Geochemistry
Research Lab
Notebook
VOL. GC-02

BOBBY PABALAN
VOLUME GC-02

CNWRA
CONTROLLED
COPY 008

Geochemistry Research Lab. Notebook

VOL. GC-02

Please date and initial any entry on this lab book.

Volume 2 of Geochemistry Research Project

4/6/90 RJP

1300 hrs - Reweigh sample CDVP.
Total wt = 75.77 g.
Replace sample in dessicator.

4/9/90 RJP
1330 hrs - Reweigh sample CDVP.
Total wt = 75.77 g.

It appears that sample CDVP has equilibrated with water vapor over a saturated NaCl soln. No further change in sample weight is observed.

1400 hrs - Prepare clinoptilolite samples to be sent to Utah Research Inst. for XRD analysis.

CDVP - take about 2.46 g. from plastic container, transfer to smaller plastic vial, label CDVP 100-200 mesh. Weight of leftover CDVP is 73.31 g (sample + container)

CDVHS-1 - take about 2.7[illegible] transfer to plastic vial and label CDVHS-1 100-200 mesh. This sample is the first heavy mineral separates from sample CDVP.

CSC(ZHL) – transfer sample CSC(ZHL) from glass vial to plastic vial, label CSC(ZHL) L 325 mesh. This sample was analyzed using XRD on 12/18/89.

1430 hrs – Scrape off mordenite in vugs of basaltic(?) sample, grind dry using mortar and pestle, and place in plastic vial. Label MFD (mordenite, Fossil, Lion County, Oregon). This sample was purchased from Mineralogical Research Co.

Submit to H Saldana (Div. 6) for XRD analyses.

1600 hrs – Requisition for XRD analysis of CDVP, CDVHS-1, and CSC(ZHL) typed up by Yolanda L. QA review done by B Mabanto and Bob Bryant.

4/10/90 RHD

0900 hrs – Samples CDVP, CDVHS-1, and CSC(ZHL) sent out to UARI for XRD analyses.

1200 hrs – Prepare powdered sample

of clinoptilolite and analcime for reanalysis by H Saldana on XRD machine. Use previously ground samples kept in storage cabinet. Samples must be finer than 325 mesh grain size. Samples CH, CB, CGC, CDY, CT, CCC, ASH.
4/12/90 RHD

1100 hrs – Several chips of sample CCC (clinoptilolite, Castle Creek, Idaho) sent to SW Kochler Co. for thin-section preparation. T-S to be forwarded to Slutz of UURI for petrog. analysis.

4/12/90 RHD

1300 hrs –

Test of clinoptilolite dissolution by HNO_3 soln.

Method: Mix 50% HNO_3 solution from conc. HNO_3 and deionized H_2O.

Place about 0.5 g of sample CDV (L 450 mesh) in flask. Add about 50 ml HNO_3 soln; add stir bar and place on magnetic stir plate. Stir at low speed. Soln (A).

Place another 0.5 g in another flask, add 50 ml HNO_3 soln and stir bar. Place over heater/magnetic stir plate. Heat low, stir

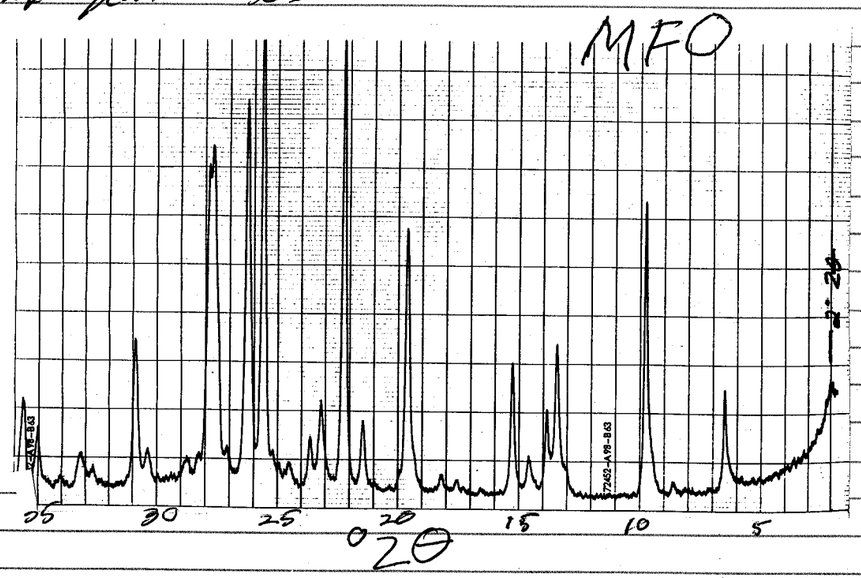
at low speed. Soln. (B).

4/13/90 RDN

1500 hrs – Both solutions (A) and (B) from 4/12/90 appear milky, probably due to fine particles in suspension. A ring of precipitates formed on both flasks above the level of the stirred solutions.

1530 hrs – The following table lists the 2θ, d-spacing and relative intensities for the XRD peaks of mordenite from Foster, Oregon (Sample MFO). The XRD strip chart is kept in a separate file. A copy is given below:

4/11/90
40 ma
40 kV
400 cps
τ = 4 sec
0.5° 2θ/min



SAMPLE MFO - MORDENITE, FORSTER, LINN COUNTY, OREGON

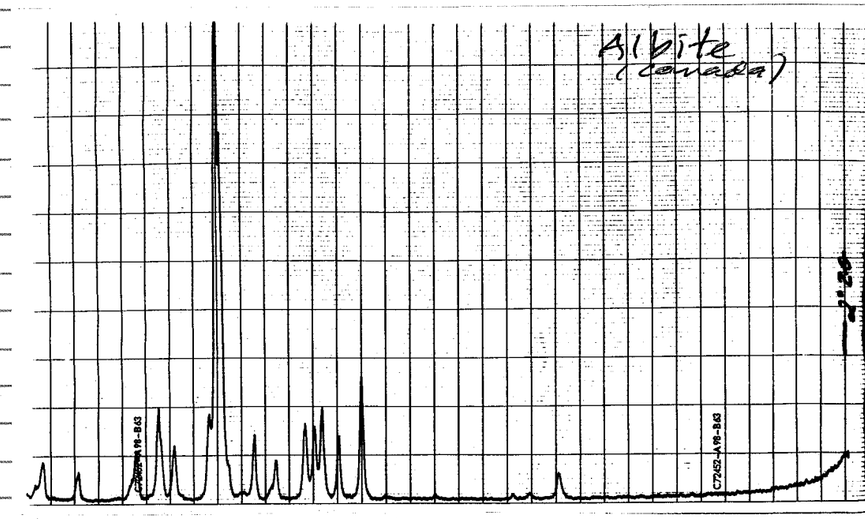
2THETA	D-SPACING	I/IO	hkl 29-1257	hkl 6-239 & JCPDS Files
6.51	13.566	22.7	110	110
8.65	10.214	5.5	020	
9.78	9.036	57.8	200	200
13.02	6.794	8.2		← shoulder on 13.43 peak
13.43	6.587	31.0	111	111
13.85	6.388	18.9	130	130
14.6	6.062	10.2	021	021
15.28	5.794	27.5	310	310
16.6	5.336	4.1		
17.55	5.049	5.5		221
18.2	4.870	6.4	131	131
19.3	4.595	9.1	311	
19.65	4.514	52.4	400	330
21.45	4.139	16.4	420	420
22.24	3.994	90.0	150	150
23.18	3.834	20.0	241	241
23.64	3.760	13.4	002	002
24.5	3.630	8.6	421	112
24.95	3.566	8.5	510	510
25.2	3.531	10.9	022	
25.62	3.474	100.0	202	202
26.28	3.388	76.4	350	350
27.1	3.288	11.6	222	222
27.68	3.220	67.6	511	511
27.8	3.206	64.2	530	
28.25	3.156	10.2	312	
28.75	3.103	9.1	441	441
30.38	2.940	10.9	261	531
30.9	2.891	31.4	402	332
32.7	2.736	7.7	152	152
33.2	2.696	10.0	171	171
34	2.635	5.5	370	370
35	2.561	14.7	461	461
35.65	2.516	20.0	442	442
36.5	2.460	8.6	641	641
36.8	2.440	8.8	023	741
39.2	2.296	5.6	172	622
39.55	2.277	5.8	570	570
40.48	2.226	7.3	480	480
41.84	2.157	7.3	181	191
42.72	2.115	5.5	082	712
44.28	2.044	12.2	513	391
44.45	2.036	8.7	732	
44.95	2.015	9.1	443	353
45.45	1.994	7.3	841	841
46.55	1.949	11.8	680	680
47	1.932	6.4	911	182
RDN 4/13/90 ~~47.64~~ 47.35	~~1.909~~ 1.918	6.4	482	482
48.4	1.879	12.2	004	004
50.35	1.811	11.8	1680	1101
50.85	1.794	9.1	713	1111

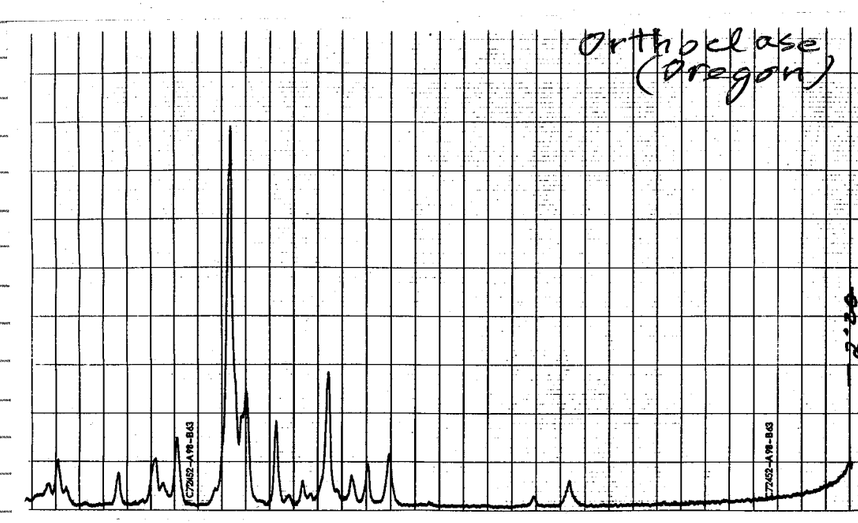
4/18/90 RJD
0900 – Solutions (A) and (B) from 4/12/90 have ~~some~~ HF still not dissolved the clinoptilolite sample.

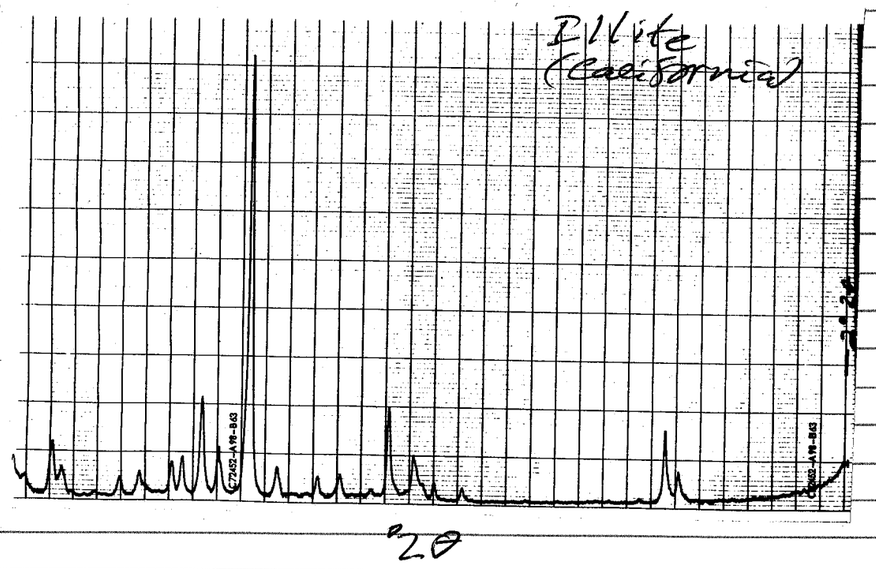
4/18/90 RJD
1000 hrs – Prepare powder samples (2325 mesh) of single mineral samples of albite, orthoclase, heulandite, and illite. These samples were purchased from D.J. Minerals (P.O. Box 761, Butte Montana) with the following stated localities respectively, Canada, Oregon, California, and Oregon. Also prepare powder sample from heulandite specimen purchased from Mineralogical Research Co. (15840 East 4th Way, San Jose, CA 95127-1737), with a locality stated to be Poonah, India. Label this last sample HRI.

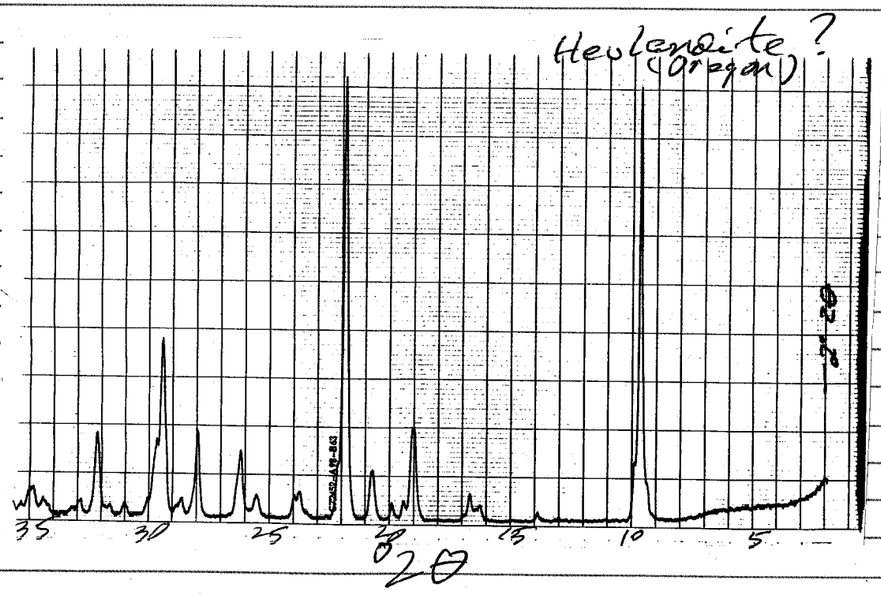
The following are copies of the XRD pattern run on Div 06 Siemens D-500 diffractometer, using 40 ma, 40 KV, time constant = 4 sec., 0.5° 2θ/min scan rate.

Peak identifications labeled on the patterns (hkl indices) are based on JCPDS data files whose ~~#~~ numbers are given. Original XRD patterns are kept in a separate file.

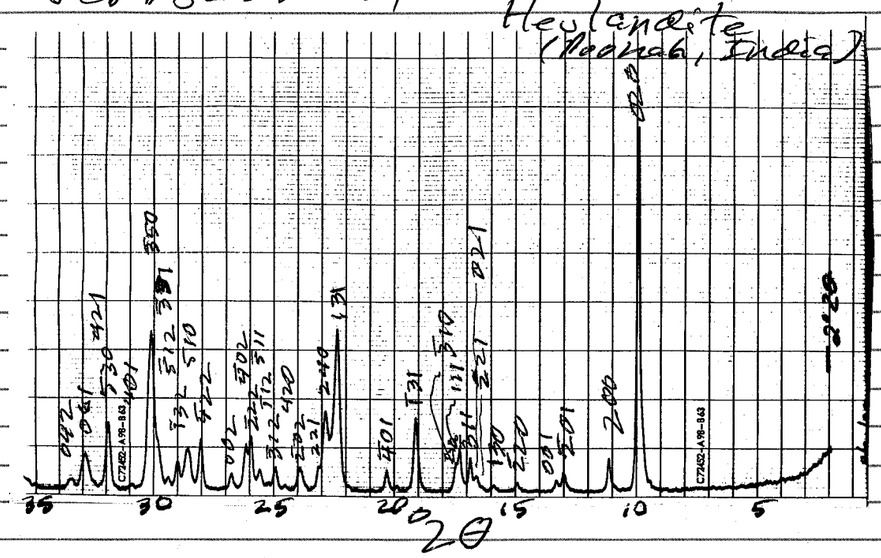








JCPDS-25-144



4/19/80 RDM

Verification of XRD and Petrographic Analysis of Clinoptilolite Specimens

Objective: To check XRD results and petrographic description of samples CH, CB, CGC, CPV, CT, and CCC. These samples have all been analyzed using semi-quantitative XRD by Susan Cutt at Utah Research Institute, and the first five samples have been analyzed petrographically by S. Cutz. The approximate percentages of identified minerals will be tabulated, and will be

based on comparisons with JCPDS powder data files, XRD patterns of standard minerals, as well as petrographic results.

Method:

Grind and sieve clinoptilolite sample to < 325 mesh, and run the XRD pattern according to TOP-004-02. Use JCPDS powder data files and XRD patterns of individual minerals determined previously to identify the mineralogy of the samples.

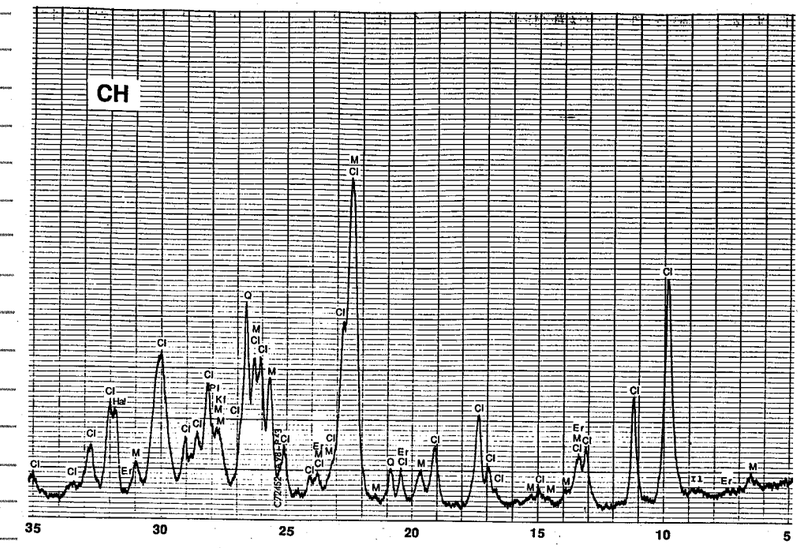
For petrographic analyses, use Bausch and ~~Lomb~~ Lomb petrographic microscope. The thin sections were prepared previously by Barbara Everett of the CNWRA and some were prepared (with cover slips) by the Steve Koeller Co. (P.O. Box 41, 306 Junction St., Grangeville, Idaho 83530).

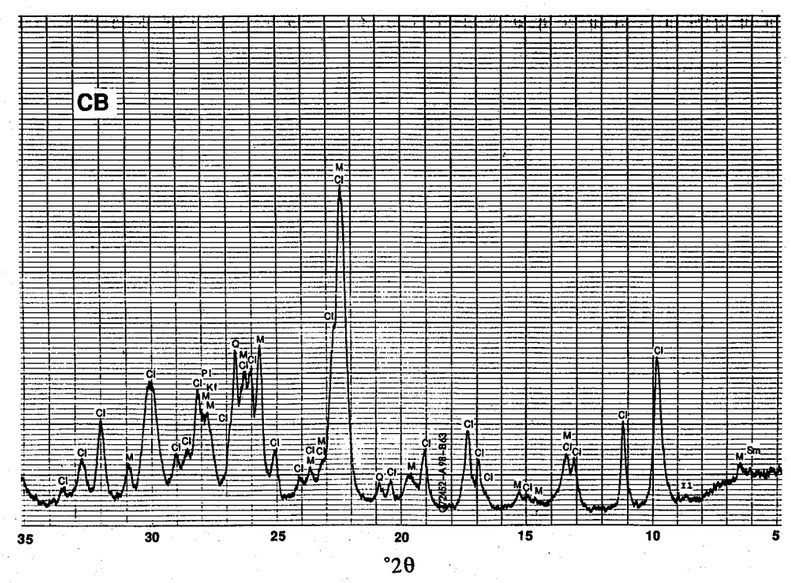
The XRD was run at 40 ma, 40 kV, 400 counts/sec., time constant = 4 secs., 0.5°2θ/min.

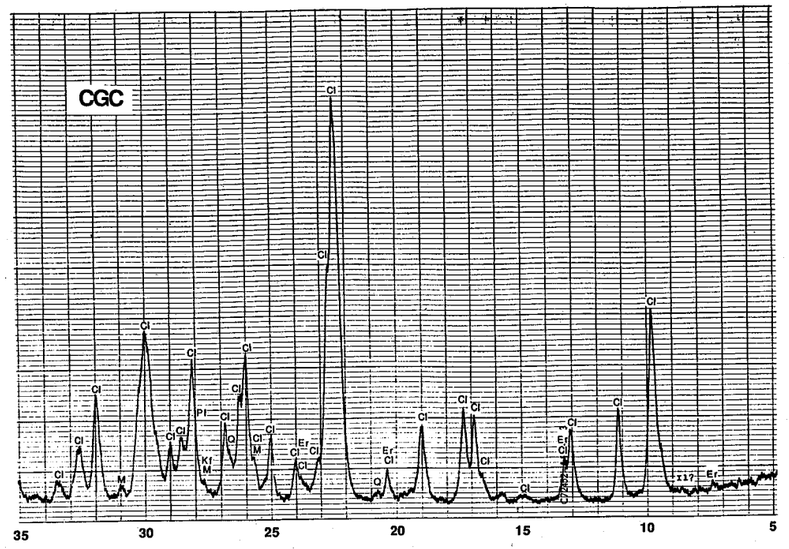
Results:

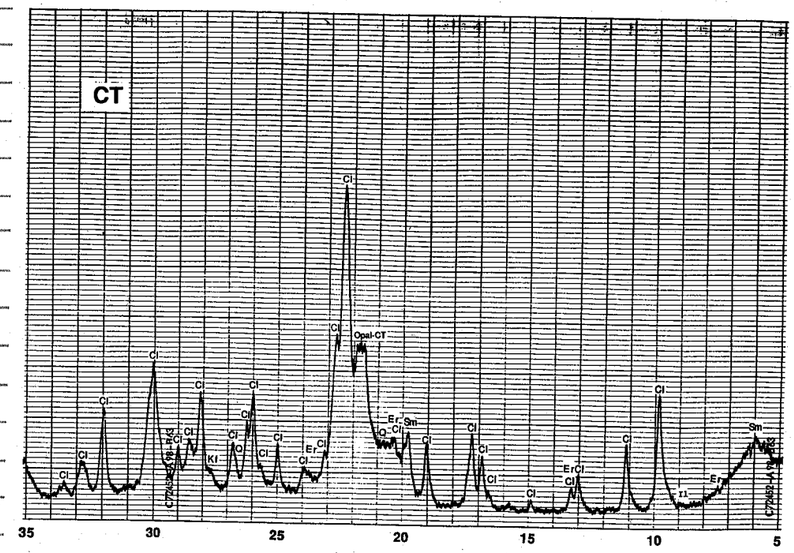
The following are copies of XRD patterns, with labeled peaks, of the clinoptilolite samples.

