18.5±0.1
Fe_2O_3	2.65±0.13 (3)	2.6-4.2 (2)	1.00 (2)	0.49 (2)	4.0 (2)	2.6 (2)	1.52 (1)	2.80
FeO	8.59±0.08 (3)	2.5-3.6 (2)	2.96 (2)	1.23 (2)	0.99 (1)	3.9 (2)	1.25 (1)	2.80
MnO	0.166±0.005 (8)	0.11±0.02 (6)	0.092±0.008 (6)	0.038±0.002 (8)	0.053±0.004 (8)	0.116±0.008 (7)	0.037±0.004 (7)	0.228±0.0
MgO	7.14±0.20 (8)	3.08±0.21 (5)	1.01±0.06 (5)	0.28±0.02 (7)	2.67±0.23 (5)	1.68±0.09 (6)	4.6±0.4 (4)	0.092±0.0
CaO	11.4±0.2 (7)	1.45±0.28 (5)	3.18±0.08 (5)	1.13±0.09 (7)	2.61±0.19 (6)	1.43±0.04 (6)	8.57±0.32 (3)	1.10±0.0
Na_2O	2.21±0.09 (8)	3.73±0.25 (6)	4.17±0.14 (7)	4.03±0.14 (10)	0.87±0.07 (6)	2.00±0.07 (8)	2.81±0.19 (4)	8.91±0.1
K_2O	0.55±0.08 (12)	3.6±0.2 (6)	3.59±0.11 (8)	4.33±0.10 (9)	2.75±0.10 (6)	3.27±0.08 (8)	1.72±0.16 (5)	4.30±0.0
TiO_2	2.65±0.09 (7)	0.72±0.04 (4)	0.62±0.03 (6)	0.27±0.03 (6)	0.67±0.11 (7)	1.00±0.04 (6)	0.28±0.05 (5)	0.15±0.0
P_2O_5	0.29±0.04 (6)	0.17±0.02 (3)	0.26±0.02 (4)	0.049±0.001 (4)	0.23±0.04 (4)	0.18±0.04 (6)	0.30±0.03 (3)	0.163±0.0
H_2O+	0.23 (2)	5.2-6.9 (2)	0.26-0.52 (2)	0.34±0.71 (2)	3.6-4.4 (2)	1.40 (2)	-	1.4
H_2O^-	0.05 (2)	2.4 (2)	0.15 (2)	0.10 (2)	1.7-2.4 (2)	0.05-0.16 (2)	0.45 (1)	1.8
H_2O-T	0.26 (1)	8.55 (1)	0.82 (1)	1.01 (1)	5.64 (1)	1.59 (1)	-	1.7
CO_2	0.04±0.02 (4)	8.6±0.4 (3)	0.010±0.001 (3)	0.01±0.01 (5)	3.1±0.6 (4)	0.05-0.14 (3)	10.5 (2)	0.08
Fe_2O_3-T	12.2±0.3 (10)	7.0±0.4 (7)	4.32±0.13 (8)	1.87±0.05 (10)	5.14±0.13 (6)	7.03±0.17 (8)	2.97±0.19 (7)	5.21

NBS 278 Obsidian Rock

21 Aug 95 PB

Sample ID	Analysis	Sample Result
20F	Si as SiO_2	74.6%
	Al as Al_2O_3	13.0%
	Fe as Fe_2O_3	1.96%
	Mg as MgO	0.223%
	Ca as CaO	0.900%
	Na as Na_2O	5.23%
	K as K_2O	4.48%
	Ti as TiO_2	0.226%
	Ba as BaO	0.106%
	LOI (Loss @ 1000°C)	0.691%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	6	4
Lead	15	10
Lithium	48	4
Manganese	403	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	115	80
Selenium	<10	10
Silver	<10	10
Strontium	65	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	59	20

data from NBS 278 certificate

Constituent[1]	Content wt %	Constituent[1]	Content wt (µg/g)
C (Total Carbon)[j]	(0.05)	Ba [f]	(1140)
CO_2 [d]	(0.01)	B [g]	(25)
F [h]	(0.05)	Ce [f]	(62.2)
MgO [d]	(0.23) (0.13%)	Co [f]	(1.5)
		Cr [f]	(6.1)
		Cs [f]	(5.5)
		Eu [f]	(0.84)
		Gd [g]	(5.3)
		Hf [f]	(8.4)
		Lu [f]	(0.73)
		Sb [f]	(1.5)
		Sc [f]	(5.1)
		Sm [f,g]	(5.7)
		Ta [f]	(1.2)
		Tb [f]	(1.0)
		Yb [f]	(4.5)
		Zn [f]	(55)