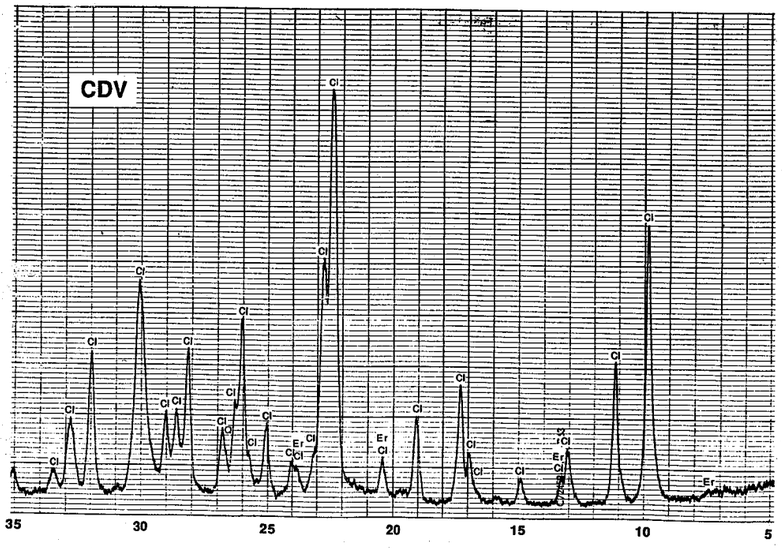
Cl = clinoptilolite ; Pl = plagioclase
Er = erionite ; Sm = smectite
M = mordenite ; Il = illite
Q = quartz ; Hal = halite
Kf = K-feldspar ; Dol = dolomite
Gyp = gypsum

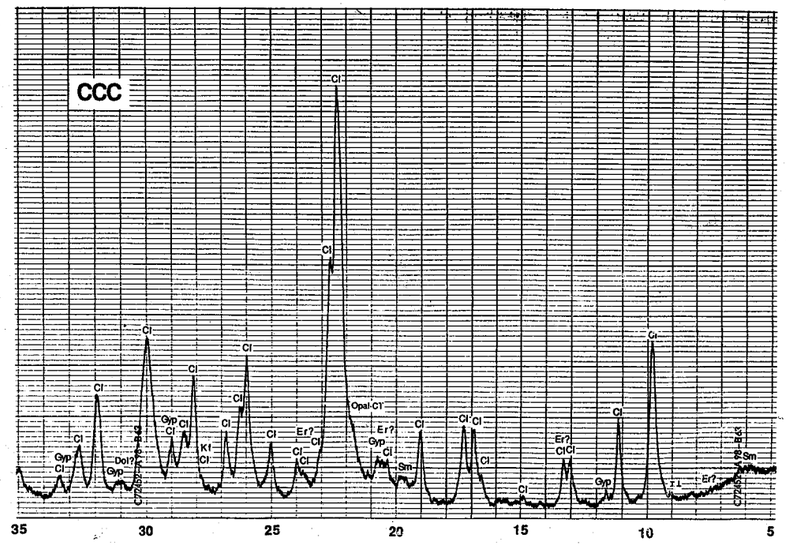





CGC
35
30
25
20
15
10
5
°2θ


CT
Opal-CT
35
30
25
20
15
10
5
°2θ


CDV
35
30
25
20
15
10
5
°2θ


CCC
Opal-CT
35
30
25
20
15
10
5
°2θ

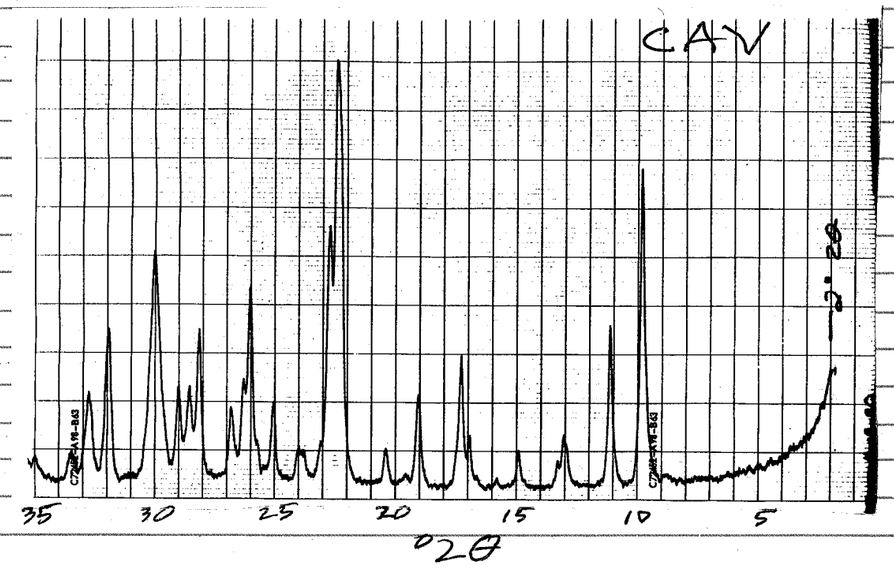
ZEOLITIZED TUFFS	MINERALOGY, APPROXIMATE WT. %																	
BULK XRD SAMPLE NO.	OPAL - CT	QUARTZ	PLAGIOCLASE	K - FELDSPAR	GEOTHITE	HEMATITE	AMPHIBOLE	GYPSUM	ANHYDRITE	ILLITE (±10Å MICA)	SMECTITE	CLINOPTILOLITE	MORDENITE	ERIONITE	HALITE	AMORPHOUS/ BELOW DETECTION		
CT	25	1	TR?	2						2	33	35		2		-		
CDV		.1	1	1	TR		TR			TR	2	86	TR?	1		6		
CCC	5	1	TR	2	TR		TR	5	1	2	16	65		2		-		Tr DOLOMITE?
CB		6	9	8	37	TR	TR			4		58	9	TR?		2		
CH		6	6	5			TR			4		61	11	2	4	-		
CGC		2	3	2		4				2		72	3	2		10		
																		From thin-sections:
																		Illite + Mica is biotite.
																		Amphibole is hornblende.

Tr = TRACE ? = TENTATIVE IDENTIFICATION

The above estimated mineral percentages for the clinoptilolite ~~percentages~~ for the samples are based on the XRD and petrographic analyses.

4/17/90, KPP
1600 hrs — Prepare powdered sample of zeolitized tuff, clinoptilolite rich sample from Amargosa Valley Nevada. Bulk samples were taken by E. Pearcy from rock pile along unnamed road and about 15 miles northwest of Nevada State Highway (52). The rock pile is believed to be generated by mining activities for clinoptilolite. See trip report by the Center's GIS group, currently being prepared by

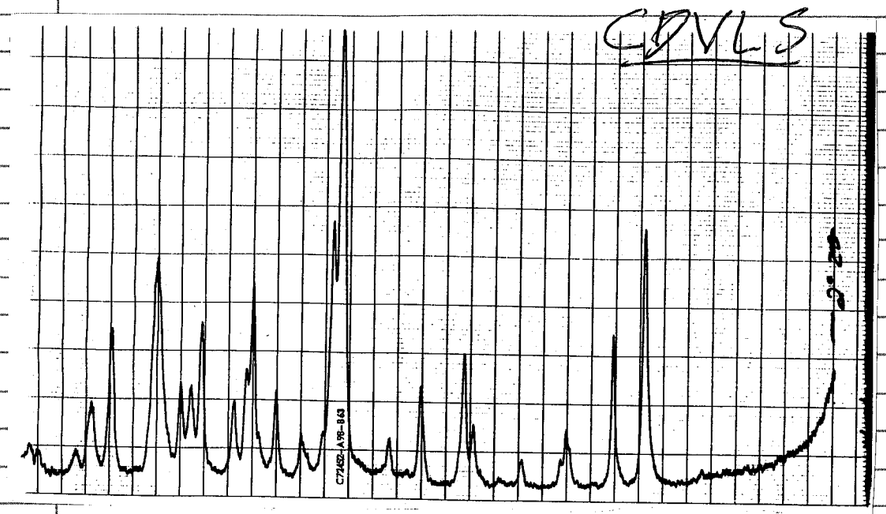
Steve Young. A copy of the XRD pattern derived from Div 86 machine is given below:



In addition, also submit powder samples of CDVLS ~~derived~~ and CDVHSW derived from activities on 2/13/90 (heavy liq. treatment at CDV (00-20 check samples) to H Saldana, Div 86, for XRD analyses.

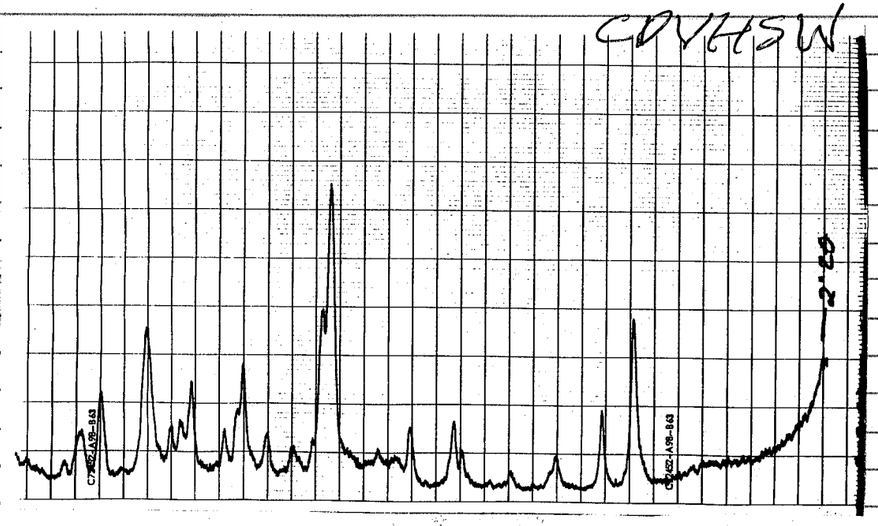
Copies of the XRD patterns for CDVHSW and CDVLS are given below, and original copies are kept in a separate file.

CDVLS



°2θ

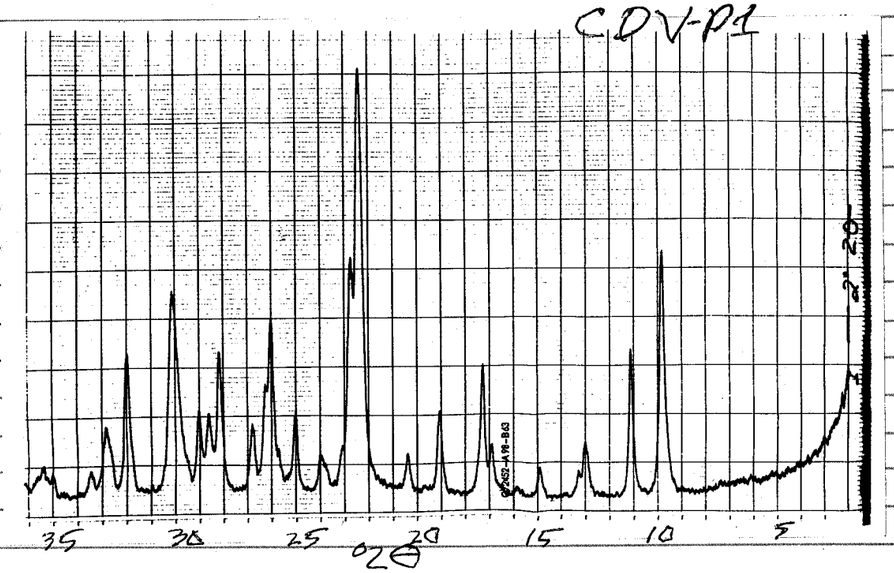
CDVHSW



°2θ

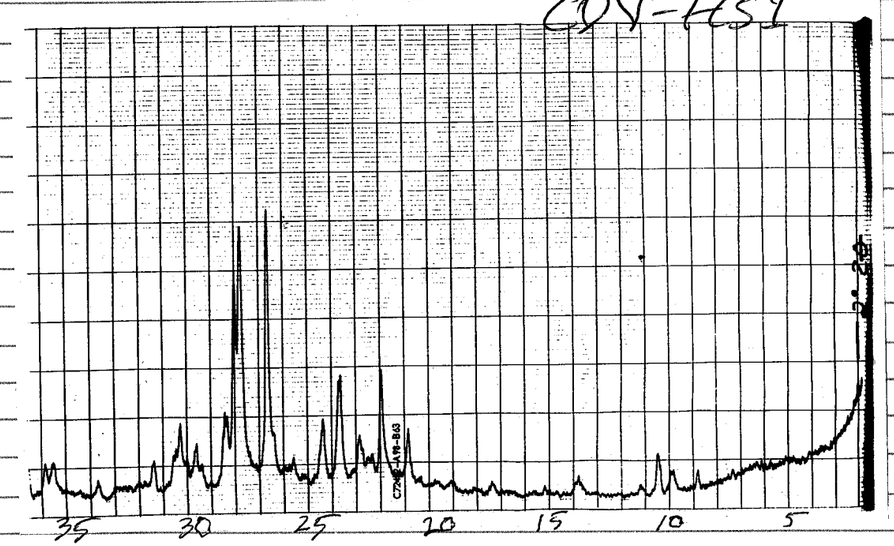
The following are XRD patterns of zeolites samples CDV-P1 and CDV-HS1 from 1/25/80 (See lab book GC-01, p. 141)

CDV-P1



°2θ

CDV-HS1



°2θ

5/2/90 KM

1030 hrs - Test of Fisher Isotemp Muffle Furnace Model 184A

Objective:
Generate plot of correlation between Temperature control position and pyrometer reading.

Method: As in Instruction Manual for Fisher Isotemp Muffle Furnace.

Results:

None / Approximately 1/2 of all muffle furnace units are not working and need replacement. Unit is being sent back to vendor.

1530 hrs - XRD analysis of Clinoptilolite and Heulandite samples heated at 450°C.

Objective: Determine XRD pattern of clinoptilolite samples CDV, CH, CCC, and also heulandite reference

sample HPI (see page GC-02-09). Heulandite and clinoptilolite are isostructural, but heulandite loses its structure upon heating to 450°C overnight. This study will confirm if the zeolitized tuff samples are indeed rich in clinoptilolite, and not heulandite.

Method:
Use the Fisher Isotemp Muffle Furnace model 184A in Div. 01. Preheat the furnace to 450°C, leave the samples in the furnace overnight. Cool the samples in a desiccator for about a day over a saturated NaCl soln., then submit for XRD analyses to Div. 06 (H. Saldana).

1600 hrs - Samples CDV, CH, CCC, CCC CB, CT and HPI were placed in the furnace set at about 450°C. These samples are the same ones analyzed previously (see pages GC-02-07 and GC-02-09 to -14). Samples are in 50 ml beakers.

5/3/90 KM

0915 hrs - Zeolite samples were taken out of the oven. After cooling to room temp., the samples were weighed (+ beaker + parafilm cover) using the Mettler balance in B57.

Samples:	Wt. sample + beaker + paraffin
CBI-HT	31.905 g
CDV-HT	32.234
CGC-HT	32.078
CTI-HT	30.683
CCC-HT	31.685
CHZ-HT	30.769
HPI-HT	31.234

The samples were put in a desiccator over a saturated NaCl solution. The containers were left open.

5/4/90 MW

0915 hrs – Samples from 5/3/90 were reweighed:

CBI-HT	32.013 g
CDV-HT	32.401
CGC-HT	32.169
CTI-HT	30.737
CCC-HT	31.721
CHZ-HT	30.929
HPI-HT	31.235

Samples CDV, CCC, CHZ and HPI-HT were transferred to plastic vials, and submitted to H Goldsmith (Vinson) for XRD analysis.

1300 hrs – Received results of XRD analysis of samples CDVP, CDVHS-1, and CSC (2HL) (see page GC-02-02), and petrographic

analysis of sample CCC (see page GC-02-03) from Utah Research Inst. Copies of the results are shown below:

Invoice

Payor's Name and Address	Payor's Reference
Southwest Research Institute P.O. Box 28510 6220 Culebra Road San Antonio, TX 78238-5166	P.O. No. 38984/37776 Reg. No. 673977 Total Amount $265.00
Payee's Name and Address UNIVERSITY OF UTAH RESEARCH INSTITUTE Research Park 391 Chipeta Way, Suite C Salt Lake City, Utah 84108-1295 801-524-3422	Payee's Reference * 5-84701-587 Invoice # 4 — Invoice Date 4-28-90 Billing Period April 1990

* Please include Payee's reference number with payment

Description		Amount
Semi-quantitative XRD; Bulk Samples	3	$240.00 P.O. No. 3777
Qualitative XRD; Bulk Samples	0	$0.00
Semi-quantitative XRD; Clay Mineralogy	0	$0.00
Qualitative XRD; Clay Mineralogy	0	$0.00
SUBTOTAL		$240.00
SPECIAL CHARGES		
1 General petrographic description (CCC)		$25.00 PO No. 38984
Samples of zeolitized tuff: CDVP CDVHS-1 CSC		
RUSH CHARGE 50%		$0.00
TOTAL		$265.00

MAY 04 1990
CNWRA

SOUTHWEST RESEARCH INSTITUTE – ROBERTO PARALAN

MINERALOGY, APPROX. WT.% ✓ (or) RELATIVE ABUNDANCE

ZEOLITIZED TUFFS BULK XRD SAMPLE NO.	QUARTZ	PLAGIOCLASE	K-FELDSPAR	MORDENITE	CRISTOBALITE	CALCITE	HALITE	SMECTITE	ILLITE (MICA)	CLINOPTILOLITE	MONOCLINIC	SCLIOMITE	CHABAZITE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
CDVP 100-200 mesh					2					98		TR?	TR?		–			
CDVHS-1 100-200 mesh	6	30	6	12	4	6	11?	2	3	20					–			
CSC (2HL) -325 mesh									TR	97	1	1?	1		–			

MM = PREDOMINANT M = MAJOR m = MINOR Tr = TRACE ? = TENTATIVE IDENTIFICATION

SUMMARY OF X-RAY DIFFRACTION ANALYSIS
UNIVERSITY OF UTAH RESEARCH INSTITUTE, EARTH SCIENCE LABORATORY

S. Lutz

PETROGRAPHIC ANALYSIS

SAMPLE NAME CCC

TYPE OF ROCK Quartz Latite or Dacite Tuff

MATRIX 96%
- Zeolitized glass shards
- Brownish-green smectite 3 to 15%
- Reddish-brown iron oxide <1%

GRAINS 4%
- Phenocrysts:
  - Quartz 1%
  - Feldspar (mostly sanidine) 2%
  - Biotite 2%, some altered
  - Hornblende Tr
  - Magnetite Tr-1%

TEXTURE

Poorly welded with well-preserved shard texture. Most of the shards are thin and long and average about 200 microns in length. These shards may be Pele's hair (although this term implies a more basaltic composition). Other shards are more y-shaped.

This sample is finely laminated; most of the rounded and abraded phenocrysts are in one slightly coarser-grained lamination. The grains of quartz, feldspar and biotite are smaller in size (100 to 200 microns) than the shards. The phenocrysts and the smectite are concentrated along the base of the graded lamination.

Grains of magnetite are very irregular in shape.

ALTERATION\MINERALIZATION

Zeolites have replaced nearly all the glass; only 1% of the more blocky (Y-shaped) shards are still glassy.

Most of the smectite is present in altered lithic fragments; from their elongate shape, these may have been biotites. Some of the more rounded patches of smectite may have filled pores (dissolved-out shards or the interior of bubble shards?) in the tuff.

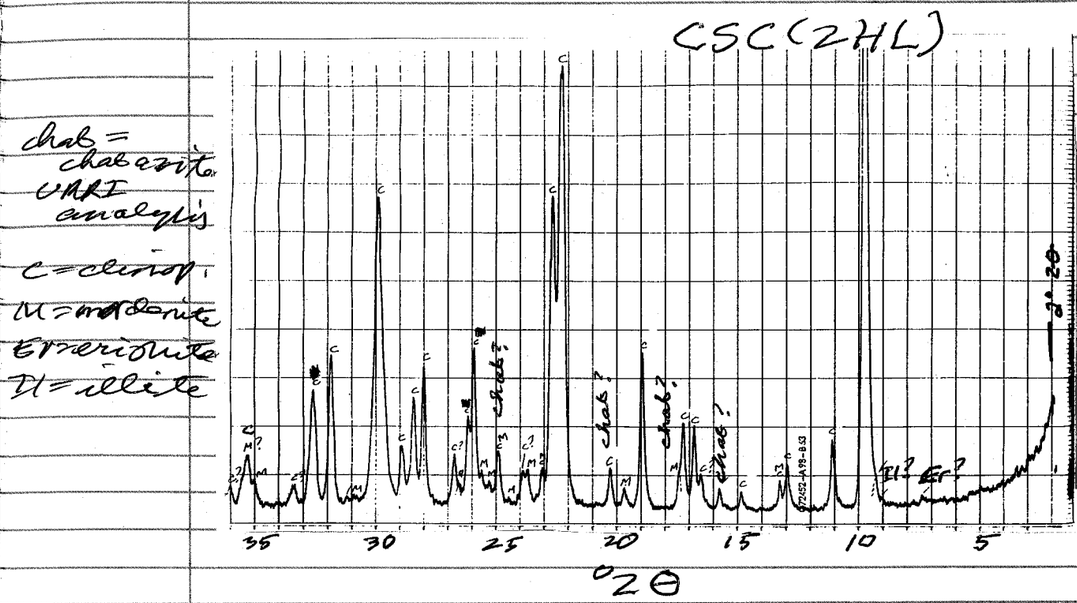
Nodules of iron-oxide (hematite) are located along the periphery of the thin-section. They may present surficial weathering (desert varnish).

One very fine fracture is present in the thin-section; it probably formed during thin-section preparation.

The gypsum and anhydrite indicated in the XRD analysis of this sample (12-5-89) are not evident in this thin-section. Perhaps these minerals represent irregularly distributed fracture-filling or weathering phases not present in the sample prepared for petrographic analysis.

DEPOSITIONAL ORIGIN Fallout tuff

The x-ray diffraction patterns of CDVP and CDVHS-1 determined by UNRI are compared with those by SwRI (Div. 06) (see page GC-02-17), and the XRD pattern on GC-02-17 are labeled after checking the mineral identification of UNRI with JCPDS data files or reference patterns of monomineralic samples. Also compare XRD pattern of sample CSC (2HL) with that derived previously (see page GC-01-126). A copy of the XRD pattern of sample CSC (2HL) determined on 12/18/89 is given below:



The chabazite identification labeled on the pattern above is based on UNRI identification w/c in turn is based on XRD pattern of their reference material. However, those labeled chab above do not seem to match very well the JCPDS data file # 34-137. SEM photomicrographs (see p. GC-01-120) also do not show chabazite crystals. It is possible that what has been labeled as chab. peaks may actually belong to clinoptilolite although not identified on JCPDS data files. Likewise with the labeled Er and Il peaks.

5/4/90 RW

The following is a copy of the chemical analytical results on NBS standards and zeolite samples performed by SwRI (Div. 01) personnel. Copies of tables of raw data (computer printouts) are kept in a separate file.

SOUTHWEST RESEARCH INSTITUTE
San Antonio, Texas 78228-0510

Page:

Project: 2C-3104-013

Potsdam

Sample id	251.6 Si	288.2 Si	Fe	Na	Ca	Al	Mg	K
CDYH2		30.29%	0.31%	3.99%	0.25%	5.80%	0.13%	1.48%
CDYP		30.89%	0.34%	2.49%	0.05%	5.90%	0.17%	3.11%
CDY		30.04%	0.46%	2.56%	0.35%	6.05%	0.20%	3.15%
CDYH81		28.39%	0.28%	~~4.0~~ 4.05%	<0.25%	11.13%	0.14%	1.66%
NBS 70A		27.81%	0.05%	1.78%	<0.25%	9.06%	0.034%	9.46%
NBS 99A		25.80%	0.04%	4.46%	1.15	9.79%	0.009%	4.32%
Reag. Blank		~~<0.02%~~ <0.12%	0.007%	<0.25%	<0.25%	0.03%	0.001%	<0.25%

Date: 5/4/90 Signature: Becky Ridder

see page 26-27 for Si & Al results RB 5/10/90

The following are summaries of the chemical analyses done on the NBS standards 70A and 99A performed to date by Jordan Labs, Core Labs, and SwRI (Div. 01). SwRI had tried different sample quantities and different amounts of $LiBO_2$ during the sample fusion step, due to problems with silica and aluminum losses caused by reprecipitation of silica. After consulting with Melanie Barnes of Tech Tech Univ., Dept. of Geology, a method of using 0.2g sample + 1.2g $LiBO_2$ was tried on 4/19/90 and appeared to work well. The acid matrix used in this method was 5% HCl solution instead of the usual 5% HNO_3 called for by ASTM procedure D4503-86. Also, the molten bead was quickly poured into a 250 ml beaker containing about 150 ml of HCl soln w/c is being stirred with a magnetic teflon stir bar. This appeared to facilitate dissolution of the bead. Before, the molten bead was allowed to cool down a little bit before being poured into the acid solution, and a magnetic stir bar was not used.

50 RB 5/4/90

The results for NBS 99A and NBS 70 determined on 4/19/90 were within 5% of the stated

weight %. Silicon analyses were done using two wavelengths of Si, namely: 251 and 288 nm.

The results for the NBS standards, done on 5/3/90, however, showed low recoveries for Si and Al. These samples will be reanalyzed for Si and Al to determine if this was caused by sample preparation or during the ICP analyses. Sample CDVH1 also appeared to be too high in Al and low in Si compared to the other CDV samples.

Samples CDV (untreated sample) and CDVP (treated by heavy liquid separation) appeared to be quite similar in composition except possibly for Ca for which CDV had higher concentration. The Ca content of CDVP will need to be reanalyzed by flame AA because it is below the detection limit of ICP.

The conversion of CDVP to a homoionic Na-form appeared to be somewhat successful. From an initial R w/o of about 3.1, this was reduced to about 0.5 for sample CDVH2. It is necessary to extend the treatment period to determine if this will cause further exchange.

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 99A
SAMPLE A

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	4.99	3.9	1.25	0.002	0.043	11.3	31.8
CORE LAB	4.8	5.7	1.75	<0.02	0.0403	12.0	32.4
SWRI	4.89	3.9	1.35	0.008	0.049	8.78	0.17
	5.0	4.0	1.4	<0.05	0.06	10.9	5.0
							3.5
(3/28/90)	3.92	3.8	1.33		0.045	6.67	19.5
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	21.9
(4/2/90, 0.05g sample, 0.5g LiBO2)						18.2	16.1
(4/11/90, .02g sample, 0.5g LiBO2)						10.5	27.0
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						11.0	30.3
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							29.5
(5/3/90)	4.46	4.32	1.15	0.009	0.04	9.79	25.2
9/5/90						9.19	25.24
NBS	4.6	4.315	1.53	0.012	0.0455	10.85	30.48

see page GC-02-41

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 70A
SAMPLE E

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	2.1	9.61	0.076	0.003	0.051	9.6	32.5
CORE LAB	2.24	4.3	0.093	0.018	0.0784	9.1	28.8
SWRI	1.74	7.67	<0.12	0.0097	0.0531	5.34	0.10
(3/28/90)	1.93	9.33	<0.25		0.059	7.87	13.99
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	20.7
(4/2/90, 0.05g sample, 0.5g LiBO2)						8.7	18.4
(4/11/90, 0.02g sample, 0.5g LiBO2)						6.1	26.1
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						9.7	30.7
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							32.3
(5/3/90)	1.78	9.46	<0.25	0.024	0.05	9.06	27.81
9/5/90						8.15	26.8
NBS	1.89	9.8	0.079	0.0	0.0525	9.474	31.37

see p. GC-02-41

5/7/80 DRT

0900 hrs – Reweigh samples CT, CB and CGC HT which are equilibrating with water vapor over a saturated NaCl soln. Refer to pages GC-02-19 and GC-02-20.

Samples	Wt sample + beaker + paraffin
CT-HT	30.747 g
CB-HT	32.016 g
CGC-HT	32.178

0930 hrs – Prepare zeolite samples and NBS standards for chemical analyses in Div. 01. Place about 1 g of powdered sample or standard in plastic vials. Label:

A – NBS 99A; same material used for Smith chemical analysis (see p. GC-01-86)

B – CGC, 200-325 mesh; same as sample B of GC-01-86.

C – Analcime, <325 mesh; same as in sample C of GC-01-86.

D – CDVH1; same material previously analyzed by Smith (Div. 01) (see page GC-02-24)

E – CDVH2; same material as in GC-02-24.

F – CH; same material as sample F for Smith analysis (see p. GC-01-88).

G – NBS 70A; same material used for Smith chemical analysis (sample E on page GC-01-88)

These samples were given to Roger Pierson of Div. 01 at about 1000 hrs.

1030 hrs – Prepare 5 EM mounts of samples MFD, CDV, and CCC. These will be used to obtain photomicrographs of the morphology of mordenite (MFD) and clinoptilolite (CDV and CCC).

1600 hrs –
Preparation of Homoionic Na-clinoptilolite

Objective: To obtain Na-form of clinoptilolite by equilibrating powdered CDV samples with NaCl solutions.

Method: Use CDVH2 obtained previously (see page GC-01-160). Chemical analyses performed on this

sample indicates wt% K equal to about 1.5%, compared to an initial value of about 3.2% for the unexchanged material. CDVH2 will be exchanged further with NaCl soln, this time with 3m NaCl. The temp will be 70°C to speed up the exchange.

3m NaCl soln was prepared from reagent grade NaCl (J.T. Baker, Lot # 886673) and deionized H_2O. Sample CDVH2 had a total weight of about 80 grams. About 40g were put in 600ml polyethylene plastic bottles, and about 500ml of 3m NaCl soln were added to each bottle. Each bottle was shaken to initially homogenize the mixture, then placed in a shaker water bath preheated to 70°C. The shaker was turned on at about 1650 hrs, set at 80 rpm.

5/8/90 MJP

0900 hrs – The bottles containing CDVH2 + 3m NaCl were shaken to homogenize the mixture, then were return to the shaker water bath at the same settings.

0915 hrs – SEM mounts of samples CCC, CN, and MED were given to H Gallema (Div. 08) for gold coating prior to SEM analysis.

1400 hrs – Observed $LiBO_2$ fusion of samples A – G (see p. CCC-02-28), plus blank solution. The blank solution and sample A dissolved almost completely, except for a few flakes and some carbon particles coming from the crucible. Samples B and C had problems dissolving: quite a bit of undissolved flakes remained, sample C became colloidal, and sample B also had a small amount of colloidal particulate. Samples D, E, F and G appeared to dissolve relatively well, probably with a few flakes remaining in some cases.

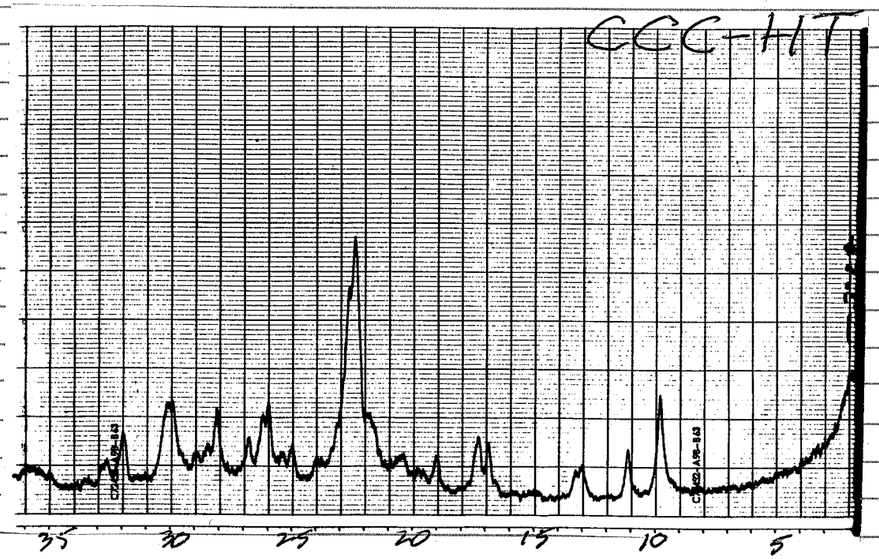
Samples B and C will be rerun until complete dissolution is observed.

In all cases, 5% HCl solution was used as the matrix. The sample and $LiBO_2$ reagent were initially mixed in a porcelain crucible, then transferred to a graphite crucible using a spatula and finally a camel's hair brush. Some powder was observed to fall on the graphite crucible edges, w/c the technician (Mrs. Presas) did not attempt to sweep into the crucible. Some powder was also observed to cling to the sides of the crucibles. Do these dissolve? Fuse and coalesce with the rest of the sample? Need to swirl

the molten bead well to pick them up.

1530 hrs – Received XRD reports of heated samples CDV-HT, CCC-HT and HPI-HT. The following are copies of the XRD patterns which were run on the Siemens D-500 diffractometer in Div. 06.

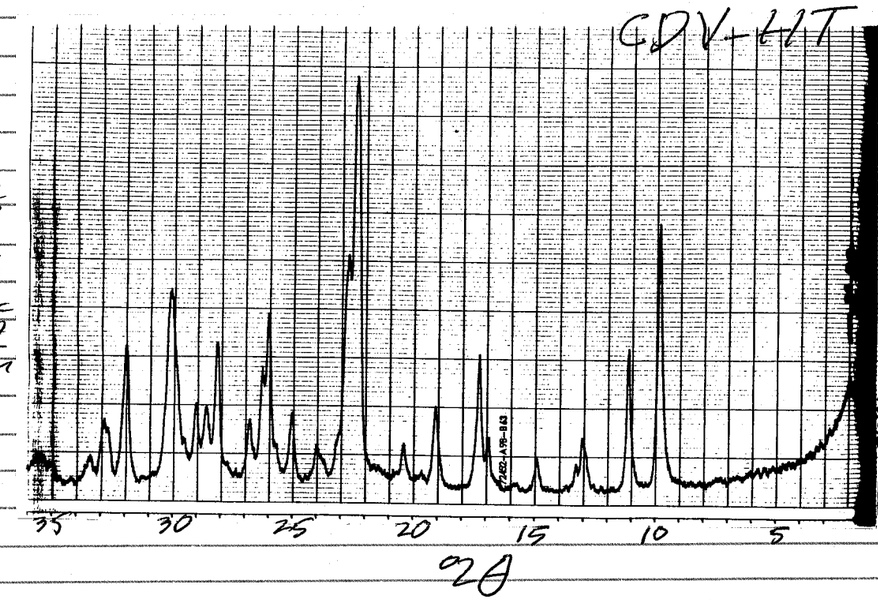
5/21/80
40 ma
40 kV
400 c.p.s.
ζ = 4 sec
0.5° 2θ/min



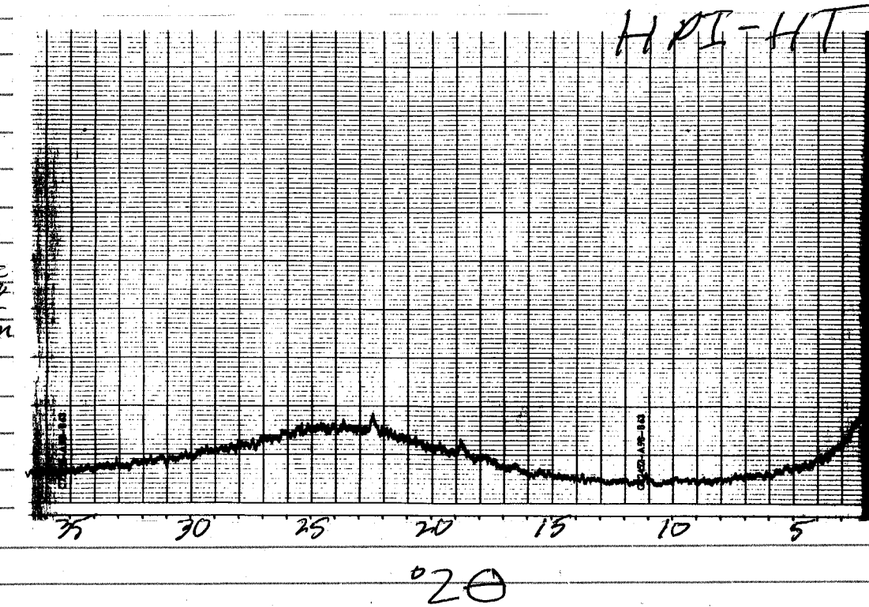
HPI-HT

The heulandite sample lost its crystalline structure after heating overnight at 400°C, while the zeolite samples retained their structures. Sample CCC-HT however, showed a marked decrease in clinoptilolite peak intensities compared to unheated CCC (p. GC-02-

5/8/80
40 ma
40 kV
400 cps
ζ = 4 sec
0.5° 2θ/min

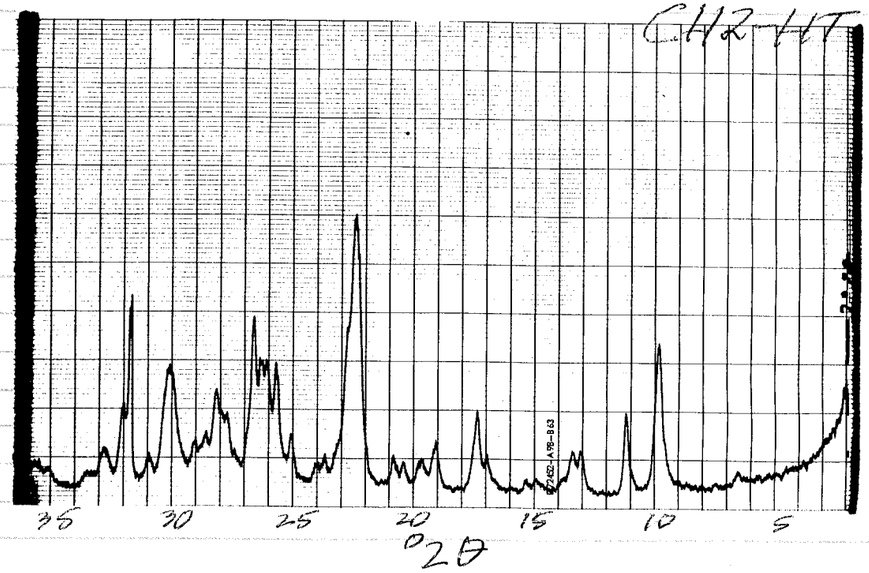


5/8/80
40 mA
40 kV
400 cps
ζ = 4 sec
0.5° 2θ/min



5/9/90 RDD

The following is a copy of the XRD pattern of sample CHZ-HT (sample of Hector clinoptilolite, heated ~~at~~ 450°C overnight; see p. GC-02-18).



There is a small decrease in intensity of clinoptilolite peaks compared to unheated sample (see page GC-02-11), but the sample retained its crystalline structure as indicated by sharp and well-defined XRD peaks.

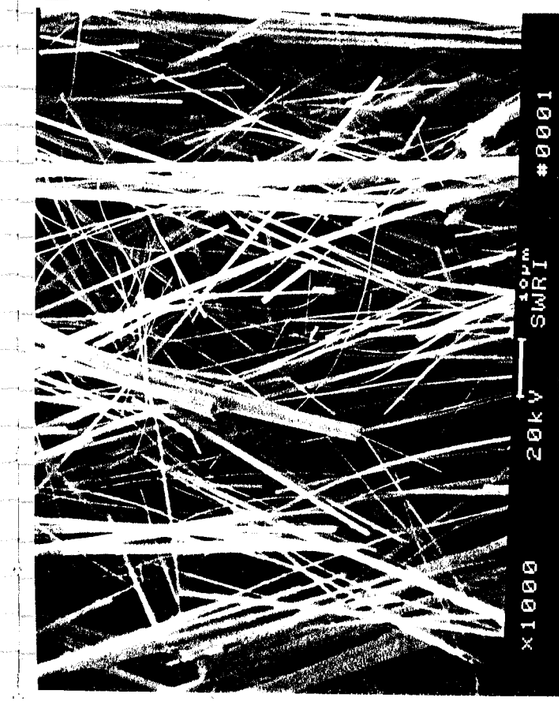
1600 hrs — The bottles containing CDV&Z in the water bath were shaken to get a homogeneous mixture. Temp reading = 70°C. Replaced in water bath with same settings.

1615 hrs — Reweigh samples CT-HT, CB-HT, and CGC-HT.

Sample	Wt Sample + beaker + parafilm
CT-HT	30.748 g.
CB-HT	32.016 g.
CGC-HT	32.179 g.

Relatively unchanged (1.011 g) since 5/7/90 (see p. GC-02-28).

1700 hrs — The following are SEM photomicrographs of samples MTO, CDV and CCC taken by H. Saldana using SWRI (Div. 06) equipment. (see p. GC-02-29, 1030 hrs). MTO is a ~pure mordenite sample.



(Page # 36)

(Page # 37)

Light # 2243

Light # 2243

Light # 2243

Light # 2243

Light # 2243

Light # 2243

Light # 2243

Light # 2243

(log # 4278)

(log # 4280)

(log # 4278)

(log # 4279)

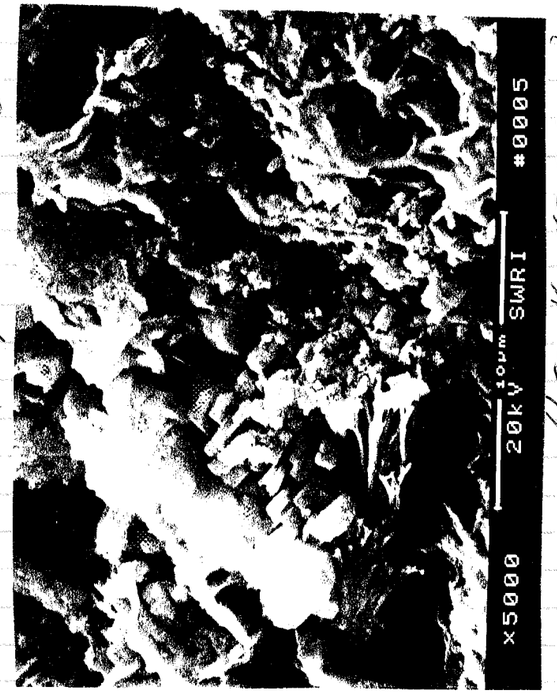
(log # 4279)

(log # 4280)

(log # 4278)

(log # 4280)

(Lot # 6344)



(Lot # 6344)

The following are copies of the results of reanalyses of ICBS standards 99A and 70A, w/c were initially analyzed on 5/4/90 (see p. GC-02-24)

\033Q1$SOUTHWEST RESEARCH INSTITUTE
6220 Culebra Rd.
San Antonio, Texas 78228-0510

Dept. of Environmental Sciences
Inorganic Section

ELEMENTAL ANALYSIS BY ICAP-AES

Data Analyzed: 90/05/07 — Time Analyzed 10:18
Analyst: Villasenor — Report Date: 90/05/07
Project Name: Pablon Reds — Project Number: 20-3704-013

	CLP-A11 ug/ml
ICV-3&4	5.481
ICB	-0.019
1-70A	Over Range
1-1:10	6.860
1-1:10DUP	6.505
1-1:10TRP	6.487
1-1:10QUAD	6.420
1-99A	Over Range
1-1:10	7.468
1-1:10DUP	7.266
1-1:10TRP	7.380
1-1:10QUAD	7.541
ICV-3&4	5.449
ICB	0.397
ICB	0.321

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6220 Culebra Rd.
San Antonio, Texas 78228-0510

Dept. of Environmental Sciences
Inorganic Section

ELEMENTAL ANALYSIS BY ICAP-AES

Data Analyzed: 90/05/07 — Time Analyzed 10:47
Analyst: Villasenor — Report Date: 90/05/07
Project Name: Pablon Rerun — Project Number: 20-3704-013

	CLP-Si1a ug/ml	CLP-Si2 ug/ml
ICV-15	15.65	15.83
CCB	-0.17	-0.24
70A	Over Range	Over Range
6-1:10	21.88	22.04
6-1:10DUP	21.27	21.52
6-1:10TRP	10.21	1.52
7-1:10	20.38	20.27
7-1:10DUP	20.29	20.27
7-1:10TRP	20.16	20.06
ICV-15	15.49	15.10
CCB	-0.21	-0.25

5/20/80 RDP

1100 hrs — One bottle containing the CDVHZ powder + 3 m NaCl solution was taken out of the water bath. The NaCl solution was decanted, and the powder rinsed with dei H_2O 6 times (about 440 ml H_2O each time). Test of Cl^- presence w/ $AgNO_3$ after 4th time did not indicate visible AgCl precipitate. The bottle was filled w/ about 400 ml H_2O and placed back in the water bath. Temp setting = 70°C, shaker speed = 80 rpm.

The second bottle was shaken to mix the contents and then placed back in the water bath. Label this bottle B, label the first one A.

1300 hrs — Notes from Alietti et al. (1974; Heat behavior of cation exchanged zeolites with heulandite structure, Tschermaks Min. Petr. Mitt., 21, p. 291–298)

Cation exchanged zeolites prepared by exchanging 1 g powdered material with 30 cc of 1N soln of salt of cation at 295°C; Teflon tube container oscillated in an oven; renewed N-soln after 3 days; after 10 days ion exchange appears to be nearly complete as indicated by chemical analyses.

Composition of zeolites shown on Fig 1 on next page.

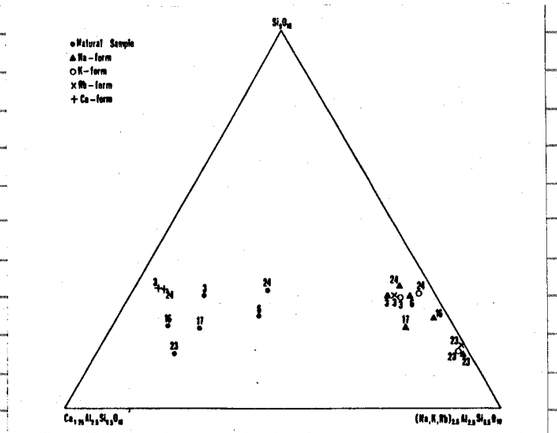


Fig. 1. Mole plot of the natural and exchanged phases in the upper part of the triangle Si_3O_{18} — $Ca_{4.5}Al_9O_{18}$ — $(Na, K, Rb)_9Al_9O_{18}$.

1410 hrs — Repair technician from Fisher repaired the Fisher Isotemp 184A muffle furnace; the pyrometer was replaced.

1430 hrs — Remove bottle A (see p. 40-42) from water bath. Decant small amount of water into small beaker; test Cl presence using 0.1N $AgNO_3$ solution. Result: AgCl precipitate observed. Decant completely and rinse the powder CDVHZ sample with dei H_2O 3 times. No precipitate observed after second rinse. Remove about 4 gms of wet powder from bottle A, put in 60 ml plastic bottle, add about 45 ml dei H_2O, and place bottle (w/ label A1) in water bath (still set at 70°C). This sample will be rinsed until no Cl is detected,

and will be analyzed chemically to check if ~~homogenous~~ pH 5/10/80 exchange with NaCl soln. has proceeded further.

New batch of 3M NaCl solution was prepared. Bottle B (see page GC-02-42) was decanted, and about 350 ml of new NaCl solution was added. Bottle B was then put back in the shaker water bath.

The dei H_2O in bottle A was replaced with 3M NaCl solution for further exchange. Bottle A was then placed back in water bath. Same settings.

1450 hrs —
Test of Fisher Isotemp Muffle Furnace 18-4A

Objective: Develop graph correlating power control setting to pyrometer readings. (see p. GC-02-18).

Pyrometer reading adjusted to read about 22°C when furnace is off. Furnace power set to LOW at 1450 hrs.

Time	T °C
1506	79
1535	138
1555	168
1610	180
1615	190
1643	202
(5/11/80) 1040	255

1600 hrs — Test A1 for Cl^-: no AgCl obs. Rinse, decant mixture into 50 ml Beaker, place in oven

5/11/80 MP

1040 hrs — Set power setting on muffle furnace to #2.

Time	Temp (°C)	Time	Temp (°C)
1040	255	1438	528
1113	383		
1137	436		
1200	462		
1250	503		
1305	507		
1325	518		
1415	526		

1100 hrs — Place A1 (in beaker, covered w/ parafilm) in dessic.

1250 hrs — Reweigh samples CT-HT, CGC-HT and CB-HT

Sample	Wt sample + beaker + parafilm	Wt beaker + parafilm	Wt sample
CT-HT	30.747 g	29.923	0.824
CB-HT	32.015 g	30.758	1.257
CGC-HT	32.178 g	30.811	1.367

Sample CT-HT gained 0.064 g after equilibrating with water vapor over saturated NaCl solution; CB-HT gained 0.110 g; CGC-HT gained 0.100 grams. In terms of weight % of the hyperous sample (fourth column above), CT-HT, CB-HT and CGC-HT have 7.77, 8.75, and 7.32 wt% H_2O in their respective structures. Time of equilibration ~ 96 hrs or less.