Table 2. Information Values

Table 1. Certified Values of Constituents

Constituent[1]	Content[2] (wt %)	Constituent[1]	Content[2] wt (µg/g)
Al_2O_3 [d]	14.15 ± 0.15	Cu [e]	5.9 ± 0.2
CaO [e]	0.983 ± 0.002	Ni [e]	3.6 ± 0.3
FeO [i]	1.36 ± 0.02	Rb [a,e]	127.5 ± 0.3
Fe_2O_3 [i,f] (Total Fe as Fe_2O_3)	2.04 ± 0.02	Sr [e]	63.5 ± 0.1
K_2O [a,d,e]	4.16 ± 0.02	Th [e,f]	12.4 ± 0.3
MnO [b,f]	0.052 ± 0.002	Tl [e]	0.54 ± 0.04
Na_2O [a,d,f]	4.84 ± 0.05	U [e]	4.58 ± 0.04
P_2O_5 [b,d]	0.036 ± 0.003	Pb [e]	16.4 ± 0.2
SiO_2 [d]	73.05 ± 0.13		
TiO_2 [b,g]	0.245 ± 0.007		

[1] Methods of Analysis:
[a] Atomic Absorption
[b] Colorimetry
[c] Emission Spectrometry
[d] Gravimetry
[e] Isotope Dilution Mass Spectrometry
[f] Neutron Activation Analysis
[g] Prompt-gamma Activation Analysis
[h] Specific Ion Electrode Potentiometry
[i] Titrimetry
[j] Volumetry

[2] The estimated uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods and material variability of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

Na - SAz-1

29 Aug 95 PB

Sample ID	Analysis	Sample Result
20H	Si as SiO_2	59.6%
	Al as Al_2O_3	16.0%
	Fe as Fe_2O_3	1.41%
	Mg as MgO	5.35%
	Ca as CaO	0.803%
	Na as Na_2O	2.83%
	K as K_2O	0.144%
	Ti as TiO_2	0.195%
	Mn as MnO	0.180%
	LOI (Loss @ 1000°C)	14.9%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	266	4
Beryllium	4	2
Boron	<20	20
Cadmium	<4	4
Chromium	25	4
Cobalt	<10	10
Copper	<4	4
Lead	41	10
Lithium	256	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Strontium	112	10
Thallium	<10	10
Tin	<20	20
Vanadium	62	20
Zinc	75	20

low Pb 8/28/95

SiO_2 seems high, even when corrected for differences in water (LoZ) content.

Given on data sheet (p. 149) LOI = 9.4%
this analysis LOI = 14.9%
5.0% difference

⇒ SiO_2 (0.95) = data sheet 8/28/95 PB

but, 56.6 ≠ 60.4

60.4 (0.95) = 57.4 = 59.6 ⇒ fairly close considering other numbers.

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET FOR MAJOR METALS

chefo SAz-1

Lab Name: Southwest Research Institute Client: Division 20

Lab Code: SWRI Date Received: 07/13/95

Lab System ID: 56770 Project No.: 20-5704-152

29 Aug 95 PB

Sample ID	Analysis	Sample Result
20I	Si as SiO_2	46.7%
	Al as Al_2O_3	12.5%
	Fe as Fe_2O_3	1.11%
	Mg as MgO	4.43%
	Ca as CaO	1.99%
	K as K_2O	0.153%
	Ti as TiO_2	0.161%
	LOI (Loss @ 1000°C)	25.5%
	TOTAL	92.6%

Chefo SAz-1
Ca form
raw

ARCH INSTITUTE
DATA SHEET
METALS

Sample ID 20I

Lab Name: Southwest Research Institute Client: Division 20

Lab Code: SWRI Date Received: 07/13/95

Matrix: Solid Date Analyzed: 08/07/95

Lab System ID: 56770 Project No.: 20-5704-152

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	299	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	<4	4
Lead	26	10
Lithium	212	4
Manganese	562	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Sodium	605	25
Strontium	286	10
Thallium	<10	10
Tin	<20	20
Vanadium	57	20
Zinc	53	20

Chefo SAz-1, Ca form, raw

Sample ID	Analysis	Sample Result
20K	Si as SiO_2	58.1%
	Al as Al_2O_3	15.3%
	Fe as Fe_2O_3	1.37%
	Mg as MgO	5.46%
	Ca as CaO	2.49%
	K as K_2O	0.191%
	Ti as TiO_2	0.199%
	LOI (Loss @ 1000°C)	25.5%
	TOTAL	109%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	315	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	<4	4
Lead	21	10
Lithium	221	4
Manganese	594	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Sodium	629	25
Strontium	299	10
Thallium	<10	10
Tin	<20	20
Vanadium	61	20
Zinc	59	20

data for SAz-1 from Van Olphen & Fripiat, 1979.

Data Summaries CMS 25

SAz-1 MONTMORILLONITE ARIZONA (CHETO)

ORIGIN Bidahochi formation (pliocene)
Apache County, State of Arizona.
Location: SE 1/4 NE 1/4 Sec. 26 T 21 N? R 29 E; topographic map: Gallup (1 : 250,000). Collected from pit after overburden was stripped, 8 May, 1973
CHEMICAL COMPOSITION (%)
SiO_2: 60.4 Al_2O_3: 17.6 TiO_2: 0.24 Fe_2O_3: 1.42 FeO: 0.08 MnO: 0.099 MgO: 6.46 CaO: 2.82 Na_2O: 0.063 K_2O: 0.19 P_2O_5: 0.020 F: 0.287 Loss on heating -550°C: 7.54; 550-1000°C: 2.37
CATION EXCHANGE CAPACITY
120 meq/100g, major exchange cation Ca.
SURFACE AREA
N_2 area: 97.42 ± 0.58 m^2/g
THERMAL ANALYSIS
DTA: endotherms at 200°C, shoulder at 240°C, desorption of water; 685°C, dehydroxylation; shoulder at 895°C; exotherms at 1020°C, 1065°C, 1160°C
TG: Loss in dehydroxylation range: 4.69% (theory: 5.0%)
INFRARED SPECTROSCOPY
The spectrum indicates a low octahedral iron content. A silica phase (band at 790 cm^{-1}) is detectable.

large difference in SiO_2 reported for these two samples (11.4%!)

19 Aug 95 PB

Stz-1, Na form, freeze dried

Sample ID	Analysis	Sample Result
20J	Si as SiO_2	53.5%
	Al as Al_2O_3	14.3%
	Fe as Fe_2O_3	1.27%
	Mg as MgO	4.84%
	Na as Na_2O	3.44%
	Ti as TiO_2	0.185%
	LOI (Loss @ 1000°C)	23.4%
	TOTAL	101%

Sample ID	Analysis	Duplicate Result
20J	Si as SiO_2	55.0%
	Al as Al_2O_3	14.8%
	Fe as Fe_2O_3	1.31%
	Mg as MgO	5.00%
	Na as Na_2O	3.61%
	Ti as TiO_2	0.190%
	LOI (Loss @ 1000°C)	23.4%
	TOTAL	103%

Analysis	Sample Result (ug/g)	Duplicate Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	<10	10
Arsenic	<25	<25	25
Barium	53	53	4
Beryllium	4	3	2
Boron	<20	<20	20
Cadmium	<4	<4	4
Calcium	253	251	20
Chromium	8	12	4
Cobalt	<10	<10	10
Copper	<4	<4	4
Lead	26	29	10
Lithium	226	229	4
Manganese	263	263	4
Molybdenum	<4	<4	4
Nickel	<12	<12	12
Phosphorus	<80	<80	80
Potassium	530	550	40
Selenium	<10	<10	10
Silver	<10	<10	10
Strontium	<10	<10	10
Thallium	<10	<10	10
Tin	<20	<20	20
Vanadium	<20	<20	20
Zinc	63	61	20

Sample ID	Analysis	Sample Result
20L	Si as SiO_2	61.4%
	Al as Al_2O_3	16.7%
	Fe as Fe_2O_3	1.47%
	Mg as MgO	5.63%
	Na as Na_2O	4.07%
	Ti as TiO_2	0.213%
	LOI (Loss @ 1000°C)	24.4%
	TOTAL	114%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	52	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Calcium	261	20
Chromium	8	4
Cobalt	<10	10
Copper	<4	4
Lead	20	10
Lithium	230	4
Manganese	258	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Potassium	560	40
Selenium	<10	10
Silver	<10	10
Strontium	<10	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	58	20