1438 hrs — Set power setting on muffle furnace to #3. Read pyrometer as a function of time:

Time	Temp (°C)	Time	Temp °C
1438	528		
1452	601		
1520	680		
1547	718		
(5/12/90) 1724 hrs.	780°C		
furnace	OFF.		

1700 hrs – The following is a copy of the chemical analytical results for samples A – G (see p. GC-02-28)

SOUTHWEST RESEARCH INSTITUTE
San Antonio, Texas 78228-0510

Page: 1
Project: 20-3704-013

Pahalan (mg/g = ppm)

	Sample id		Al	Si	Ca	Na	K	Fe		
NBS 99A →	A		98603	24.3015	14951	45993	47696	453		
CGC →	B		63678	260168	25050	8383	8110	10913		
ASH →	C		133767	209357	~~25000~~ <2500	106041	<2500	157		
CDYH1 →	D		58333	256182	<2500	37760	14478	3237		
CDYH2 →	E		58987	255682	<2500	38486	15770	3357		
CH →	F		64401	274002	<2500	38805	10512	6213		
NBS 70A →	G		98648	283185	<2500	17096	11209	714		

Data printouts provided Monday and Magnesium

fax 522-5155

Date: 5/11/90 Signature: MRiddle

The results for silicon appear to be low. Need to try if doing our own sample preparation (in Bldg 57), will give better results. Also need to try submitting standards for Si and Al together with the aqueous samples to check if there is problem with ICP analysis.

5/12/90 RJP

1725 hrs – Stop shaker bath. Remove bottle B & decant NaCl soln into beaker, then take about 4 grams of wet powder sample CDYH2 and place it in a 60 ml plastic bottle. Rinse 4 times with about 45 ml deI H_2O. Test for Cl^-: $AgCl\downarrow$ barely observed. Rinse once more; test shows no $AgCl\downarrow$. Rinse once more, decant, replace with ~45 ml deI H_2O then replace in water bath. Pour back NaCl soln into bottle B, swirl and put back in water bath. Swirl bottle A and replace in bath.

Label B1.

1740 hrs – Reweigh sample A1 w/c is equilibrating w/ water vapor over a saturated NaCl soln.

Wt A1 = 32.448 g.
+ beaker + parafilm

5/13/90 RJP

1825 hrs – Test B1 water for Cl^-. $AgCl\downarrow$ observed. Rinse once: no

AgCl ppt seen. Wash twice more and place back in water bath. Swirl A and B, then replace in water bath.

1830 hrs — Reweigh A1.
Wt A1 + beaker + paraffin = 32.580 g

5/14/90 MM

1058 hrs — Check B1 for Cl⁻. No AgCl observed. Rinse 2 times, swirl, decant mixture into 50 ml beaker. Let settle, decant H_2O. Place in oven set at ~80°C.

1100 hrs — Turn muffle furnace on. Set to 4 on dial.

Time	Temp (°C)	Time	Temp (°C)
1100	22	1450	960
1113	350	1556	965
1138	625	1640	965
1321	920	Turn off at 1640 hrs	
1334	934		
1410	948		

1105 hrs — Decant NaCl soln from bottle B. Transfer about 4 g wet powder into 60 ml btl, rinse 3 times; test for Cl⁻: AgCl observed. Rinse 2 more times: no ppt. Rinse and return to batch (with label B2)

Prepare 3 m NaCl solution

using Fisher ACS reagent lot # 886663. Put about 325 ml into btl. B, swirl then place B in water bath.

Decant btl. A, replace the NaCl soln. with newly prepared batch (~325 ml). Swirl, then put back in water bath.

1115 hrs — Reweigh A1.
Wt A1 + beaker + paraffin = 32.581 g, compare to yesterday's 32.580 g. Appears to have equilibrated. Time of equilibration ~55 hrs.

1455 hrs — Check B2 for Cl⁻: AgCl observed. Rinse 2 times: No AgCl. Replace deI H_2O, then put back in bath.

1600 hrs — Take sample B1 out of oven, let cool in desiccator for about 15 mins. Weigh.

Wt B1 (+ beaker + paraffin) = 31.269 g.

1620 hrs — Check B2 for Cl⁻: No AgCl observed. Rinse once; replace deI H_2O, ~~then put back in water bath~~, decant mixture to beaker. Refill water bath with ultrapure H_2O to replace evaporation loss. Turn back on at 1630 hrs after swirling bottles A & B.

Then put in oven set at ~80°C for drying. MM 5/14/90

5/15/90 MJP

0900 — Shaker water bath found not shaking. Could have been caused by low water level warning light observed yesterday (and ignored because the tank has just been refilled). Temp reading = 70.1°C. Motor restarted by pressing start button.

0905 — Turn on ~~water bath~~ PP listed muffle furnace, power set to 5.

Time	Temp (°C)	Time	Temp (°C)
0905	22	1021	1008
0912	200	1320	>1100 Reset due to 4.25
0925	535		
0932	640		
0956	880		
1015	980		

0910 hrs — Take sample B2 from oven, cover beaker w/ parafilm, let cool in dessicator for about 15 mins. Then weigh.

Wt B2 + beaker + parafilm = 31.692

Also reweigh A1 and B1

A1 = 32.581 g.
B1 = 31.391 g.

1545 hrs — Reweigh A1, B1, B2

A1 = 32.579
B1 = 31.389
B2 = 31.781

1555 = Reweigh A1, B1, B2

A1 = 32.578
B1 = 31.389
B2 = 31.787

5/16/90 MJP

0900 hrs — Turn muffle furnace on set to 5.75 (max.). Adjust later to give 1000°C.

0905 — Reweigh A1, B1, B2

A1 = 32.582
B1 = 31.393
B2 = 31.811

Almost all of NaCl salt in dessicator is expended. Add more salt to maintain saturation.

0945 hrs — Stop shaker bath. Remove bottle B; take about 4 g. of wet powder and transfer into clean bottle, label B3. Swirl bottles B and A, return to bath & restart shaker.

Rinse B3 four times with deI H_2O; test for Cl^-: $AgCl$ observed. Rinse 2 more times; test for Cl^- shows negative result. Rinse once more, replace deI H_2O then place in water bath at 20°C.

1000 hrs — Prepare 5% HCl from concentrated HCl (Fisher lot # 886194).

2030 hrs —
Lithium Metaborate Fusion
of Clinoptilolite Samples

Objective: Dissolution of clinoptilolite samples for ICP and/or AA analysis. Samples to be dissolved are CDV powder exchanged with 3M NaCl solutions over a period of several days.

Method: Procedure based on ASTM 4503-86. Technical operating procedure being developed based on results of this work. See also page GC-02-25.

(1) Blank ($LiBO_2$ flux only)

9.7363 wt porcelain crucible
(1.2 wt $LiBO_2$ to be added
10.9363) expected weight

10.9370 actual wt, crucible + $LiBO_2$
9.7361 wt porcelain crucible after transferring contents to graphite crucible

$\boxed{\text{Wt } LiBO_2 \text{ flux} = 1.2007\text{ g}}$

(2) CDVH3 (taken from sample B1, page GC-02-43)

9.8946 wt porcelain crucible
(1.2
11.0946)

11.0953 wt. crucible + $LiBO_2$
(0.2 wt sample to be added
11.2953) expected weight

11.2959 wt crucible + $LiBO_2$ + sample
9.8942 wt porcelain crucible empty

$\boxed{\text{Wt sample CDVH3} = 0.2006\text{ g}}$

(3) CDVH4 (taken from sample B1, page GC-02-47)

8.5254 wt crucible
(1.2
9.7254)

9.7253 wt crucible + $LiBO_2$
(0.2
9.9253)

9.9273 wt cruc. + $LiBO_2$ + sample
8.5251 wt crucible empty

$\boxed{\text{Wt sample CDVH4} = 0.2020\text{ g}}$

(4) CDVH5 (from sample B2, p. GC-02-50)

9.7362 wt crucible
(1.2
10.9362)

10.9337 wt cruc + $LiBO_2$
(0.2 / 10.1337)

11.1345 wt cruc + $LiBO_2$ + sample
9.7360 wt crucible empty

Wt sample CDVH5 = 0.2008 g.

(5) NBS 99 A (same aliquot as in page GC-02-28).

9.8946 wt crucible
(1.2 / 11.0946)

11.0938 wt cruc + $LiBO_2$
(0.2 / 11.2938)

11.2942 wt cruc + $LiBO_2$ + sample
9.8942 wt crucible empty

Wt NBS 99A = 0.2004 g

Samples were fused at ≥ 1000°C for about 20 mins. using graphite crucibles and Fisher Isotemp muffle furnace.
Complete dissolution was observed for all samples except NBS 99A which has a few small crystals left in the beaker. However these crystals appear to have redissolved upon dilution to 250 ml in a

volumetric flask. Some small fused beads were also observed in the cooled graphite crucible used for NBS 99 A.
The solutions were transferred into pre-washed 250 ml plastic bottles, and given to B. Riddle for ICP and/or AA analysis (1430 hrs).

1315 hrs — Test B3 for Cl^-; $AgCl_4$ observed. Rinse twice; no $AgCl_4$ observed. Decant and replace deI H_2O; put back in water bath at 70°C; restart shaker.

1600 hrs — Reweigh A1, B1, B2
A1 = 32.380
B1 = 31.280
B2 = 31.608

Compare with weights on 5/16/80 / 0800,
Wt loss A1 = 0.202
Wt loss B1 = 0.203
Wt loss B2 = 0.203

Compare with weight of sample

CDVH3 0.201
CDVH4 0.202
CDVH5 0.201

Additional loss during transfer to crucible, but not much, ≤ 2 mg.

1610 hrs — Test B3 for Cl⁻: no AgCl detected. Rinse once more, decant powder into beaker (50 ml) then place in oven for drying.

5/17/90 RDP

0915 hrs — Remove sample B3 from oven; cover w/ parafilm & let cool in dessicator for about 15 mins.

Shaker water bath found not shaking. Cause unknown, no warning light on. ~~Restart~~ RDP 5/19/90 Refill water, then restart.

0950 hrs — Weigh B3 = 32.470 g. Put in dessicator over solid NaCl soln.

Reweigh A1, B1, B2:
A1 = 32.381
B1 = 31.191
B2 = 32.609

1200 hrs — Reweigh B3 = 32.542 g.

1645 hrs — Reweigh B3 = 32.564 g

5/18/90 RDP

0930 hrs — Reweigh A1, B1, B2, B3

A1 = 32.381
B1 = 31.191
B2 = 31.610
B3 = 32.588

1135 hrs — Reweigh B3 = 32.588 g.

1610 hrs — Reweigh B3 = 32.587 g.

Below is a copy of the chemical analysis on samples A – G including the flame AA results for Mg and Ca. (see page GC-02-28 and GC-02-46).

SOUTHWEST RESEARCH INSTITUTE
San Antonio, Texas 78228-0510

Page: 1
Project: 20-3704-013

Potsdam (mg/g = ppm)

	Sample id	Al	Si	Ca	Na	K	Fe	Flame Mg	Flame Ca
NBS99A →	A	98603	243015	14951	45993	47696	453	<25	3382
CGC →	B	63678	260168	25050	8383	8110	10913	9102	26042
ASH →	C	133767	209357	<2500 ~~25000~~	106041	<2500	157	<25	<2.5
CDVH1 →	D	58333	256182	<2500	37760	14478	3237	556	173
CDVH2 →	E	58987	255682	<2500	38486	15770	3357	417	452
CH →	F	64401	274002	<2500	38805	10512	6213	485	1031
NBS 70A →	G	98642	285185	<2500	17096	112049	714	49.4	235

Data printouts provided Monday and Magnesium

fax 522-5155

Date: 5/11/90 Signature: M Riddle

5/19/90 RDP
1600 hrs – Reweigh sample B3.
wt B3 = 32.588 g.

1700 hrs – Take sample B4 from bottle B in water bath. Approximately 4g wet sample. Rinse B4 several times and check for Cl⁻ using $AgNO_3$: AgCl observed. Rinse 2 more times: no AgCl. Rinse once more, replace del H_2O, then put into water bath.
Renew bottles A and B with new batch of 3mNaOH soln. Swirl A and B and replace in water bath. Refill bath with H_2O.

5/20/90 RDP
1445 hrs – check B4 for Cl⁻: AgCl observed. Rinse once: no AgCl. Rinse twice, then decant powder into 50 ml beaker; put in oven for drying.

5/21/90 RDP

0815 hrs – Take B4 out of the oven. Cover with aluminum foil then quickly put in dessicator for cooling to room temperature.

1000 hrs – Weight B4 = 32.359 g.

1808 hrs – wt B4 = 32.462 g.

5/22/90 RDP
1115 hrs – Weight B4 = 32.478 g.

1350 hrs – Weight B4 = 32.478 g

5/23/90 RDP
1530 hrs – Reweigh A1, B1, B2, B3, B4
weight (g.)
A1 = 32.388
B1 = 31.191
B2 = 31.610
B3 = 32.588
B4 = 32.478

1535 hrs – Refill water bath. Swirl bottles A and B. Restart bath using the same settings.

5/24/90 RDP

0940 hrs – Reweigh A1, B1, B2, B3, B4
A1 = 32.388
B1 = 31.192
B2 = 31.611
B3 = 32.589
B4 = 32.479

Turn on muffle furnace, set to maximum.

1000 hrs – Prepare your first NIST standards for $LiBO_2$ fusion. Analyze NBS 70A, NBS 99A, CDVH3 (from A1), CDVH4 (from B1), CDVH5 (from B2), CDVH6 (from B3), and CDVH7 (from B4). Dilute to 100 ml w/ 5% HCl.

① NBS70A

9.8461 wt porcelain crucible
(1.2 / 11.0461)

11.0448 + $LiBO_2$
(0.2 / 11.2448)

11.3160 + sample
9.8461 empty crucible

Rezero balance. Reading ~.0002 when empty.

② CDVH3 (from sample A1)

9.9504 wt crucible
(1.2 / 11.1504)

11.1579 + $LiBO_2$
(0.2 / 11.3579)

11.3952 + sample
9.9502 empty crucible

③ CDVH4 (from sample B1)

8.5250 wt crucible
(1.2 / 9.7250)

9.7262 + $LiBO_2$

9.9264 + sample
8.5250 empty crucible

④ CDVH5 (from B2)

9.7359
(1.2 / 10.9359)

10.9318 + ~~sam~~ $LiBO_2$
11.1317 + sample
9.7360 empty crucible

⑤ ~~9.8945~~ NBS2420
CDVH6 (from B3)

9.8945 wt crucible
11.0981 + $LiBO_2$
11.3025 + sample
9.8942 empty crucible

⑥ CDVH7 (from B4)

8.8241 wt crucible
10.0258 + $LiBO_2$
10.2342 + sample
8.8240 empty crucible

⑦ NBS99A
9.8084 wt crucible
11.0002 + $LiBO_2$
11.2009 + sample
9.8080 empty crucible

⑧ Blank 10.5755 wt crucible + sample
9.7393 wt crucible 9.7350 empty cruc.

1520 — Submit samples prepared above to B Middle (D.V.O1) for ICP and AA analysis. Label samples NC are in 125 ml plastic bottles.

A — NBS70A = 0.2712 g.
B — CDVH3 = 0.1873 g.
C — CDVH4 = 0.2002
D — CDVH5 = 0.1999
E — CDVH6 = 0.2044
F — CDVH7 = 0.2084
G — NBS99A = 0.2007
Blank ($LiBO_2$) = 0.7802

1700 hrs — The following are summaries of chemical analyses done on NBS standards and solute samples, as of 5/21/90.

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 99A
SAMPLE A

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	4.99	3.9	1.25	0.002	0.043	11.3	31.8
CORE LAB	4.8	5.7	1.75	<0.02	0.0403	12.0	32.4
SWRI	4.89	3.9	1.35	0.008	0.049	8.78	0.17
	5.0	4.0	1.4	<0.05	0.06	10.9	5.0
							3.5
(3/28/90)	3.92	3.8	1.33		0.045	6.67	19.5
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	21.9
(4/2/90, 0.05g sample, 0.5g LiBO2)						18.2	16.1
(4/11/90, .02g sample, 0.5g LiBO2)						10.5	27.0
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						11.0	30.3
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							29.5
(5/3/90)	4.46	4.32	1.15	0.009	0.04	9.79	25.2
(5/11/90)	4.60	4.77	1.50		0.045	9.86	24.3
(5/14/90)			0.34	<0.0025			
(5/21/90;Si1)	4.64	4.21	1.29	0.0062	0.05	9.93	28.62
(5/21/90;Si2)							28.38
NBS	4.6	4.315	1.53	0.012	0.0455	10.85	30.48

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 70A
SAMPLE E

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	2.1	9.61	0.076	0.003	0.051	9.6	32.5
CORE LAB	2.24	4.3	0.093	0.018	0.0784	9.1	28.8
SWRI	1.74	7.67	<0.12	0.0097	0.0531	5.34	0.10
(3/28/90)	1.93	9.33	<0.25		0.059	7.87	13.99
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	20.7
(4/2/90, 0.05g sample, 0.5g LiBO2)						8.7	18.4
(4/11/90, 0.02g sample, 0.5g LiBO2)						6.1	26.1
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						9.7	30.7
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							32.3
(5/3/90)	1.78	9.46	<0.25	0.024	0.05	9.06	27.81
(5/11/90)	1.73	11.20	<0.25		0.07	9.86	28.52
(5/14/90)			0.024	0.0049			
NBS	1.89	9.8	0.079	0.0	0.0525	9.474	31.37

RESULTS OF CHEMICAL ANALYSIS OF CGC (35-200 MESH)
SAMPLES B AND D

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB (B)	1.05	0.96	2.38	0.68	0.98	6.45	28.8
(D)	1.07	0.999	2.36	0.71	0.89	6.2	28.9
CORE LAB (B)	<0.01	0.81	0.93	0.69	1.15	6.00	31.2
(D)	1.08	0.88	2.6	0.64	1.2	6.0	31.6
SWRI (B)	0.99	0.89	2.38	0.943	0.990	5.36	0.08
(D)	1.10	0.9	2.35	0.966	1.06	5.22	0.12
(5/11/90;D)	0.84	0.81	2.51		1.09	6.37	26.02
(5/14/90;D)			2.60	0.910			

RESULTS OF CHEMICAL ANALYSIS OF ANALCIME
SAMPLES C (TRANSLUCENT) AND G (WHITE)

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB (C)	10.4	0.028	0.008	0.0001	0.017	13.0	27.2
(G)	10.3	0.026	0.011	0.0003	0.015	12.6	31.6
CORE LAB (C)	10.8	<0.00005	0.019	0.0007	0.016	12.9	27.1
(G)	10.8	0.87	0.02	0.0027	0.0193	12.1	25.3
SWRI (C)	10.34	<0.12	<0.12	0.0012	0.0223	10.8	0.19
(G)	9.36	<0.12	<0.12	0.0012	0.0223	9.77	0.26
(5/11/90;C)	10.6	<0.25	<0.25		0.0157	13.38	20.94
(5/14/90;C)			<.0025	<.0003			

RESULTS OF CHEMICAL ANALYSIS OF CLINOPTILOLITE, DEATH VALLEY JUNCTION
SAMPLES F AND H

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB (F)	4.01	1.06	0.36	0.039	0.47	6.22	31.4
(H)	4.02	1.04	0.37	0.039	0.47	6.21	31.5
CORE LAB (F)	4.4	11.4	0.36	0.13	0.40	5.7	33.6
(H)	4.4	1.1	0.35	0.12	0.42	5.9	33.4
SWRI (F)	4.09	0.75	0.45	0.090	0.55	5.03	0.26
(H)	4.26	0.87	0.45	0.091	0.56	4.97	0.25
(5/11/90;F)	3.88	1.05	<0.25		0.62	6.44	27.4
(5/14/90;F)			0.103	0.0485			

RESULTS OF CHEMICAL ANALYSIS OF CLINOPTILOLITE, DEATH VALLEY JUNCTION

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
CDV (5/4/90)	2.56	3.15	0.35	0.20	0.46	6.05	30.04
CDVP (5/4/90)	2.49	3.11	<0.25	0.17	0.34	5.90	30.89
CDVH1 (5/4/90)	4.05	1.66	<0.25	0.14	0.28	11.13	28.39
(5/11/90)	3.78	1.45	<0.25		0.33	5.83	25.61
(5/14/90)			0.017	0.056			
CDVH2 (5/4/90)	3.99	1.48	<0.25	0.13	0.31	5.82	30.29
(5/11/90)	3.85	1.58	<0.25		0.34	5.90	25.57
(5/14/90)			0.045	0.042			
CDVH3 (5/21;S11)	5.35	1.23	0.050	0.146	0.38	7.4	36.95
(5/21;S12)							35.9
CDVH4 (5/21;S11)	4.37	0.73	0.047	0.116	0.33	0.23	28.38
(5/21;S12)							28.18
CDVH5 (5/21;S11)	4.43	0.67	0.047	0.115	0.34	5.39	30.13
(5/21;S12)							29.31
~~CDVH3~~	~~4.28~~	~~.95~~	~~.04~~	~~.117~~	~~.304~~	~~5.92~~	~~29.6~~
							~~28.7~~

RM 6/6/90
Recalculated because wrong sample was diluted 200 ml instead of 100 ml flask (wrong flask used). Blank also diluted to 200 ml instead of 100 ml flask

Some decrease still observed in K concentration with corresponding increase in Na concentration. Al and Si analyses appear to be erratic.

5/25/90 RJH

0900 hrs – Prepare 3 m NaCl soln from ACS grade NaCl (Fisher Lot # 901163).

0915 – Take sample B4 from bottle B in water bath. Rinse 4 times. Check Sol. Cl⁻: AgCl observed. Rinse 2 times, check Sol Cl⁻: no AgCl. Rinse once more, replace dil HNO₃, then place in water bath.

1000 hrs –

Constant Weight CDV Samples at 900 °C

Objective: Check how long it takes for CDV samples to achieve constant weight when heated at 900 °C. This procedure is needed to determine how long it takes to get the weight of water in the zeolite samples by heating at 900 °C.

Method: Use untreated ~~100–200~~ 35–100 mesh sized CDV samples, quartz air-tight crucibles (Fisher Cat # 10-490-4) and Fisher Systems muffle furnace. Wear a clean pair of gloves when handling the quartz crucibles.

Weigh the crucibles empty. Place about 2 g. of CDV (35–100 mesh) in each. Measure total

wt of crucible + sample before putting in ~~Furnace~~ Place in furnace which is set at 900°C. ~~Then~~ After one hour at 900°C, take out and let cool, then reweigh. Cooling should be done inside a dessicator. After weighing replace in furnace, heat for additional ~~two~~ hours. Repeat until increasing heating time.

Crucible #	Wt empty	Actual time / Weight		
		Start: 1143 hrs End: 1243 hrs	Start: 1340 hrs End: 1440 hrs	Start: 1500 End: 1600
1	11.1000 g.	13.1179 g.	⊘	⊘
		12.8539	12.8530	12.8536
2	10.4310 g.	12.4460 g.	⊘	⊘
		12.1835	12.1831	12.1832
3	11.4300 g.	13.4434 g.	⊘	⊘
		13.1813	13.1810	13.1810
4	11.4266 g.	13.4450 g.	⊘	⊘
		13.1812	13.1809	13.1802

The procedure described above assumes negligible readsorption of H_2O from the atmosphere by the zeolites during the cooling and weighing process.

Furnace dial set at 3.75 which controls the temp between 900 to 925°C.

Result: Two hours appears to give constant weight.

1330 — Check sample B5 for Cl^-: $AgCl$ observed. Rinse twice. Check again for Cl^-: no $AgCl$. Rinse, replace deI H_2O, then put back in water bath.

1545 hrs — Reweigh A1, and B1 to B4

A1 = 32.189 g	32.388	0.199
B1 = 30.990	31.192	0.202
B2 = 31.409	31.611	0.202
B3 = 32.383	32.589	0.206
B4 = 32.269	32.479	0.210

⊘ wts as of 5/24/90; see page GC-02-59

Δ wt

compare with values on page GC-02-62; shows + 0.002 g. Not much lost during weighing process for $Li_2B_4O_7$ fusion

1600 hrs — Check sample B5 for Cl^-: no $AgCl$ observed. Rinse twice; decant powder into 50 ml beaker; place in oven at 80°C for drying.

5/28/90 MDD

0845 hrs — reweigh crucibles 1 to 4.

Crucible	Wt.
1	12.8540
2	12.1837
3	13.1814
4	13.1805

Compared with weights on 5/25/90 (page GC-02-66), showed gain of 0.3 to 0.5 milligrams. Transfer contents of each ~~vial to~~ crucible to a plastic vial; label CDV 900°C.

0855 hrs – Take sample B5 out of oven. Cover w/ aluminum foil, place in dessicator, let cool for about 20 mins. Then weigh, after replacing foil w/ ~~B5~~ parafilm as cover.

Wt B5 + beaker + parafilm = 32.4174 g

1630 hrs – Reweigh B5 = 32.5047 g.

5/29/90 RDM
1530 hrs – Reweigh B5 = 32.5363 g

5/30/90 RDM
0825 hrs – Reweigh B5 = 32.5375

5/31/90 RDM

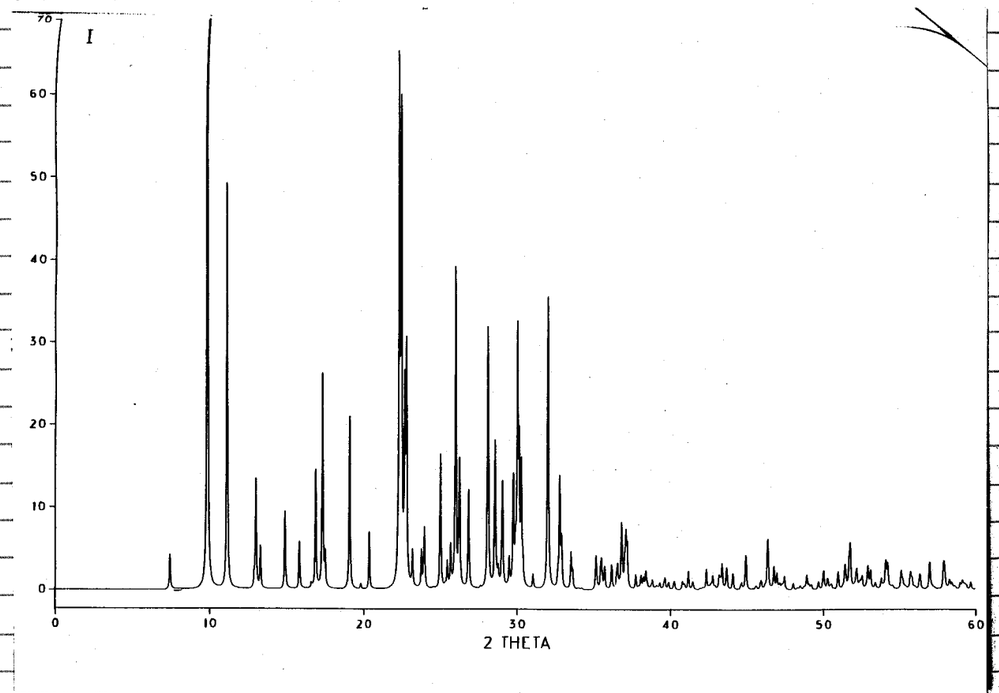
1445 hrs – Take sample B6 from bottle B in water bath. Rinse four times, check for Cl^-: AgCl ↓ observed. Rinse 2 more times, check

for Cl^-: AgCl ↓ observed. Rinse 2 additional times: no AgCl ↓. Rinse once, replace dist H_2O. Place in water bath.

1500 hrs – Prepare 3 m NaCl using reagent NaCl, ~~Lot #~~ 901163. Refill NaCl solutions in bottles A and B, swirl bottles; replace in water bath.

Conducted training of Paul Bertetti (summer intern from UTSA) on $LiBO_2$ fusion of rock and mineral samples.

Below is a copy of simulated XRD pattern of clinoptilolite. Some peaks are not identified in JCPDS files, e.g. 7.46° 2θ. Will need to reevaluate identification of XRD peaks for zeolitized tuff specimens, as reported in quarterly research report #1 (see pages CC-02-10 to CC-02-14). Some peaks may have been assigned to mordenite and/or erionite.



CLINOPTILOLITE

COMPOSITION: $Na_{1.8}K_{1.68}Mg_{0.25}Ca_{1.16}Al_{6.33}Si_{29.67}O_{72}(H_2O)_{20.1}$
Agoura, USA

CRYSTAL DATA: Monoclinic, C2/m, no. 12.
a = 17.662 Å, b = 17.911 Å, c = 7.407 Å
α = 90.00 °, β = 116.40 °, γ = 90.00 °
single crystal refinement, R_w = 0.09.

REFERENCE: K. Koyama and Y. Takéuchi, Zeit.Kristallogr., 145, 216-239 (1977).

POWDER DIFFRACTION DATA:

2θ	d	Irel	h	k	l
7.46	11.857	4.3	1	1	0
9.88	8.956	100.0	0	2	0
11.19	7.910	50.9	2	0	0
12.94	6.842	1.8	-1	1	1
13.06	6.780	13.5	-2	1	1
13.35	6.635	5.4	0	0	1
14.94	5.929	9.8	2	2	0
15.87	5.586	1.1	1	1	0
16.63	5.331	0.6	0	2	1
16.91	5.243	15.7	-3	1	1
17.36	5.110	27.0	1	1	1
17.53	5.058	4.4	3	1	0
19.10	4.648	22.0	-1	3	1
19.83	4.478	9.8	0	3	0
20.40	4.354	7.4	4	0	0
22.36	3.976	64.7	1	3	1
22.48	3.955	22.6	4	2	0
22.50	3.952	40.9	3	3	0
22.71	3.916	24.6	-4	0	1
22.82	3.897	29.2	2	4	0
23.21	3.832	4.8	2	0	1
23.82	3.736	5.0	-2	4	1
23.98	3.712	2.9	0	4	1
24.04	3.702	3.3	-2	0	2
25.05	3.555	18.5	-3	1	2
25.36	3.511	0.5	1	4	1
25.50	3.494	3.5	-1	5	0
25.73	3.463	5.5	-5	1	1
26.05	3.421	43.1	-2	3	2
26.29	3.390	15.6	-4	0	2
26.32	3.386	3.6	1	0	1
26.88	3.317	13.2	0	0	2
28.15	3.170	36.5	-4	2	2
28.60	3.121	19.5	-4	4	1
28.70	3.111	1.1	0	2	2
28.80	3.100	2.3	-3	3	2
29.01	3.078	1.5	2	4	1
29.07	3.072	15.0	-1	3	2
29.51	3.027	7.4	-5	3	1
29.79	2.999	14.9	-3	5	1
29.92	2.986	1.9	3	3	1
29.93	2.985	2.5	0	6	0
30.06	2.973	33.7	1	5	1
30.15	2.964	0.9	4	4	0
30.16	2.963	16.2	3	5	0
30.30	2.950	15.7	1	1	2
30.41	2.940	3.1	-6	0	1
31.05	2.880	2.2	4	0	1
32.01	2.796	33.1	-5	3	0
32.95	2.793	11.5	-6	2	1
32.66	2.742	1.9	-6	0	2/4
32.78	2.732	14.6	-2	6	1
32.80	2.731	0.6	-5	3	2
32.90	2.722	6.4	0	6	1
32.52	2.674	5.2	1	3	2
33.62	2.665	3.5	0	4	2/2
35.13	2.555	4.9	2	2	2

2θ	d	Irel	h	k	l
35.21	2.549	0.6	-3	5	2
35.44	2.533	2.0	-1	5	2
35.49	2.529	3.6	6	2	0
35.54	2.526	0.9	1	7	0
35.71	2.514	3.1	-5	0	1
36.16	2.484	1.4	3	5	1
36.19	2.482	0.7	-7	1	1
36.50	2.462	1.6	-7	6	1
36.57	2.457	3.2	-6	4	1
36.83	2.441	8.5	-2	6	1
36.85	2.439	1.4	5	1	1
37.02	2.428	4.0	-4	5	1
37.11	2.422	7.0	4	4	1
37.20	2.417	5.5	-7	3	1
37.76	2.383	0.2	4	6	0
38.09	2.362	2.0	-4	2	3
38.27	2.352	0.7	0	8	0
38.41	2.344	2.6	-2	2	3
38.50	2.338	0.6	-6	4	2
38.75	2.324	0.5	-2	6	2
38.84	2.319	1.5	3	7	0
39.13	2.302	0.6	3	7	0
39.34	2.290	1.1	2	4	2
39.60	2.276	0.4	5	5	1
39.67	2.272	1.8	6	0	0
39.99	2.260	1.0	-6	6	1
40.26	2.240	0.8	-4	6	2
40.28	2.239	0.7	0	8	0
40.80	2.212	1.3	0	0	3
40.93	2.205	0.9	3	3	3
41.20	2.191	2.9	-6	2	3
41.49	2.176	1.2	-1	3	3
42.35	2.134	1.2	-2	8	1
42.52	2.126	0.6	-2	8	1
42.66	2.120	0.8	4	0	3
42.78	2.114	2.1	7	3	0
42.94	2.106	0.4	-7	1	3
43.19	2.095	2.0	-6	6	1
43.27	2.091	1.1	-3	7	2
43.38	2.086	1.8	6	2	1
43.67	2.073	2.9	4	6	1
43.69	2.072	0.7	-5	7	1
44.07	2.055	1.8	3	7	1
44.09	2.054	0.15	1	8	1
44.66	2.029	1.1	5	5	1
44.93	2.018	3.0	-4	4	3
44.96	2.016	3.0	-7	5	2
45.89	1.976	0.5	8	0	0
45.92	1.976	0.7	6	6	0
45.96	1.975	0.5	-5	9	0
46.18	1.966	0.6	-5	7	2
46.35	1.959	3.3	-8	4	1
46.38	1.958	1.5	-8	2	1
46.39	1.958	4.6	-1	5	3
46.78	1.942	3.6	-9	1	2
46.98	1.934	2.6	8	4	1

2θ	d	Irel	h	k	l
47.17	1.927	1.0	-8	2	3
47.30	1.922	0.8	-1	9	1
47.46	1.916	2.1	-2	8	2
48.02	1.895	1.1	5	1	2
48.49	1.877	0.8	7	2	3
48.74	1.868	0.6	-4	8	2
48.92	1.862	2.4	3	9	0
49.07	1.857	0.8	-9	3	1
49.23	1.851	0.9	-4	0	4
49.69	1.835	1.4	-5	1	4
49.98	1.825	0.7	-7	5	3
50.04	1.823	1.0	-3	1	4
50.27	1.815	1.1	-8	3	2
50.31	1.814	1.1	-6	0	4
50.55	1.806	0.9	-8	4	3
50.89	1.791	2.0	0	10	0
51.01	1.790	0.8	-7	7	1
51.40	1.778	0.5	-6	2	4
51.42	1.777	1.2	0	6	3
51.44	1.776	2.4	-3	7	3
51.51	1.774	1.3	4	8	1
51.72	1.768	1.0	4	8	1
51.78	1.766	5.6	-7	7	2
51.80	1.765	1.1	-10	0	2
52.07	1.756	0.5	-2	3	4
52.16	1.754	0.9	-7	3	4
52.22	1.752	3.0	-3	3	4
52.44	1.745	1.3	-9	0	2
52.58	1.741	1.9	-5	7	3
52.95	1.729	3.7	0	10	1
53.14	1.724	2.8	3	9	1
53.45	1.714	1.0	3	8	2
53.83	1.703	1.9	5	3	3
53.95	1.700	0.5	8	0	1
54.11	1.695	2.5	-9	2	1/-9
54.15	1.694	2.8	-7	7	0
54.26	1.691	3.5	-10	0	3
54.47	1.690	0.5	1	10	2
54.59	1.681	0.7	-6	4	4
55.15	1.665	3.1	-8	6	2
55.24	1.663	1.1	-1	3	4
55.76	1.649	2.7	8	4	0
55.86	1.646	1.5	-2	8	3
56.35	1.633	0.8	-3	7	3
56.39	1.632	1.4	4	10	0
56.40	1.631	0.9	-3	5	4
57.03	1.615	0.8	5	6	3
57.93	1.592	3.6	3	5	3
57.99	1.591	2.7	-6	8	1
58.05	1.589	0.9	8	4	1
58.33	1.582	1.5	10	0	0
58.49	1.578	1.2	9	5	0
59.03	1.565	1.2	7	1	3
59.17	1.561	1.0	-8	8	1
59.32	1.558	0.7	10	2	0
59.71	1.549	1.4	6	8	1

COLLECTION OF SIMULATED XRD POWDER PATTERNS FOR ZEOLITES

Roland von Ballmoos

MOBIL RESEARCH AND DEVELOPMENT CORPORATION
Central Research Division
P.O. Box 1025,
Princeton, NJ 08540, USA

Issued by
the Structure Commission of the International Zeolite Association

6/1/90 RJP

0950 hrs — Check sample B6 for Cl⁻: AgCl observed. Rinse 2X, check for Cl⁻: no AgCl. Replace dei H_2O, put back in water bath.

1005 hrs — Reweigh B5 = 32.5368 g

1345 hrs — Check B6 for Cl⁻: no AgCl observed. Rinse 2X. Decant mixture into 50 ml beaker. Pour off H_2O. Place in oven for drying.

1630 — Take B6 out of oven. Cover w/ aluminum foil, let cool in dessicator.

1645 — Weigh B6 = 32.3469 g including beaker & parafilm cover

Place in desiccator over saturated NaCl solution.

1655 - Reweigh BS = 32.5351 g.

Below are the results for the samples submitted on 5/24/90 (see pp. CC-02-59 to 62). Samples E & F were inadvertently switched w/ G and Blank, respectively; their concentrations were recalculated and given in the second row of each sample name.

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Pabglan 5/23/90

	Sample id	Na	Fe	Al	Si1	Si2	Ca	Mg	K
				(% by wt)	(251)	(288)			
	A	1.86	0.045	9.82	28.80	28.91	~~0.0024~~ 0.0231	~~0.1095~~ 0.1097	9.624
	B	4.08	0.292	5.27	29.19	28.94	0.0096	0.1145	0.96
	C	4.09	0.317	5.63	28.22	28.12	0.0085	0.1099	0.78
	D	4.40	0.317	5.85	27.61	27.91	0.0085 ~~1.764~~	0.1131	0.84
NBS 99A →	E	4.46	0.034	11.19	26.86	26.17	1.764	0.0089	4.27
		4.52	0.035	11.40	27.36	26.65	1.797	0.007	4.35
Blank →	F	<0.1	<0.001	<0.005	<4.8	<4.8	.0197	0.0005	<0.2
E	G	4.46	0.259	3.47	28.45	28.10	.0070	0.1106	0.65
		4.38	0.271	3.41	27.94	27.59	.0069	0.1086	0.64
F	Reag. Blank *	4.38	0.330	6.27	31.70	31.55	0.0096	0.1100	0.6830
		4.34	0.317	6.02	30.42	30.28	0.0092	0.106	0.655
	F *	4.34	0.317	6.02	30.43	30.29	.0092	0.106	0.656

* at sample weight of 0.20 grams was used to calculate the Reagent Blank values.

X - value calculated using reagent blank w/ weight for Sample F.

fax: 522-5155

Date: 5/27/90 - 5/31/90 Signature: Becky Riddle

(Shift 17) Rev 1/89

Below are summaries of chemical analyses done to date on NBS standards A and E and clinoptilolite samples from Death Valley Junction, CA. Problems with Si and Al analyses still remain, as well as with Ca and Mg.

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 99A
SAMPLE A

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	4.99	3.9	1.25	0.002	0.043	11.3	31.8
CORE LAB	4.8	5.7	1.75	<0.02	0.0403	12.0	32.4
SWRI	4.89	3.9	1.35	0.008	0.049	8.78	0.17
	5.0	4.0	1.4	<0.05	0.06	10.9	5.0
							3.5
(3/28/90)	3.92	3.8	1.33		0.045	6.67	19.5
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	21.9
(4/2/90, 0.05g sample, 0.5g LiBO2)						18.2	16.1
(4/11/90, .02g sample, 0.5g LiBO2)						10.5	27.0
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						11.0	30.3
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							29.5
(5/3/90)	4.46	4.32	1.15	0.009	0.04	9.79	25.2
(5/11/90)	4.60	4.77	1.50		0.045	9.86	24.3
(5/14/90)			0.34	<0.0025			
(5/21/90;Si1)	4.64	4.21	1.29	0.0062	0.05	9.93	28.62
(5/21/90;Si2)							28.38
(5/25;100ml;Si1)	4.52	4.35	1.797	0.007	0.035	11.40	27.36
(5/25;100ml;Si2)							26.65
NBS	4.6	4.315	1.53	0.012	0.0455	10.85	30.48

RESULTS OF CHEMICAL ANALYSIS OF NBS STANDARD 70A
SAMPLE E

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
JORDAN LAB	2.1	9.61	0.076	0.003	0.051	9.6	32.5
CORE LAB	2.24	4.3	0.093	0.018	0.0784	9.1	28.8
SWRI	1.74	7.67	<0.12	0.0097	0.0531	5.34	0.10
(3/28/90)	1.93	9.33	<0.25		0.059	7.87	13.99
(4/2/90, 0.1g sample, 0.5g LiBO2)						9.3	20.7
(4/2/90, 0.05g sample, 0.5g LiBO2)						8.7	18.4
(4/11/90, 0.02g sample, 0.5g LiBO2)						6.1	26.1
(4/19/90, 0.2g sample, 1.2g LiBO2, Si1)						9.7	30.7
(4/19/90, 0.2g sample, 1.2g LiBO2, Si2)							32.3
(5/3/90)	1.78	9.46	<0.25	0.024	0.05	9.06	27.81
(5/11/90)	1.73	11.20	<0.25		0.07	9.86	28.52
(5/14/90)			0.024	0.0049			
(5/25;100ml;Si1)	1.86	9.62	0.024	0.0147	0.045	9.82	28.80
(5/25;100ml;Si2)							28.91
NBS	1.89	9.8	0.079	0.0	0.0525	9.474	31.37

RESULTS OF CHEMICAL ANALYSIS OF CLINOPTILOLITE, DEATH VALLEY JUNCTION

WEIGHT PERCENT

	NA	K	CA	MG	FE	AL	SI
CDV (5/4/90)	2.56	3.15	0.35	0.20	0.46	6.05	30.04
CDVP (5/4/90)	2.49	3.11	<0.25	0.17	0.34	5.90	30.89
CDVH1 (5/4/90)	4.05	1.66	<0.25	0.14	0.28	11.13	28.39
(5/11/90)	3.78	1.45	<0.25		0.33	5.83	25.61
(5/14/90)			0.017	0.056			
CDVH2 (5/4/90)	3.99	1.48	<0.25	0.13	0.31	5.82	30.29
(5/11/90)	3.85	1.58	<0.25		0.34	5.90	25.57
(5/14/90)			0.045	0.042			
CDVH3 (5/21;Si1)	5.35	1.23	0.050	0.146	0.38	7.4	36.95
(5/21;Si2)							35.9
(5/25;100ml;Si1)	4.08	0.96	0.009	0.1145	0.292	5.27	29.19
(5/25;100ml;Si2)							28.94
CDVH4 (5/21;Si1)	4.37	0.73	0.047	0.116	0.33	0.23	28.38
(5/21;Si2)							28.18
(5/25;100ml;Si1)	4.09	0.78	0.0085	0.110	0.317	5.63	28.22
(5/25;100ml;Si2)							28.12
CDVH5 (5/21;Si1)	4.43	0.67	0.047	0.115	0.34	5.39	30.13
(5/21;Si2)							29.31
(5/25;100ml;Si1)	4.40	0.84	0.0085	0.113	0.317	5.85	27.61
(5/25;100ml;Si2)							27.91
CDVH6							
(5/25;100ml;Si1)	4.38	0.64	0.0069	0.109	0.254	3.41	27.94
(5/25;100ml;Si2)							27.59
CDVH7							
(5/25;100ml;Si1)	4.34	0.66	0.0092	0.106	0.317	6.02	30.43
(5/25;100ml;Si2)							30.29

6/5/90 RDN

0900 hrs — Water level in bath found to be low; level is lower than bottom of plastic bottles. Bath was refilled w/ RO H_2O.

0900 hrs — Reweigh A1 and B1-B6

A1 = 32.6883
B1 = 30.9896
B2 = 31.2083
B3 = 32.3824
B4 = ~~32.5363~~ 32.2683 RDN 6/5/90
B5 = 32.5363
B6 = 32.4398

0930 hrs — Decant bottles A and B. Rinse 4X with dei H_2O, check for Cl^-: AgCl observed in both bottles. Rinse 2X, check for Cl^-: slight AgCl observed. Rinse once, replace dei H_2O, then place back in water bath at 70°C.

1330 hrs — Test of rock chemical analyses using NBS reference materials as standards

Objective: Check if matching matrix of standards and samples will give better ICP results, particularly for Si and Al.

Method: Use NBS 70A, 99A, 278, and 688 to generate calibration curves, analyze them as samples.

Paul Bertetti RDN 6/5/90

RDN 5 Jun 90 $LiBO_2$ lot # C 30702 BAKER

Sample #1 (NBS 70A)

crucible wt. 9.8460
(+ 1.2 / 11.0460) $LiBO_2$

crucible + $LiBO_2$ 11.0467
(+ 0.2 / 11.2467) sample NBS 70A

crucible + $LiBO_2$ + sample 11.2476
empty crucible 9.8457

Sample #2 (NBS 99A)

empty crucible 9.9502
$LiBO_2$ (+ 1.2 / 11.1502)

crucible + $LiBO_2$ 11.1503
sample (99A) (+ 0.2 / 11.3503)

crucible + $LiBO_2$ + (99A) 11.3509
empty crucible 9.9498

Sample #3 (NBS 278)

empty crucible 8.5251
$LiBO_2$ (+ 1.2 / 9.7251)

crucible + $LiBO_2$ 9.7251
sample 278 (+ 0.2 / 9.9251)

cruc. + $LiBO_2$ + sample ~~9.9247~~ 9.9251
empty crucible 8.5248

Sample # 4 (NBS 688)

crucible 9.7360
$LiBO_2$ (+ 1.2 / 10.9360)

crucible + $LiBO_2$ 10.9364
sample 688 (+ 0.2 / 11.1364)

cruc + $LiBO_2$ + sample 11.1369
empty cruc. 9.7358

sample #5 (blank)

crucible 9.8943
$LiBO_2$ (+ 1.2 / 11.0943)

cruc. + $LiBO_2$ 11.0943
9.8938

RM

Wt. of sample #1 (NBS 70A) = 0.2009 g
" " " #2 (NBS 99A) = 0.2006 g
" " " #3 (NBS 278) = 0.2000 g
" " " " 4 (NBS 688) = 0.2005 g
" " Blank ($LiBO_2$) = 1.2000 g

~~NBS 70A:~~ RM 6/5/90

~~Si ppm = 252.1~~
~~Al ppm = 76.13~~
~~Na ppm = 15.19~~ RM 6/5/90
~~K ppm =~~
~~Ca ppm =~~

The following are the concentrations of Si, Al, Na, K, Ca in the NBS standard solutions as calculated from:

$$ppm = wt\% \text{ element} \times \frac{\text{gram sample}}{200} \times 10^4$$

from certification provided by NBS

NBS 70A:
Si = 252.1 ppm
Al = 76.13 ppm
Na = 15.19 ppm
K = 78.75 ppm
Ca = 0.635 ppm

NBS 99A:
Si = 244.6 ppm
Al = 87.06 ppm
Na = 36.91 ppm
K = 34.5 ppm
Ca = 12.28 ppm

NBS 278:
Si = 273.2
Al = 59.92
Na = 28.72
K = 27.8
Ca = 5.624

NBS 688: Si = 181.5 ppm
Al = 73.7 ppm
Na = 12.75 ppm
K = 1.24 ppm
not certified (Ca = 69.8 ppm)

Samples 1 to 4, which are NBS reference materials, and the blank sample #5 will be fused and diluted to 250 ml. The 250 ml solutions of 1, 2, 3 and 4 will be split into two 125 ml solutions each, and will be transferred to 125 ml plastic bottles. One set will be labeled with NBS #'s, together with corresponding concentrations of Si, Al, Na, K, and Ca. The other set will be used as samples.