21 Aug 95 PB

Sco-1 Cody shale

Sample ID	Analysis	Sample Result
20M	Si as SiO_2	68.6%
	Al as Al_2O_3	14.4%
	Fe as Fe_2O_3	5.47%
	Mg as MgO	2.81%
	Ca as CaO	2.64%
	Na as Na_2O	1.12%
	K as K_2O	3.29%
	Ti as TiO_2	0.578%
	P as P_2O_5	0.238%
	LOI (Loss @ 1000°C)	10.0%
	TOTAL	109%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	613	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	74	4
Cobalt	16	10
Copper	29	4
Lead	28	10
Lithium	48	4
Manganese	419	4
Molybdenum	<4	4
Nickel	29	12
Selenium	<10	10
Silver	<10	10
Strontium	174	10
Thallium	<10	10
Tin	<20	20
Vanadium	142	20
Zinc	119	20

Sample ID	Analysis	Sample Result
20N	Si as SiO_2	74.7%
	Al as Al_2O_3	14.8%
	Fe as Fe_2O_3	5.62%
	Mg as MgO	2.93%
	Ca as CaO	2.74%
	Na as Na_2O	1.19%
	K as K_2O	3.43%
	Ti as TiO_2	0.613%
	P as P_2O_5	0.271%
	LOI (Loss @ 1000°C)	10.0%
	TOTAL	116%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	582	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	66	4
Cobalt	19	10
Copper	28	4
Lead	23	10
Lithium	46	4
Manganese	388	4
Molybdenum	<4	4
Nickel	28	12
Selenium	<10	10
Silver	<10	10
Strontium	165	10
Thallium	<10	10
Tin	<20	20
Vanadium	130	20
Zinc	108	20

reported values correlate well (especially for minor constituents) PB 22 Aug 95

except for large error in SiO_2 content (high values). Al_2O_3 is also high. Sample splits do not correlate well w/ each other (SiO_2) note large mass balance totals.

30 Aug 95
PD

To aid in the evaluation of the chemical data reported by DIV 01 for the SAz-1 samples, an analysis of the loss of H_2O / combustibles upon heating was performed.

Samples were transferred into weighed, self-sealing quartz crucibles and heated as shown below.

crucible:	1	2	3	4	5
content:	SAz + Na + PD	SAz + Na + PD	SAz + Na + PD	Na-SAz-1	SAz + Na + PD
wt. crucible empty	11.0997	11.5311	12.7120	12.1995	12.6465
crucible + clay	12.1214	12.1546	14.3759	12.5448	13.4460
after heating to 100°C for 18 hours	N/A	N/A	14.0306	N/A	13.2787
after heating to 920 ± 20°C for 2 hours	11.8625	11.9967	13.9544	12.4628	13.2435
mass clay	1.0217	0.6235	1.6639	0.3453	0.7995
mass clay after 100°C	N/A	N/A	1.3186	N/A	0.6322
after 940°C	0.7628	0.4656	1.2424	0.2633	0.5970
adsorbed H_2O	N/A	N/A	0.3416	N/A	0.1667 93 PD
loss on ignit	0.2589	0.1579	0.4215	0.0820	0.2025

Note: SAz + Na + PD = SAz + Na + PD

30 Aug 95
PD

	1	2	3	4	5
% initial wt of adsorbed water	N/A	N/A	20.75	N/A	20.93
% wt LOI	25.34	25.32	25.33	23.75	25.33
% wt LOI based on wt clay after 100°C heating	N/A	N/A	5.78	N/A	5.57

Comparison of % LOI for Samples 1, 2 and 4 are very similar to the values reported by DIV 01. Samples 3 & 5, heated in 2 steps, confirm these values.

6 Sep 95
PD

Because of the poor results for SiO_2 in most of the samples run by DIV 01, I submitted several for repeat analysis. Samples submitted will be reanalyzed, provided with QA/QC reports and reported in a fashion similar to the previous samples.

SAMPLE NO	CONTENT
20A	SCo-1
20B	SAz-1
20C	SCo-1
20D	SAz + Na
20E	SAz-1
20F	NBS-99a (Na. feldspar)
20G	SAz + Na
20H	NBS-99a

1/8/97

This project
was terminated
due to lack
of funding.

R. Wabakken

Courtesy of the
U.S. Congress.
E.C.
1/8/97