The calibration curves for each element will be generated using the following NBS materials:

Si: NBS 688 = 181.5 ppm
278 = 273.2 ppm

Al: NBS 278 = 59.2 ppm
99A = 87.6 ppm

Na: NBS 688 = 12.75 ppm
99A = 36.9 ppm

K: NBS 688 = 1.24 ppm
70A = 78.75 ppm

Ca: NBS 70A = 0.635 ppm
99A = 12.28 ppm

Transfer 125 ml of NBS 70A into plastic bottle labeled A; NBS 99A into bottle labeled B; NBS 278 into bottle labeled C; NBS 688 into bottle labeled D.

Give samples and standards to Becky Riddle (Div 01) for chem analyses.

1620 hrs – Check bottles A and B for Cl^-: AgCl observed in both. Rinse 2X. Replace dCH_2 and put back in water bath.

6/6/90 MW
0945 hrs – Check bottles A and B for Cl^-: AgCl observed in both. Rinse 2X. Replace dCH_2; put back in water bath at 70°C.

1000 hrs – Check bottles A and B for Cl^-: AgCl was not observed. Rinse 2X. Decant powder into separate 100 ml beakers, and place in drying oven.

1615 hrs – Dave B. reported that the conductivity meter for the nanopure water (deionized) system read 15.9 while Walt M. was filling up a large plastic jug.

Need to check if exchange columns are out and need to be replaced. Paul B also refilled the salt tank for the water softener with a fresh bag of salt.

6/7/90 MJP
0930 hrs — Take samples A & B from the drying oven. Cool the beakers with foil, let cool in desiccator for about 1/2 hr. Then weigh.
A = 85.1498 g
B = 73.0241 g
Remove foil covers, place in desiccator over saturated NaCl soln. Need to monitor weight over time.

1630 hrs — Reweigh A and B.
A = 85.3388 grams
B = 73.1934 grams

6/8/90 MJP
0930 hrs — Reweigh A and B
A = 85.8556 g (7150) MJP 6/8/90
B = 73.6683 g (5442)

1640 hrs — Reweigh A and B
A = 85.8556 g
B = 73.6683 g

6/9/90 MJP
1935 hrs — Reweigh A & B
A = 86.2815 g
B = 73.9347 g

6/10/90 MJP
2000 hrs = Reweigh A & B
A = 86.5388
B = 74.0336

6/11/90 MJP
0935 hrs — Reweigh A & B
A = 86.6316 g
B = 74.0596 g

1600 hrs = Reweigh A & B
A = 86.6671 g
B = 74.0697 g

6/12/90 MJP
0930 hrs — Reweigh A & B
A = 86.7430
B = 74.0957

6/12/90 MJP
1700 hrs — Reweigh A & B
A = 86.7640
B = 74.0974

Below is a copy of the chemical analysis for the NBS standards, using the same solutions as standards for the ICP/AA analyses (except for Mg and Fe). Calibration lines were generated for Si, Al, Na, K and Ca using the NBS reference materials specified on page GC-02-78. Mg was analyzed using flame AA. ~~Al appears too high in two samples.~~ Si is [illegible]

JUN 12 '90 11:36 SWR 5125223649 3840 P.2

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Parlan w/ NBS stnds (6/5/90)

Sampleid	Si	Mg*	Al	Fe*	Ca	Na	K
A	249.2	0.110	81.7	0.46	0.7	14.6	81.9
B	261.5	<0.05	99.8	0.36	13.6	38.0	37.5
C	256.4	0.75	61.3	10.5	~~5.65~~ 5.7 MRC	27.8	26.7
D	176.8	40.0	76.0	~~23.75~~ 51.4 MRC	69.9	12.0	<1.0
blank	MRC <1.0	<0.05	~~<1.0~~ MRC	<0.1	—	—	—

NOTE: These are solution concentrations not corrected for sample weights or final volumes in preparation

* Regular cal stnds used - all other elements calibrated w/ NBS stnds provided.

— blank used in instrument calibration

Date: 6/12/90

Signature: Becky Riddle

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Sample	Element	Expected Conc.	Analytical Result
A (NBS 70A)	Na	1.89	1.82
	K	9.80	10.2
	Ca	0.079	0.087
	*Mg	—	0.014
	*Fe	~~9.47~~ 0.052 MRC 6/13/90	0.057
	Al	9.47	10.2
	Si	31.37	31.0
B (NBS 98A)	Na	4.60	4.74
	K	4.3	4.67
	Ca	1.53	1.69
	*Mg	0.012	<0.006
	*Fe	0.045	0.045
	Al	10.85	12.44
	Si	30.48	32.63
C (NBS 278)	Na	3.59	3.48
	K	3.45	3.34
	Ca	0.703	0.713
	*Mg	(0.139)	(0.094)
	*Fe	~~2.49~~ 1.43 MRC 6/13/90	1.31
	Al	7.49	7.66
	Si	31.15	32.05
D (NBS 688)	Na	1.59	1.50
	K	0.155* below lowest stand. conc.	<0.12
	Ca	(8.70)	(8.09)
	*Mg	(5.07)	(4.99)
	*Fe	~~13.18~~ 7.24 MRC 6/13/90	6.41
	Al	9.19	9.48
	Si	22.63	21.97

* Analyzed using regular standards | (—) = NBS certificate only states for information only

Results above show relatively good results overall. Na is within 6% or less, due to tight range of NBS standard concentrations (13–37 ppm). K is within 3–8%, except for sample D w/c is below the lowest standard concentration. Ca ranges from 7–12% deviation, and may be due to large range between high and low standards. Mg does not look very good, may be due to flame AA method used. Fe is excellent for the two low concentrations (4.3%) samples, but are ~~about~~ ~~lower than~~ the true value at the high conc. end. Al appears too ~~high~~ in two samples. Si is better than in previous analyses, and are within ±7%.

Procedure of using rock standards appears to be promising. Need to generate a procedure using at least 4–5 standards for more accurate analyses of rock and mineral samples.

6/13/90 MDD
0925 hrs — Reweigh A & B
A = 86.8089 g
B = 74.1121 g

1620 hrs = Reweigh A & B

A = 86.8205 g
B = 74.1131 g

6/14/90 MDD
1320 — Reweigh A & B
A = 86.8529 g
B = 74.1220 g

Reweigh A1 and B1 to B6
A1 = 32.1876
B1 = 32.8805
B2 = 31.4159
B3 = 32.3826
B4 = 32.2680
B5 = 32.5378
B6 = 32.4387

6/18/90 MDD
1525 hrs — Reweigh A & B
A = 86.8776 g.
B = 74.1398

6/20/90 MDD
1620 hrs = Reweigh A & B
A = 86.8245
B = 74.1385

1630 hrs = Reweigh A1 and B1 – B6

A1 = 32.1911
B1 = 30.8896
B2 = 31.4181
B3 = 32.3820
B4 = 32.2703
B5 = 32.5363
B6 = 32.4373

20 Jun 90 PB - XRD sample prep. (references)

Prepared monomineralic samples to be used as references in XRD analysis.

- 1-3 g sample crushed and ground in agate motar until all passed through ~~35~~ # 325 mesh sieve.

5% Al powder added (by weight), to the portion of each sample to be analyzed, as an internal standard.

Al powder from Mallinckrodt lot # 3116 KEBZ
(estimated values in parentheses)

1. Halite, Michigan

wt sample + vial 5.6908
− wt. vial 5.1675 5.6908
Sample wt 0.5233 x (.05) = 0.0262
(5.7170)

Final wt. Sample + vial + Al = 5.7171

2. Orthoclase (w/ Lepidolite inclusions)
Himalaya Mine, Mesa Grande
San Diego, Co., Calif.

Sample + vial 5.9824
vial 5.1682 5.9824
0.8142 x (.05) = 0.0407
(6.0231)

Sample + vial + Al = 6.0236

3. Analcime
Drain, Oregon

Sample + vial 5.8505
vial 5.0272 5.8505
0.7652 x (.05) = 0.0383
(5.8888)

Sample + vial + Al = 5.8885

4. Muscovite
South Dakota

Sample + vial 5.3405
vial 5.0853 5.3405
0.2552 x (.05) = 0.0128
(5.3533)

Sample + vial + Al = 5.3547

5. Augite
Oregon

Sample + vial 5.9254
vial 5.0724 5.9254
0.8530 x (.05) = 0.0427
(5.9681)

Sample + vial + Al = 5.9680

6. Phillipsite
Grant Co., Oregon

Sample + vial 5.7893
vial 5.1089 5.7893
0.6804 x (.05) = 0.0340
(5.8233)

Sample + vial + Al = 5.8233

7. Cristobalite
Glass Mountain, Calif

sample + vial 5.8675
vial 5.0667 5.8675
0.8008 x (.05) = 0.0400
(5.9075)

Sample + vial + Al = 5.9074

8. Magnetite
Nevada

Sample + vial 6.5059
vial 5.1086 6.5059
1.3973 x (0.05) = 0.0699
(6.5758)

Sample + vial + Al = 6.5759

9. Ilmenite
Wyoming

Sample + vial 7.3117
vial 5.1432 7.3117
2.1685 x (0.05) = 0.1084
(7.4201)

Sample + vial + Al = 7.4199

10. Montmorillonite
Montana

sample + vial 6.0336
vial 5.0563 — 6.0336
0.9773 x (.05) = 0.0489
(6.0825)

Sample + vial + Al = 6.0823

11. Sanidine
Daichi, Japan

sample + vial 6.1411
vial 5.1047 — 6.1411
1.0364 x (.05) = 0.0518
(6.1929)

Sample + vial + Al = 6.1946

12. Kaolinite
Montana

sample + vial 5.7667
vial 5.1046 — 5.7667
0.6621 x (.05) = 0.0331
(5.7998)

Sample + vial + Al = 5.7994

13. Anorthite
Yochi City, Japan

sample + vial 6.0922
vial 4.9389 — 6.0922
1.1533 x (.05) = 0.0577
(6.1499)

Sample + vial + Al = 6.1498

14. Opal
Queretaro, Mexico

Sample + vial 5.8810
vial 5.0887 — 5.8810
0.7923 x (.05) = 0.0396
(5.9206)

Sample + vial + Al = 5.9205

15. Sylvite
New Mexico

sample + vial 5.9804
vial 5.1002 — 5.9804
0.8802 x (.05) = 0.0440
(6.0244)

Sample + vial + Al = 6.0241

16. Chabazite
Beech Creek ~~Co., OR.~~
Grant County, Oregon

Sample + vial 5.7518
vial 5.0487 — 5.7518
0.7031 x (.05) = 0.0352
(5.7870)

Sample + vial + Al = 5.7866

17. Albite
Val Curnera,
Switzerland

Sample + vial 6.3638
vial 5.1483 — 6.3638
1.2155 x (.05) = 0.0608
(6.4246)

Sample + vial + Al = 6.4243

18 Calcite
Mexico

Sample + vial 6.1578
vial 5.0888 — 6.1578
1.0690 x (.05) = 0.0535
(6.2113)

Sample + vial + Al = 6.2116

19. Gypsum
Utah

Sample + vial 5.8681
vial 5.1127 — 5.8681
0.7554 x (.05) = 0.0378
(5.9059)

Sample + vial + Al 5.9055

X-RD samples taken to Div Ob for analysis.

21 Jun 90 PB - Ion Exchange Kinetic Experiment
Na-K and Na-Ca.

0745 A. Solution Preparation

- solutions required for experiment:
  0.01 M, 0.05 M, 0.1 M KCl
  0.02 M, 0.05 M, 0.1 M $CaCl_2$

- prepared 0.05 M KCl and 0.01 M KCl by series dilution of 0.1 M KCl standard. 200 ml of each was prepared and stored in 250 ml poly bottles.

- prepared 0.02 M $CaCl_2$ and 0.05 M $CaCl_2$ by series dilution of 0.1 M $CaCl_2$ standard. 200 ml of each was prepared and stored in 250 ml poly bottles.

KCl - Fisher lot # 885967
$CaCl_2$ - Corning 0.1 M Ca^{++} standard # 0540613

B. Experiment Setup and Method.

1. 0.2 g of Na-clinoptilolite (Batch B) to be placed in a 60 ml poly bottle. 50 ml of each solution to be placed in the bottle, swirled, and placed in a water bath set at 25°C and 80 rpm.

- Sample weights

a. Sample + 0.1 M KCl

			est.
Sample + 60 ml bottle	9.6409		0.2
- 60 ml bottle	9.4399		9.4399
sample wt.		0.2010 g	(9.6399)

Ion exchange Kinetic Exp. (cont'd)

b. Sample + 0.05 M KCl

Sample + 60 ml bottle	9.6240		0.2
60 ml bottle	9.4232		9.4232
sample wt.		0.2008	(9.6232)

c. Sample + 0.01 M KCl

Sample + 60 ml bottle	9.6642		0.2
60 ml bottle	9.4623		9.4623
sample wt.		0.2019	(9.6623)

d. Sample + 0.1 M $CaCl_2$

Sample + 60 ml bottle	9.5739		0.2
60 ml bottle	9.3737		9.3737
sample wt.		0.2002	(9.5737)

e. Sample + 0.05 M $CaCl_2$

Sample + 60 ml bottle	9.7238		0.2
60 ml bottle	9.5226		9.5226
sample wt.		0.2012	(9.7226)

f. Sample + 0.02 $CaCl_2$

Sample + 60 ml bottle	9.6310		0.2
60 ml bottle	9.4302		9.4302
sample wt.		0.2008	(9.6302)

0945 - Solution addition

50 ml of each solution was added to samples using 50 ml volumetric pipet. As a reference, 50 ml of each solution was added to 60 ml poly bottles containing no clinoptilolite sample.

- Clinoptilolite Sample B used.

Ion Exchange Exp. (cont'd)

1000 C. Sampling ~~and~~ of solutions

1. each solution + sample and solution reference was placed in water bath at 80 rpm and 25°c.

2. At each sampling interval, 1 ml of the sample solutions will be taken, made up to 10 ml in a 10 ml volumetric flask (with ultrapure H_2O), and placed in a 15 ml plastic beaker. ISA for the ion to be analyzed will be added in a 1:10 ISA to solution ratio.

K^+ and Ca^{2+} from sample solutions will be analyzed at each interval.

Na^+ from each sample solution will be analyzed every other interval starting with the second interval.

Two reference solutions from each ion group will be analyzed every other interval.

Several samples will be taken at periodic intervals the first day, two samples will be taken the second, and one sample will be taken the third day. Subsequent sampling will be as required.

3. Sampling method

a. 1 ml of each sample is placed in a 10 ml vol flask using a 1 ml volumetric pipet. The solution is diluted to the mark with ultrapure H_2O from a poly. squeeze bottle. After transfer to a 15 ml plastic beaker, 1 ml of ISA is added using a 1 ml volumetric pipet.

Ion Exchange Exp. (cont'd)

b. The test meter is calibrated using standards designed to bracket the expected readings.
For KCl : 0.0005 M and 0.01 M standards are used.
For $CaCl_2$: 0.0005 M and 0.05 M standards are used.
These standards are made by diluting the 0.05 M and 0.01 M ~~standards~~ solutions used ~~for~~ in the experiment.
ISA at a 10:1 ratio is added to each standard.
20 ml of each standard are used and measured for ions in a ~~glass~~ plastic 50 ml bkr.

New standards are made from the stock solutions daily.

Because the 15 ml have limited space, the ~~bk~~ beakers are turned by hand to provide mixing during sample analysis. The calibration solutions are mixed using magnetic stir bars.

4. Sample readings

1200 a. Sample Interval #1

- Calibration values	Before	After
0.0005 M KCl	0.0005 M	0.00048 M
0.01 M KCl	0.01 M	0.01 M

1.) KCl

0.01 KCl + Samp. = 0.00097 M
0.05 KCl + Samp. = 0.0048 M
0.1 M KCl + Samp. = 0.0095 M

0.01 KCl reference = 0.00098 M
0.05 KCl reference = 0.0048 M

Ion Exch Exp (cont'd)

2.) Ca^{2+}

calibration values	Before	After
0.0005 m $CaCl_2$	0.0005 m	0.00048 m
0.05 m $CaCl_2$	0.05 m	0.05 m

0.02 m $CaCl_2$ + samp = 0.0019 m
0.05 m $CaCl_2$ + samp = 0.0047 m

0.1 $CaCl_2$ + samp = 0.011 m
0.02 $CaCl_2$ ref = 0.0019 m
0.05 $CaCl_2$ ref = 0.0048 m

1430 b. Sample Interval #2

1.) KCl

calibration values	Before	After
0.0005 m KCl	0.0005 m	0.00046 m
0.01 m KCl	0.01 m	0.01 m

0.01 m KCl + Samp = 0.00079 m
0.05 m KCl + samp = 0.0043 m
0.1 m KCl + Samp = 0.0085 m

2.) $CaCl_2$

calibration values:	Before	After
0.0005 m $CaCl_2$	0.0005 m	0.00046 m
0.05 m $CaCl_2$	0.05 m	0.053 m

0.02 $CaCl_2$ + Samp = 0.0018 m
0.05 $CaCl_2$ + Samp. = 0.0048 m
0.1 $CaCl_2$ + Samp = 0.0090 m

0.1 $CaCl_2$ reference = 0.0092 m

3.) Na^+

calibration values:	Before	After
blank	–	0.0000
~~0.0017 m Na^+~~ 0.0017 m Na^+	~~0.00017 m~~ 0.0017 m	~~0.00015 m~~ 0.0015 m
0.017 m Na^+	0.017 m	0.017 m

Ion Exch Exp (cont'd)

3.) cont'd (Na^+)

0.01 KCl + samp = 0.0032 m Na^+
0.05 KCl + samp = 0.0036 m Na^+
0.1 KCl + samp = 0.0034 m Na^+

0.02 $CaCl_2$ + samp = 0.0030 m Na^+
0.05 $CaCl_2$ + samp = 0.0034 m Na^+
0.1 $CaCl_2$ + samp = 0.0030 m Na^+

1630 c. Sample Interval #3

1.) KCl

calibration values:	Before	After
0.0005 m KCl	0.0005 m	~~0.0005 m~~ 0.000471
0.01 m KCl	0.01 m	0.01 m

0.01 KCl + Samp = 0.00078 m
0.05 KCl + Samp = 0.0041 m
0.1 KCl + Samp = 0.0088 m

0.01 KCl reference = 0.00085 m
0.05 KCl reference = 0.0044 m

2.) $CaCl_2$

calibration values:	Before	After
0.0005 m $CaCl_2$	0.0005 m	0.00047 m
0.05 m $CaCl_2$	0.05 m	0.045 m

0.02 $CaCl_2$ + Samp = 0.0021 m
0.05 $CaCl_2$ + Samp = 0.0045 m
0.1 $CaCl_2$ + Samp = 0.0089 m

0.02 $CaCl_2$ reference = 0.0019 m
0.1 $CaCl_2$ reference = 0.0091 m

Ion Exch Exp (cont'd)

22 Jun 90 PJ

0830 d. Sample Interval #4

1.) KCl

calibration values:	Before	After
0.0005 m KCl	0.0005m	0.00051 m
0.01 m KCl	0.01 m	0.0096 m

0.01 KCl + Samp : 0.00088 m
0.05 KCl + Samp : 0.0045 m
0.1 KCl + Samp : 0.0089 m

2.) $CaCl_2$

calibration values :	Before	After
0.0005 m $CaCl_2$	0.0005m	0.00046m
0.05m $CaCl_2$	0.05 m	0.046 m

0.02 $CaCl_2$ + Samp : 0.0017 m
0.05 $CaCl_2$ + Samp : 0.0038 m
0.1 $CaCl_2$ + Samp : 0.0077 m

0.1 $CaCl_2$ reference : 0.0082 m

1100 3.) Na^+

calibration values:	Before	After
0.00017 m Na^+	0.00017 m	0.00017 m
0.017 m Na^+	0.017 m	0.018 m

0.01 KCl + Samp : 0.0027 m Na^+ 0.02 $CaCl_2$ + Samp : 0.0030 m Na^+
0.05 KCl + Samp : 0.0027 m Na^+ 0.05 $CaCl_2$ + Samp : 0.0030 m Na^+
0.1 KCl + Samp : 0.0029 m Na^+ 0.1 $CaCl_2$ + Samp : 0.0028 m Na^+

Ion Exch Exp (cont'd)

1400 Ca^{2+} sensor was replaced due to slow ~~rep~~ response time for the other Ca^{2+} sensor module.

1500 e. Sample Interval #5

1.) KCl

calibration values:	Before	After
0.0005m KCl	0.0005m	0.00047m
0.01 m KCl	0.01m	0.0097m

0.01 KCl + Samp : 0.00077 m
0.05 KCl + Samp : 0.0044m
0.1 KCl + Samp : 0.0085 m

0.01 KCl reference : 0.00088m
0.1 KCl reference : 0.0086m

2. $CaCl_2$

calibration values :	Before	After
0.0005 m $CaCl_2$	0.0005m	0.00040 m
0.05 m $CaCl_2$	0.05 m	0.0048 m

0.02 $CaCl_2$ + Samp : 0.0012 m
0.05 $CaCl_2$ + Samp : 0.0036 m
0.1 $CaCl_2$ + Samp : 0.0080 m

0.02 $CaCl_2$ reference : 0.0016 m
0.1 $CaCl_2$ reference : 0.0090 m

Ion Exch Exp (cont'd)

23 Jun 90 PD

1315 f. Sample Interval #6

1.) KCl

calibration values:	Before	After
0.0005 m KCl	0.0005 m	0.00047 m
0.01 m KCl	0.01 m	0.0095 m

0.01 m KCl + samp = 0.00082 m
0.05 KCl + Samp = 0.0045 m
0.1 KCl + Samp = 0.0098 m

0.05 KCl reference = 0.0043 m
0.1 m KCl reference = 0.0092 m

2.) $CaCl_2$

calibration values	Before	After
0.0005 $CaCl_2$	0.0005 m	0.00047 m
0.05 m $CaCl_2$	0.05 m	0.053 m

0.02 $CaCl_2$ + Samp = 0.0013 m
0.05 $CaCl_2$ + Samp = 0.0047 m
0.1 $CaCl_2$ + Samp = 0.0095 m

0.02 $CaCl_2$ reference = 0.0015 m
0.1 $CaCl_2$ reference = 0.0098 m

1430 A test for ~~them~~ the measurement of the release of Na^+ by the clinoptilolite was begun.

- 0.2 g Sample B was added to a poly bottle (60 ml) mixed with 50 ml ultrapure H_2O and placed in the water bath at 25 °C and 80 rpm.

Ion Exch Exp (cont'd)

- Sample + 60 ml bottle 9.6655
  60 ml bottle 9.4657
  sample wt. 0.0198

- In addition, a blank 60 ml bottle containing 50 ml ultrapure water was also placed in the water bath.
- 50 ml H_2O added to ~~each~~ each bottle using 50 ml volumetric pipet.

1445 ISA Solution preparation

4 m KCl (for Ca^{2+}) and 5 m NaCl (for K^+) solutions were required for ISA.

1. 149.12 g of KCl was dissolved and made up to 500 ml in a volumetric flask.

KCl + 1600 ml bkr 372.80 KCl = Fisher lot # 885967
wt 600 ml bkr 223.67
wt KCl 149.13 g

2. 146.1 g of NaCl was dissolved and made up to 500 ml in a volumetric flask.

NaCl + 600 ml bkr 369.29 NaCl = Fisher lot # 901163
600 ml bkr 223.18
wt NaCl 146.11

3. Both solutions transferred to 500 ml poly bottles.

Ion Exch. Exp. (cont'd)

25 Jun 90 PB

1500 g.) Sample Interval #7

1.) KCl

calibration values:	Before	After
0.0005 m KCl	0.0005 m	0.0005 m
0.01 m KCl	0.01 m	0.0098 m

0.01 KCl + sample = 0.00084 m
0.05 KCl + samp = 0.0045 m
0.1 KCl + samp = 0.0090 m

2.) $CaCl_2$

calibration values:	Before	After
0.0005 m $CaCl_2$	0.0005 m	0.00053 m
0.01 m $CaCl_2$	0.01 m	0.01 m

0.02 $CaCl_2$ + samp = 0.0013 m
0.05 $CaCl_2$ + samp = 0.0034 m
0.1 $CaCl_2$ + samp = 0.0070 m

3.) Na^+

calibration values:	Before	After
0.0005 m Na^+	0.0005 m	0.00052 m
0.01 m Na^+	0.01 m	0.0094 m

0.01 KCl + samp = 0.00078 m
0.05 KCl + samp = 0.00083 m
0.1 KCl + samp = 0.00083 m

0.02 $CaCl_2$ + samp = 0.00079 m
0.05 $CaCl_2$ + samp = 0.00075 m
0.1 $CaCl_2$ + samp = 0.00077 m

samp + H_2O (only) = 0.00002 m
H_2O blank = —

Ion Exch. Exp (cont'd)

26 Jun 90 PB

1505 h. Sample Interval #8

1.) KCl

calibration values:	Before	After
0.0005 m KCl	0.0005 m	0.0005 m
0.01 m KCl	0.01 m	0.0098 m

0.01 KCl + sample = 0.00083
0.05 KCl + sample = 0.0045 m
0.1 KCl + sample = 0.0092 m

2.) $CaCl_2$

calibration values:	Before	After
0.0005 m $CaCl_2$	0.0005 m	0.0005 m
0.010 m $CaCl_2$	0.010 m	0.0098 m

0.02 $CaCl_2$ + samp = 0.0014 m
0.05 $CaCl_2$ + samp = 0.0035 m
0.1 $CaCl_2$ + samp = 0.0074 m

3.) Na^+

calibration values:	Before	After
0.0005 m NaCl	0.0005 m	0.0005 m
0.010 m NaCl	0.010 m	0.010 m

0.01 KCl + samp = 0.00084 m
0.05 KCl + samp = 0.00091 m
0.10 KCl + samp = 0.00094 m

0.02 $CaCl_2$ + samp = 0.00094 m
0.05 $CaCl_2$ + samp = 0.00088 m
0.10 $CaCl_2$ + samp = 0.00088 m

Ion Exch Exp. (cont'd)

27 Jun 90 RB

1530 i. Sample Interval #9

1.) KCl

Calibration values	After
0.0005 m KCl	0.00052 m
0.010 m KCl	0.0098 m

0.01 KCl + sample = 0.00078 m
0.05 KCl + sample = 0.0047 m
0.10 KCl + sample = 0.0085 m

0.01 KCl reference = 0.00098 m
0.10 KCl reference = 0.0098 m

2.) Ca^{2+}

Calibration values	After
0.0005 m $CaCl_2$	0.00051 m
0.010 m $CaCl_2$	0.010 m

0.02 $CaCl_2$ + Sample = 0.0013 m
0.05 $CaCl_2$ + Sample = 0.0038 m
0.10 $CaCl_2$ + sample = 0.0071 m

0.02 $CaCl_2$ reference = 0.0015 m
~~0.010~~ 0.10 $CaCl_2$ reference = 0.0090 m

Ion Exch Exp (Cont'd)

28 Jun 90 RB

- Test for ion exchange in a 5g sample of clinoptilolite.

- Mix 5g sample B with 50 ml of 0.01 M KCl in a 60 ml poly bottle. Place in water bath at 80 rpm and 25°C. Measure concentration of KCl at +2 and +4 hours.

60 ml bottle + sample	14.4450 g	6.
60 ml bottle	9.4440 g	9.4440
sample wt.	5.0010 g	(14.4440)

1210 Added 50 ml of 0.01 M KCl to 5g sample and mixed by shaking. (volumetric pipet used for liquid measure)

1215 Placed solution in water bath.

1430 Sample Interval #1

Calibration	After
0.0005 M KCl	0.00047 M
0.010 M KCl	0.0097 M

sample 1 = 0.00082 M
sample 2 = 0.00083 M
0.01 M reference = 0.00093 M

1630 Sample Interval #2

Calibration	After
0.0005 M KCl	0.0005 M
0.010 M KCl	0.0098 M

sample 1 = 0.00018 M
sample 2 = 0.00017 M
0.01 M reference = 0.00087 M

Ion Exch Exp (cont'd)

29 Jun 90 PB

0930 Continued readings for 0.2g sample solutions and 5g solution (1 only)

j. Sample Interval #9

1.) KCl

Calibration values	After
0.0005 m KCl	0.00047 m
0.010 m KCl	0.0098 m

0.01 m KCl + sample = 0.00081 m
0.05 m KCl + sample = 0.0043 m
0.1 m KCl + sample = 0.0088 m

* 5g test sample = 0.00009 m

2. $CaCl_2$

Calibration values	After
0.0005 m	0.00053 m
0.010 m	0.010 m

0.02 $CaCl_2$ + sample = 0.0015 m
0.05 $CaCl_2$ + sample = 0.0042 m
0.10 $CaCl_2$ + sample = 0.0092 m

3. Na^+

Calibration values	After
0.0005 m NaCl	0.0005 m
0.010 m NaCl	0.0098 m

0.01 KCl samp = 0.00072 m
0.05 KCl samp = 0.00080 m
0.1 KCl samp = 0.00083 m

0.02 $CaCl_2$ samp = 0.00090 m
0.05 $CaCl_2$ samp = 0.00090 m
0.1 $CaCl_2$ samp = 0.00088 m

SUMMARY OF POTASSIUM VALUES

	0.01K SAMP		0.05K SAMP		0.1K SAMP	
SAMPLE NO.	MOLARITY	PPM	MOLARITY	PPM	MOLARITY	PPM
1	0.0097	379.3	0.048	1876.9	0.095	3714.7
2	0.0079	308.9	0.043	1681.4	0.085	3323.7
3	0.0078	305.0	0.041	1603.2	0.088	3441.0
4	0.0088	344.1	0.045	1759.6	0.089	3480.1
5	0.0077	301.1	0.044	1720.5	0.085	3323.7
6	0.0082	320.6	0.045	1759.6	0.088	3441.0
7	0.0084	328.5	0.045	1759.6	0.090	3519.2
8	0.0083	324.5	0.045	1759.6	0.092	3597.4
9	0.0082	320.6	0.047	1837.8	0.085	3323.7
10	0.0081	316.7	0.043	1681.4	0.088	3441.0

SUMMARY OF CALCIUM VALUES

	0.02 Ca SAMP		0.05 Ca SAMP		0.1 Ca SAMP	
SAMPLE NO.	MOLARITY	PPM	MOLARITY	PPM	MOLARITY	PPM
1	0.019	761.5	0.047	1883.8	0.110	4408.8
2	0.018	721.4	0.048	1923.8	0.090	3607.2
3	0.021	841.7	0.045	1803.6	0.089	3567.1
4	0.017	681.4	0.038	1523.0	0.077	3086.2
5	0.012	481.0	0.036	1442.9	0.080	3206.4
6	0.013	521.0	0.047	1883.8	0.095	3807.6
7	0.013	521.0	0.034	1362.7	0.070	2805.6
8	0.014	561.1	0.035	1402.8	0.074	2965.9
9	0.013	521.0	0.038	1523.0	0.071	2845.7
10	0.015	601.2	0.045	1803.6	0.092	3687.4

7/5/90 RDP
1100 - Analytical Balances in geochem lab were calibrated by Texas Scales (a SwRI qualified vendor). A copy of the results are shown below:

005280

TEXAS SCALES

PAGE 1
DATE 7-5-90

CUSTOMER & ADDRESS:
1. SWRI
SAN ANTONIO TX
2. PLANT LOCATION
BLDG 57
3. AUTHORIZED BY:
Div 20

4. CALIBRATED BY:
CHRIS CHINNI
5. SIGNATURE & T.A.D. #:
1883
6. NAT. BUREAU OF STANDARDS NO:
WEIGHT CERTIFICATION NO:
A-3100

SERVICE REPORT FOR PREVENTIVE MAINTENANCE

MAKE OR MODEL	S/N	REF. WEIGHT	ACTUAL READING	ADJUSTED READING	CALIBRATION DUE	COMMENTS
METTLER AE240	I9237	100g	99.9988	100.0000	1.91	40g RANGE ACTS FLAKEY (VIBRATION)
METTLER PM4600	I20467	1KG	999.9	1000.0	1.91	CONVEYLOAD LINEARITY CHECKS OUT OK.
SARTORIUS 3808	39030006	20000g ~~30 Kg~~ CAL	20000.8 ~~3000~~ CAL	300000	1.91	

BB - Paul Bertetti

5 Jul 90

ION EXCHANGE KINETIC EXPERIMENT

A. Solution Preparation

- all solutions prepared from NaCl - FISHER LOT # 901163
  KCl - FISHER LOT # 885967
  $CaCl_2$ - FISHER LOT # 886027
- 0.1 m Na^+, Ca^{2+}, K^+ solutions prepared by weight and dilution to 500 ml poly bottles
- 0.02 K^+, 0.05 K^+, 0.01 Ca^{2+}, 0.025 Ca^{2+}, 0.05 Ca^{2+} solutions prepared by dilution of 0.1 m solutions.

B. Set up and Method.

1. 0.2 g clinoptilolite will be added to 50 ml of solutions (0.02 K^+, 0.05 K^+, 0.1 K^+, 0.01 Ca^{2+}, 0.025 Ca^{2+}, and 0.05 Ca^{2+}) in 60 ml poly bottles.

2. The solutions are placed in a water bath and analyzed at intervals using ISE's. Water bath temperature is set at 25°C, rpm at 80.

0830

SOLUTION TYPE & BOTTLE NO.	WEIGHT OF 60 ml BOTTLE + SAMPLE (g)	WT OF 60 ml BOTTLE (g)	WEIGHT OF SAMPLE (g) CLINOPTILOLITE B
#1 0.02 M KCl	9.6174	9.4173	0.2001
#3 0.05 M KCl	9.6311	9.4308	0.2003
#5 0.1 M KCl	9.6415	9.4411	0.2004
#7 0.01 M $CaCl_2$	9.6150	9.4150	0.2000
#9 0.025 M $CaCl_2$	9.6123	9.4121	0.2002
#11 0.05 M $CaCl_2$	9.7390	9.5393	0.1997

KINETIC EXP (CONT'D) - 5 Jul 90

0900 3. 0.2g sample mixed with solution and placed in water bath. References also placed in water bath

	BOTTLE NO.		BOTTLE NO.
0.02 M KCl + Samp	1	0.01 M $CaCl_2$ + Samp	7
0.02 M KCl ref	2	0.01 M $CaCl_2$ ref	8
0.05 M KCl + Samp	3	0.025 M $CaCl_2$ + Samp	9
0.05 M KCl ref	4	0.025 M $CaCl_2$ ref	10
0.1 M KCl + Samp	5	0.05 M $CaCl_2$ + Samp	11
0.1 M KCl ref	6	0.05 M $CaCl_2$ ref	12

C. Sampling Method and Intervals

1. At each interval, 1 ml of each solution will be placed in a 10 ml volumetric flask and diluted to volume with ultrapure H_2O.

2. Each sample will be analyzed for ~~K^+, Ca~~ the ion in solution using ion selective electrodes. All solutions containing samples of clinoptilolite will be analyzed for Na^+ as well. The 10 ml dilutions are placed in 15 ml plastic beakers and mixed with ISA at a 1:100 ratio (IAW Orion catalog instructions). The reading is taken while the solution is stirred using 3mm × 10mm stir bars (teflon coated).

3. Sampling intervals

- 3 samples will be taken on day 1, two day 2, and 1 per day afterwards

KINETIC EXPERIMENT DATA TABLE

5 Jul 90

	5 JULY			6 JULY	
SAMPLE NO. ~~SOLUTION TYPE~~	1100 PB (ppm) **	1330 PB (ppm)	1600 PB (ppm)	1000 PB (ppm)	1400 PB (ppm)
1 0.02 K^+ SAMP	57	45	40	43	40
2 0.02 K^+ REFERENCE	73	66	68	72	70
3 0.05 K^+ SAMP	170	140	140	150	150
4 0.05 K^+ REF	180	160	170	180	190
5 0.1 K^+ SAMP	340	340	320	330	330
6 0.1 K^+ REF	360	350	350	360	360
7 0.01 Ca^{2+} SAMP	35	35	33	28	26
8 0.01 Ca^{2+} REF	41	45	42	40	39
9 0.025 Ca^{2+} SAMP	110	95	92	88	85
10 0.025 Ca^{2+} REF	120	100	100	99	96
11 0.05 Ca^{2+} SAMP	190	180	180	180	160
12 0.05 Ca^{2+} REF	190	190	190	200	190
0.02 K^+ (Na^+)	11	* 15	* N/A	18	8.2
0.05 K^+ (Na^+)	18	17	↓	18	8.7
0.1 K^+ (Na^+)	20	20	↓	19	9.9
0.01 Ca^{2+} (Na^+)	NOT TAKEN	* N/A	↓	15	7.5 ?
0.025 Ca^{2+} (Na^+)	NOT TAKEN	↓	↓	15	8.1
0.05 Ca^{2+} (Na^+)	NOT TAKEN	↓	↓	15	9.5
CALIBRATION VALUES BEFORE READINGS / AFTER READINGS: 0.001 K^+ cal	39 / 37	39 / 33	39 / 39	39 / 39	39 / 37
0.02 K^+ cal	780 / 760	780 / 770	780 / 750	780 / 760	780 / 750
0.0005 Ca^{2+} cal	20 / 20	20 / 24	20 / 21	20 / 20	20 / 20
0.01 Ca^{2+} cal	400 / 410	400 / 400	400 / 410	400 / 390	400 / 410
10 ppm Na^+ cal	10 / 11	10 / 11	* N/A	10 / 8.8	10 / 8.2
100 ppm Na^+ cal	100 / 100	100 / 97	* N/A	100 / 94	100 / 110

* Na^+ readings very unstable w/o stir bar, samples not taken until stir bars available (6 July)

** Values are accurate to 2 decimal places only. Improper setting on ISE meter due to operator error. (includes all values in TABLE)

ION EXCHANGE KINETIC EXPERIMENT

7 July

SAMPLE # / TYPE OF SOLUTION	JULY 7 1500	JULY 9 1500
1 0.02 K^+ SAMP	44	44
2 0.02 K^+ REF	73	72
3 0.05 K^+ SAMP	150	140
4 0.05 K^+ REF	180	180
5 0.1 K^+ SAMP	320	320
6 0.1 K^+ REF	370	350
7 0.01 Ca^{2+} SAMP	27	28
8 0.01 Ca^{2+} REF	37	41
9 0.025 Ca^{2+} SAMP	85	87
10 0.025 Ca^{2+} REF	97	100
11 0.05 Ca^{2+} SAMP	170	170
12 0.05 Ca^{2+} REF	190	190
0.02 K^+ (Na^+)	17	17
0.05 K^+ (Na^+)	17	18
0.1 K^+ (Na^+)	18	18
0.01 Ca^{2+} (Na^+)	22	19
0.025 Ca^{2+} (Na^+)	18	19
0.05 Ca^{2+} (Na^+)	17	17
0.000 K^+ cal	39/38	39/37
0.02 K^+ cal	760/740	760/770
0.0005 Ca^{2+} cal	20/22	20/22
0.01 Ca^{2+} cal	400/410	400/390
10 ppm Na cal	10/9.8	10/9.4
100 ppm Na cal	100/97	100/97

10 July 90 Exchange Reversibility Test

- Solutions ~~for~~ from bottles w/ clinoptilolite samples were drained. The sample was then washed 3 times with ultra pure H_2O. Following washing, Na^+ solutions (50 ml) in concentrations equivalent to original K^+ or Ca^{2+} solutions were added to the poly bottle/samples.

BOT No.	PREVIOUS SOLUTION	Na^+ SOLUTION ADDED
1	0.02 M K^+	0.02 M Na^+
3	0.05 M K^+	0.05 M Na^+
5	0.1 M K^+	0.1 M Na^+
7	0.01 M Ca^{2+}	0.02 M Na^+
9	0.025 M Ca^{2+}	0.05 M Na^+
11	0.05 M Ca^{2+}	0.1 M Na^+

- bottles will be sampled after approx. 1 wk., then again after approx 2 weeks to determine readsorption of Na^+ if it occurs.

13 July 90 Due to inconsistencies in meter readings vs. expected solution concentrations, the solutions used in the kinetic experiment were measured using purchased standards from Orion.

K^+

cal standards 0.1 M K^+ (purchased) Lot # TT1 ~~7/18/90~~

0.01 M K^+ (diluted from purchased solution)

1200

EXP. SOLUTIONS & EXPECTED VALUES	METER READING
0.02 M K^+	0.0196 M
0.05 M K^+	0.0499 M
0.1 M K^+	0.0994 M

13 July 90 — Ca^{2+}

0.1 m Ca^{2+} (purchased from Corning) Lot # 540668

1200 — 0.01 m Ca^{2+} (diluted from purchased solution)

experiment solution &

expected value	meter reading
0.05 m Ca^{2+}	0.0562 m Ca^{2+}
0.025 m Ca^{2+}	0.0284 m Ca^{2+}
0.01 m Ca^{2+}	0.00993 m Ca^{2+}

13 July 90

1500 — Sample readings for K-Na reversal
Ca-Na reversal

Calibration values 2290 ppm (made up → 0.1 m Na^+)
1000 ppm (purchased)

bottle no (solution)	reading
(1) 0.02 K (0.02 Na)	372 ppm
(3) 0.05 K (0.05 Na)	1020 ppm
(5) 0.1 K (0.1 Na)	2140 ppm
(7) 0.01 Ca (0.02 Na)	366 ppm
(9) 0.025 Ca (0.05 Na)	1020 ppm
(11) 0.05 Ca (0.1 Na)	2150 ppm

- low values are below calibration range ⇒ meter recalibrated at 100 – 2290 ppm Na^+
100 ppm (purchased solution)

bottle no	reading
(1) 0.02 Na	409 ppm
(3) 0.05 Na	1090 ppm
(5) 0.1 Na	2240 ppm
(7) 0.02 Na	410 ppm
(9) 0.05 Na	1080 ppm
(11) 0.1 Na	[illegible]

readings of solutions varied significantly with a change in calibration range. In addition to the change, (nearly 100 ppm in some cases) the readings seemed to drift upwards. For example an initial ready reading of 2150 ppm would drift up to 2240 ppm in 3-4 min.

1600 — meter recalibrated using 100 ppm (purchased)
2300 ppm (made up)

- all solutions have ISA added in equal proportions
- Na^+ electrode rinsed with correct rinse (diluted ISA)

25 July 90 1400

bottle no.	reading	Na reading
(1) 0.02 Na	429 ppm	275 ppm
(3) 0.05 Na	1080 ppm	804 ppm
(5) 0.1 Na	2190 ppm	1750 ppm
(7) 0.02 Na	419 ppm	271 ppm
(9) 0.05 Na	1080 ppm	753 ppm
(11) 0.1 Na	2180 ppm	1730 ppm

note redings are consistent for same concentrations, but not consistent over time. Drift may call for probe reconditioning. Probe will be reconditioned.

- position of electrodes did not affect readings. meter read same value even when electrode (Na) touched plastic beaker.

7/16/90 - RDP

A summary of the results for Na-K and Na-Ca ion exchange from 7/5 to 7/9/90 are tabulated below:

SUMMARY OF DATA : ION EXCHANGE KINETIC EXPERIMENT

POTASSIUM SOLUTIONS (RESULTS IN PPM)

SAMPLE NO	ELAPSED TIME (Hr)	0.02K	0.02K REF	0.05K	0.05K REF	0.1K	0.1K REF
0*	0	780	780	1950	1950	3900	3900
1	2	570	730	1700	1800	3400	3600
2	5	450	660	1400	1600	3400	3500
3	7	400	680	1400	1700	3400	3500
4	25	430	720	1500	1800	3200	3600
5	29	400	700	1500	1900	3300	3600
6	54	440	730	1500	1800	3300	3700
7	102	440	720	1400	1800	3200	3500

$\bar{x} = 427 \pm 23$; $\bar{x} = 1467 \pm 58$; $\bar{x} = 3267 \pm 58$

$\bar{x} = 706 \pm 27$; $\bar{x} = 1771 \pm 95$; $\bar{x} = 3571 \pm 76$

* Values of K^+ concentration at zero time are based on calculations from measured weights of reagents and known dilution factors. Actual conc. measured by ISE are generally lower. No actual readings taken at zero time.

$\Delta[K^+]_{0.02K} = (706 \pm 27) - (427 \pm 23) = 279 \pm 34$

$\Delta[K^+]_{0.05K} = (1771 \pm 95) - (1467 \pm 58) = 304 \pm 111$

$\Delta[K^+]_{0.1K} = (3571 \pm 76) - (3267 \pm 58) = 304 \pm 96$

Expected $\Delta[K^+] = 266$ ppm to 312 ppm

assume CEC = (1.7 and 2.0) x 10^-3 equiv/gm

SUMMARY OF DATA : ION EXCHANGE KINETIC EXPERIMENT

CALCIUM SOLUTIONS (RESULTS IN PPM)

SAMPLE NO	ELAPSED TIME (Hr)	0.01Ca	0.01Ca REF	0.025Ca	0.025Ca REF	0.05Ca	0.05Ca REF
0*	0	400	400	1000	1000	2000	2000
1	2	350	410	1100	1200	1900	1900
2	5	350	450	950	1000	1800	1900
3	7	330	420	920	1000	1800	1900
4	25	280	400	880	990	1800	2000
5	29	250	390	850	960	1600	1900
6	54	270	370	850	970	1700	1900
7	102	280	410	870	1000	1700	1900

$\bar{x} = 267 \pm 15$; $\bar{x} = 857 \pm 12$; $\bar{x} = 1667 \pm 58$; $\bar{x} = 1900 \pm 32$

$\bar{x} = 407 \pm 25$; $\bar{x} = 1017 \pm 82$; $\bar{x} = 1914 \pm 38$

* Values of Ca^{2+} at zero time are based on reagent weights + dilution factors. Actual concentrations determined by ISE deviate from these calculated values. No actual readings taken at zero time.

$\Delta[Ca^{2+}]_{0.01Ca} = (407 \pm 25) - (267 \pm 15) = 140 \pm 29$

$\Delta[Ca^{2+}]_{0.025Ca} = (1017 \pm 82) - (857 \pm 12) = 160 \pm 83$

$\Delta[Ca^{2+}]_{0.05Ca} = (1914 \pm 38) - (1667 \pm 58) = 247 \pm 69$

$(1914 \pm 38) - (1900 \pm 32) = 214 \pm 80$

Expected $\Delta[Ca^{2+}] = 136$ ppm to 160 ppm & compare

assuming CEC is equal to $(1.7 \text{ to } 2.0) \times 10^{-3}$ equiv/gm

SUMMARY OF DATA : ION EXCHANGE KINETIC EXPERIMENT

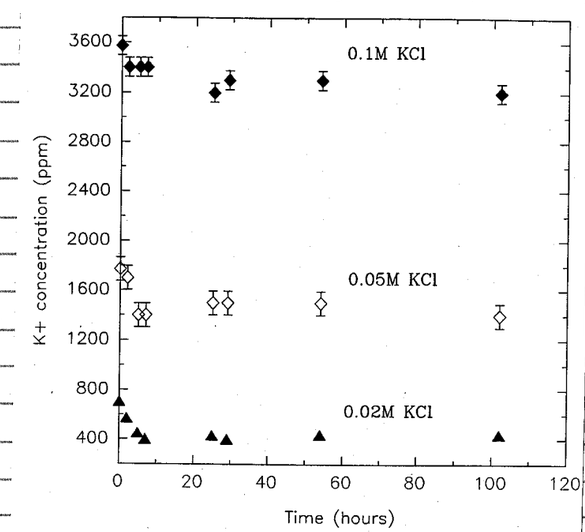
SAMPLE NO	ELAPSED TIME (Hr)	SODIUM IN SOLUTIONS (RESULTS IN PPM) (0 values indicate reading not taken) 0.02K	0.05K	0.1K	0.01Ca	0.025Ca	0.05Ca
0 *	0	0.0	0.0	0.0	0.0	0.0	0.0
1	2	110.0	160.0	200.0	0.0	0.0	0.0
2	5	150.0	170.0	200.0	0.0	0.0	0.0
3 *	7	0.0	0.0	0.0	0.0	0.0	0.0
4	25	180.0	180.0	190.0	150.0	150.0	150.0
5 #	29	82.0	87.0	99.0	75.0	81.0	95.0
6	54	170.0	170.0	180.0	220.0	180.0	170.0
7	102	170.0	180.0	180.0	190.0	190.0	170.0
X & S of ~~0,6,7~~		~~172 ± 6~~	177 ± 6	189 ± 6	235 ± 106	173 ± 21	163 ± 12

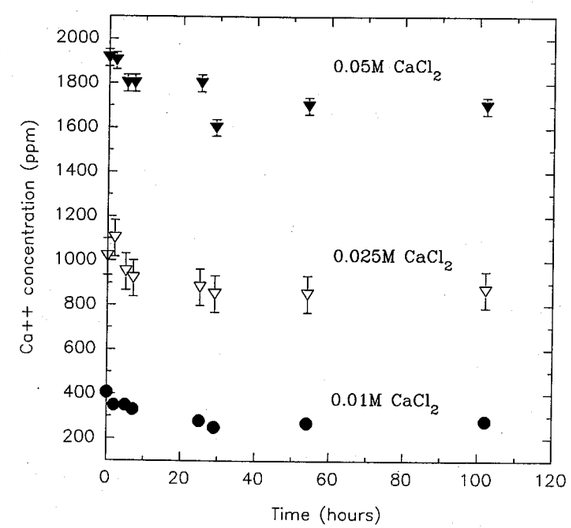
* No readings taken.

\# Possible erroneous measurements.

Expected equilibrium [Na+] = 156 to 189 ppm, assuming CEC of clinop. sample ranges from (1.7 to 2.0)×10⁻³ equiv/gm

Plotted below are the results for the ion exchange experiments from 7/5 to 7/9/90. The error bars are the standard deviations of the ISE analyses of the reference solutions (no zeolite phase present).





7/16/80 MJP

Ion exchange Kinetic Experiments:
Na-K and Na-Ca Systems

Objective: Determine the rate at w/c ion exchange equilibrium is achieved for Na-K and Na-Ca exchange.

Method: Experiments are conducted in a shaker water bath to maintain the temp at 25° ± 0.1°C, and to agitate the samples. A known amount of clinoptilolite (from Batch A, see p. GC-02-80) is placed in 60 ml polypropylene bottles which were previously washed with acid. Solutions of known composition (50 ml volume) are then added and allowed to react with the clinoptilolite.

Samples are withdrawn on a periodic basis for either ICP or ISE analysis. Samples for ICP analysis are diluted to either 50 ml or 100 ml with 10% HCl/2% HNO_3 solution. Samples for ISE analysis are diluted ~~to 10~~ MJP 7/21/80 to 10 ml from an initial 1 ml volume and transferred to screw-capped 15 ml polypropylene bottles for later analysis.

Below are detailed descriptions of the ten samples to be studied in the experiment. The calculated

expected (MJP 7/21/80)
Final concentrations of K, Na, and Ca, and the calculated expected concentration ranges for the ISE and ICP concentrations are based on the assumption that the cation exchange capacity of the clinoptilolite is 1.7×10^{-3} equiv/gm based on the work of Ames (1964). Other published papers suggest values of the CEC up to 2.2×10^{-3} eq/gm

Sample K1
0.5 g zeolite + 50 ml 0.02 M KCl

Initial: 782 ppm K^+; 0 ppm Na^+
Final: 117 ppm K^+; 390 ppm Na^+

ICP Dilution Factor: 5 ml/50 ml
ICP Conc. Range: 78.2 – 11.7 ppm K^+
0 – 39 ppm Na^+
ISE Conc. Range: 78.2 – 11.7 ppm K^+
0 – 39 ppm Na^+

Sample K2
1.0 g zeolite + 50 ml 0.05 M KCl

Initial: 1,955 ppm K^+; 0 ppm Na^+
Final: 625 ppm K^+; 782 ppm Na^+

ICP Dilution Factor: 2 ml/50 ml
ICP Conc. Range: 78.2 – 25.0 ppm K^+
0 – 31.3 ppm Na^+
ISE Conc Range: 195.5 – 62.5 ppm K^+
0 – 78.2 ppm Na

Sample K3
2.0 g zeolite + 50 ml 0.10 M KCl

Initial: 3,910 ppm K^+; 0 ppm Na^+
Final: 1,250 ppm K^+; 1,563 ppm Na^+

ICP Dilution Factor: 2 ml/100 ml
ICP Conc. Range: 78.2 – 25.0 ppm K^+
0 – 31.3 ppm Na^+
ISE Conc. Range: 391 – 125 ppm K^+
0 – 156.3 ppm Na^+

Sample K4
0.2 g zeolite + 50 ml [0.01 M KCl; 0.10 M NaCl]

Initial: 391 ppm K^+; 2,299 ppm Na^+
Final: 125 ppm K^+; 2,456 ppm Na^+

ICP Dilution Factor: 2 ml/50 ml
ICP Conc Range: 15.6 – 5.0 ppm K^+
91.86 – 98.2 ppm Na^+
ISE Conc. Range: 39.1 – 12.5 ppm K^+
230 – 246 ppm Na^+

Sample K5
0.5 g zeolite + 50 ml [0.025 M KCl; 0.10 M NaCl]

Initial: 978 ppm K^+; 2,299 ppm Na^+
Final: 313 ppm K^+; 2,689 ppm Na^+

ICP Dilution Factor: 5 ml/50 ml
ICP Conc. Range: 97.8 – 31.3 ppm K^+
229.9 – 268.9 ppm Na^+
ISE Conc. Range: 97.8 – 31.3 ppm K^+
229.9 – 268.9 ppm Na^+

Sample Ca1
0.5 g zeolite + 50 ml 0.01 M $CaCl_2$

Initial: 401 ppm Ca^{2+}; 0 ppm Na^+
Final: 61 ppm Ca^{2+}; 390 ppm Na^+

ICP Dilution Factor: 5 ml/50 ml
ICP Conc. Range: 40.1 – 6.1 ppm Ca^{2+}
0 – 39 ppm Na^+
ISE Conc. Range: 40.1 – 6.1 ppm Ca^{2+}
0 – 39 ppm Na^+

Sample Ca2
1.0 g zeolite + 50 ml 0.025 M $CaCl_2$

Initial: 1,002 ppm Ca^{2+}; 0 ppm Na^+
Final: 322 ppm Ca^{2+}; 782 ppm Na^+

ICP Dilution Factor: 5 ml/100 ml
ICP Conc. Range: 50.1 – 16.1 ppm Ca^{2+}
0 – 39.1 ppm Na^+
ISE Conc Range: 100.2 – 32.2 ppm Ca^{2+}
0 – 78.2 ppm Na^+

Sample Ca3
2.0 g zeolite + 50 ml 0.05 M $CaCl_2$

Initial: 2,004 ppm Ca^{2+}; 0 ppm Na^+
Final: 644 ppm Ca^{2+}; 1,563 ppm Na^+

ICP Dilution Factor: 5 ml/100 ml
ICP Conc. Range: 100.2 – 32.2 ppm Ca^{2+}
0 – 78.2 ppm Na^+
ISE Conc Range: 200.4 – 64.4 ppm Ca^{2+}
0 – 156.3 ppm Na^+

Sample Ca4
0.5 g zeolite + 50 ml [0.01 M $CaCl_2$ / 0.10 M NaCl]

Initial: 401 ppm Ca^{2+}; 2,289 ppm Na^+
Final: 61 ppm Ca^{2+}; 2,689 ppm Na^+

ICP Dilution Factor: 5 ml/50 ml
ICP Conc. Range: 40.1 – 6.1 ppm Ca^{2+}
229.9 – 268.9 ppm Na^+
ISE Conc. Range: 40.1 – 6.1 ppm Ca^{2+}
229.9 – 268.9 ppm Na^+

Sample Ca5
0.5 g zeolite + 50 ml [0.025 M $CaCl_2$ / 0.10 M NaCl]

Initial: 1,002 ppm Ca^{2+}; 2,289 ppm Na^+
Final: 662 ppm Ca^{2+}; 2689 ppm Na^+

ICP Dilution Factor: 5/100
ICP Conc. Range: 20.1 – 33.1 ppm Ca^{2+}
115 – 134 ppm Na^+
ISE Conc. Range: 100.2 – 66.2 ppm Ca^{2+}
229.9 – 268.9 ppm Na^+

The following are the expected concentrations of the reference solutions which are thermostatted at 25°C together with the samples. Periodic samples of these reference samples are taken for analysis to check their stability and to check the precision and reproducibility of the ICP ~~and ISE~~ analysis. To calculate these expected concentrations, the same dilution factors

as the corresponding samples are used:

ICP Reference Solution	ICP sample Expected Conc.
K1R	78.2 ppm K^+
K2R	78.2 ppm K^+
K3R	78.2 ppm K^+
K4R	15.6 ppm K^+; 92 ppm Na^+
K5R	97.8 ppm K^+; 230 ppm Na^+
Ca1R	40.1 ppm Ca^{2+}
Ca2R	50.1 ppm Ca^{2+}
Ca3R	100.2 ppm Ca^{2+}
Ca4R	40.1 ppm Ca; 230 ppm Na
Ca5R	50.1 ppm Ca; 115 ppm Na

7/17/90 Th 2P

Start ion exchange kinetic expts. Clinoptilolite powder are weighed into 60 ml plastic bottles. 50 ml of the solutions are added; the mixture is swirled, then placed in the shaker water bath set at 25°C and 80 rpm.

Sample	Time started
K1	0902
K2	0906
K3	0908
K4	0911
K5	0913
Ca1	0915
Ca2	0917
Ca3	0928 ~~0923~~ okay; repeated 10/7/90
Ca4	0923
Ca5	0926

Periodic samples for ICP analyses are taken using 2 ml or 5 ml volumetric pipettes and diluted with 10% HCl/2% HNO_3 to 50 or 100 ml using volumetric flasks. The solutions are then transferred to 60 ml ~~volumetric~~ NP 7/17/90 plastic bottles.

Samples for ICP Analyses

sample	7/17	7/17	7/17	7/17	7/17	7/17	7/18	7/18	7/20
K1	1008	1208	1305	—	1456	1630	0934	1611	—
K1R	—	—	—	1340	—	—	—	—	1400
K2	1010	1110	1307	—	1457	1631	0936	1612	—
K2R	—	—	—	1341	—	—	—	—	1401
K3	1011	1111	1308	—	1459	1631	0937	1612	—
K3R	—	—	—	1342	—	—	—	—	1402
K4	1012	1112	1309	—	1458	1632	0939	1613	—
K4R	—	—	—	1342	—	—	—	—	1403
K5	1013	1113	1310	—	1459	1632	0940	1614	—
K5R	—	—	—	1343	—	—	—	—	1404
Ca1	1014	1114	1311	—	1500	1633	0941	1615	—
Ca1R	—	—	—	1344	—	—	—	—	1405
Ca2	1015	1116	1312	—	1501	1634	0942	1615	—
Ca2R	—	—	—	1345	—	—	—	—	1406
Ca3	1016	1117	1313	—	1502	1635	0944	1616	—
Ca3R	—	—	—	1346	—	—	—	—	1407
Ca4	1017	1118	1314	—	1503	1636	0946	1617	—
Ca4R	—	—	—	1347	—	—	—	—	1408
Ca5	1018	1120	1315	—	1504	1637	0948	1619	—
Ca5R	—	—	—	1348	—	—	—	—	1409

* Sample taken on 7/17/1118, when diluted to 50 ml was overfilled by ~0.1 to ~0.2 ml.

1 ml samples are taken periodically for ion selective electrode analysis. These samples are diluted to 10 ml with ultrapure H_2O to have sufficient volume for ISE analysis. Orion ion selective electrodes and pH/ISE meters are used. Solutions are stirred with micro stir bars. 1 ml samples of the reference solns. are also taken to check the stability of the solutions as a function of time. The average time it takes to take the 10 samples is about 10 minutes.

Samples were taken from sample #'s K1 to K5 and Ca1 to Ca5 at 1100 hrs, 1400 hrs, and 1600 hrs. One set of samples so were taken from the reference solutions K1R to K5R and Ca1R to Ca5R at 1400 hrs.

7/18/90 RDP

Additional samples for ISE analysis were taken at 0900 and 1530 hrs. Samples for ICP were also taken (see p. GC-02-124)

7/19/90 RDP

One set of samples were taken at 1400 hrs. for ISE analysis. Not enough solutions left to get samples for ICP analysis. ISE analysis for Ca^{2+} initiated.

7/20/90 PJP

One set of samples from reference solutions (K1R to K5R, Ca1R to Ca5R) were taken for ICP analysis at 1400 hrs.

1410 hrs — One set of samples for ISE analysis was taken.

1430 hrs — Samples for ICP analysis, including blank solutions of 10% HCl & 2% HNO_3, were submitted to Div. 01 for analysis. Samples were received by Becky Riddle.

24 Jul / 25 Jul 90, PJP

Table of values from samples follows. Readings w/ ISE's on Orion 920 meters. Calibration solutions from purchased Orion standards Ca^{2+} Lot # - UY1 Na 1000 ppm - SY1

0.1m K^+ Lot # - TT1 Na 1000 ppm - TY1

21/25 JULY 90 PJP

All values in ppm (meter reading X 10)

DATE AND TIME OF SAMPLE / SOLUTION #	JUL 17	JUL 17	JUL 17	JUL 18	JUL 18	JUL 19	JUL 20	JUL 24	REFERENCES JUL 17	REFERENCES JUL 24
	1100	1400	1600	0900	1530	1400	1400	0800		
ELAPSED TIME	+2	+5	+7	+24	+30.5	+53	+77	+167	1100	1600
POTASSIUM K^+										
K1-ISE	326	184	142	88.3	84.8	77.9	83.4	91.2 / 86.7	704	698
K2-ISE	1080	773	670	526	514	454	457	434	1890	1820
K3-ISE	2060	1520	1380	975	933	916	911	920	3640	3700
K4-ISE	239	203	180	160	160	162	157	162	326	320
K5-ISE	538	380	341	314	315	318	323	320	887	894
CALCIUM Ca^{2+}										
Ca1-ISE	310 / 320 / 320	249 / 241 / 240	201 / 200 / 197	97.0 / 96 / 93.3	82.3 / 81.8 / 85.3	75.4 / 81.8	76.1	66.4	382 / 389	395
Ca2-ISE (25 JULY)	790 / 806 / 767	661 / 642	600 / 642	385 / 379	360 / 355	340 / 306	336	302	905 / 947	932
Ca3-ISE (24 JULY)	1410 / 1720 / (1800)	1460 / 1460 / 1430	1270 / 1290	910 / 902	846 / 852	754 / 725	729	690	1870 / 1880	1960 / 1940
Ca4-ISE (REVERIFICATION)	320 / 329 / 339	280 / 276	267 / 298	210 / 215	220 / 216	220 / 220	216	209	367 / 360	379
Ca5-ISE	850 / 845	814 / 832	807 / 805	750 / 726 / 725	770 / 719	740	746	713	855 / 855	940
Na (SODIUM)										
K1-ISE	*	208	326	347	349	*	379	⊛	NA	NA
K2-ISE	*	567	639	720	752 / 770	*	801	790	NA	NA
K3-ISE	*	1330	1400	1590	1620	*	1640	1660	NA	NA
K4-ISE	*	2160	2220	2220	2260	*	2290	2300	*	2100
K5-ISE	*	2460	2470	2520	2530	*	2610	⊛	*	2200
Ca1-ISE	89	164	218	303	327	375	421	398	NA	NA
Ca2-ISE	199	364	413	636	657	761	789	805	NA	NA
Ca3-ISE	297	524	694	1140	1230	1400	1420	1510	NA	NA
Ca4-ISE	2210	2150	2200	2290	2310	2390	2410	2430	2130	2150
Ca5-ISE	1760	1830	1830	1880	1940	2010	2060	2060	1700	1790

* - NOT TAKEN

⊛ - NOT TAKEN DUE TO LACK OF SOLUTION VOLUME

✓ - checked to within 2% agreement on subsequent readings

First readings taken were Ca^{2+} values on 24 July. An additional set (pencil) was taken 25 July. Values that differ substantially were reverified (red) 25 July. All other readings taken 25 July with random samples verified w/in 2% (pencil checks).

suspected bad value. Vol in bottle less than other samples.

Best values for Ca^{2+} are ① red followed by ② pencil followed by ③ ink. (values w/o multiple entries were taken/verified 25 July 90) PJP

7/27/90 PB

Resorption of Na^+

A. Preparation - Each sample was washed with ultrapure H_2O three times. Wash cycle included transfer of sample to 50 ml Nalgene centrifuge tube, ~~sample~~ centrifugation at ≈1500 rpm for 15 min with subsequent draining of H_2O. The tube was shaken vigorously following addition of H_2O to ensure adequate mixing occured. After washing, samples were transferred back to 50 ml poly bottles.

7/30/90 PB

B. Addition of Na^+ solutions - Varying concentrations of Na^+ and $Na^+ + K^+$, $Na^+ + Ca^{2+}$ solutions were added to the samples. Samples were then placed in the shaker-bath at 25°c and 80 rpm. A Table of solution concentration and start time follows.

(mixed solutions made by weight and diluted to volume in volumetric flasks) (~~All solutions diluted to 200 ml~~). 200 ml Na^+ only solutions diluted from purchased Orion standard (0.1 M NaCl) lot # TZ1

NaCl solid lot # Fisher 901163

KCl solid lot # Fisher 885967

$CaCl_2$ solid lot # Fisher 886027

Sample #	solution added	Time start
K1 - C1	0.02 NaCl (molar)	1258
K2 - C2	0.05 NaCl	1300
K3 - C3	0.1 NaCl	1250 (K3) 1255 (C3)
K4	0.01 NaCl + 0.1 KCl	1302
K5	0.025 NaCl + 0.1 KCl	1306
C4	0.02 NaCl + 0.1 $CaCl_2$	1310
C5	0.05 NaCl + 0.1 $CaCl_2$	1315

7/31/90 RJN

Below is a summary of the ICP analytical results on the ion exchange kinetic experiments (see p. GC-02-120). Copies of tabulated values provided by B. Riddle (Div. 01) follow. The values in the table below are corrected for dilution.

Date / Time / Δ time	7/17 1000	7/17 1100	7/17 1300	7/17 1500	7/17 1630	7/18 0930	7/18 1600	References 7/17 1340	7/20 1400
Sample Name	+1	+2	+4	+6	+7.5	+24.5	+31	+4.	+77
Potassium	7/31/90								
K1	455.1	360.6	287.9	182.6	155.6	115.9	122.1	744.5	762.6
K2	50.34	1,104	966.3	818.5	739.0	582.8	926	1941	1,935
K3	2,926	2,317	1,850	1,624	1,352	1,088	1,146	3,738	3,772
K4	342.5	311.0	287.5	268.8	271.5	256.3	270.3	362.5	356.6
K5	668.2	613.0	485.8	403.7	385.6	374.8	373.7	934.7	958.8
Calcium									
Ca1	331.3	308.1	278.3	229.7	196.2	97.6	83.2	348.0	375.7
Ca2	832	788.8	728.4	665.8	585.4	403.8	367.2	853.6	905.6
Ca3	1,561	1,563	1,441	1,320	1,190	840.4	765.4	1,631	1,721
Ca4	339	306	311.7	290.4	274.1	229.1	201.8	333.7	353.6
Ca5	884	865	855.4	836.2	812.6	710.2	703.0	910.4	890.4
Sodium									
K1	169.1	219.0	278.2	356.8	356.3	412.9	429.6	0	0
K2	342.3	434.5	501.5	652.3	674.5	781.0	815.3	0	0
K3	714.0	889.0	1,111	1,316	1,382	1,736	1,638	0	0
K4	2,259	2,210	2,248	2,222	2,231	2,359	2,421	2,177	2,243
K5	2,279	2,161	2,470	2,384	2,310	2,586	2,564	2,074	2,212
Ca1	32.9	58.6	102.0	151.6	206.7	325.6	355.7	0	0
Ca2	78.4	131.4	217.2	321.6	416.8	638.8	682.4	0	0
Ca3	140.2	224.4	362.4	563.2	731.8	1,165	1,316	0	0
Ca4	2,171	2,147	2,083	2,230	2,172	2,432	2,344	2,072	2,075
Ca5	2,234	2,185	2,178	2,286	2,292	2,353	2,415	2,136	2,203

The SS. are ICP results performed in Div. 01 and transmitted by fax. Originals are kept by B Riddle in Div. 01.

JUL 31 '90 15:26 SWR 5125223649 3848 P.1

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Date: 7/31/90 Employee No.: 8306

No. of Pages (including cover sheet): 8 Charge: 20-3704-013

TO: Bob Pabalan

OFFICE TELEPHONE: 522-5304

FACSIMILE TELEPHONE: 522-5155

FROM: Becky Riddle

TELEPHONE: 522-2181

NOTE: data summaries in ppm

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JUL 31 '90 15:26 SWR 5125223649 3848 P.2

20-3704-013

Jul26a	Na	µg/ml				µg/ml
ICV-546		49.65	38	CCB		-2.76
CCB		-2.73	39	K5-6		259.69
1-MET-BLK-1		-2.97	40	K1-7		48.96
K1-1		16.9	41	K2-7		32.61
K2-1		13.69	42	K3-7		22.76
K3-1		14.28	43	K4-7		96.82
K4-1		90.37	44	K5-7		258.35
K5-1		28.91	45	K1R-1		-2.71
K1-2		21.90	46	K2R-1		-2.98
K2-2		13.98	47	ICV-546		38.35
K3-2		17.78	48	CCB		-2.35
K4-2		84.39	49			
ICV-546		36.34	50			
CCB		2.76				
K5-2		216.68				
K1-3		29.87				
K2-3		20.06				
K3-3		22.27				
K4-3		89.91				
K5-3		246.95				
K1-4		35.66				
K2-4		26.09				
K3-4		26.32				
K4-4		88.66				
ICV-546		37.33				
CCB		-2.79				
K5-4		238.44				
K1-5		35.63				
K2-5		26.98				
K3-5		29.64				
K4-5		89.22				
K5-5		231.01				
~~K6-1~~ K1-6		41.29				
~~K6-2~~ K2-6		31.21				
~~K6-3~~ K3-6		34.71				
~~K6-4~~ K4-6		94.36				
ICV-546		41.31				

JUL 31 '90 15:27 SWR 5125223649 3848 P.3

20-3704-103
July 26, 1990

Jul26b	Na	µg/ml				µg/ml
ICV-546		37.04	38	CA1-5		20.67
CCB		-2.24	39	ICV-546		95.77
CAR-1		-2.89	40	CCB		-2.31
CA1R-1		87.06				
CA5R-1		207.39				
CA1R-2		-2.25				
CA2R-2		-2.26				
CA3R-2		-2.24				
CA4R-2		87.77				
CA5R-2		221.16				
BLK		-2.27				
CA1-1		3.29				
ICV-546		35.80				
CCB		-2.33				
CA2-1		3.92				
CA3-1		7.01				
CA4-1		217.18				
CA5-1		111.69				
CA1-2		5.86				
CA2-2		6.57				
CA3-2		11.22				
CA4-2		214.32				
CA5-2		109.24				
CA1-3		10.20				
ICV-546		33.84				
CCB		-2.33				
CA1-4		35.00				
CCB		-2.30				
CA2-3		10.86				
CA3-3		18.12				
CA4-3		208.30				
CA5-3		108.92				
CA1-4		15.16				
CA2-4		16.08				
CA3-4		28.16				
CA4-4		222.07				
CA5-4		114.78				

JUL 31 '90 15:27 SWR 5125223649 3848 P.4

20-3704-103
July 26, 1990

Jul26c	Na	µg/ml	µg/ml	
			Not a sample	
ICV-546		37.04	38.85	
CCB		-2.24	-2.17	
CA2-5		-2.99	22.84	
CA3-5		87.06	36.59	
CA4-5		207.39	224.37	
CA5-5		-2.25	114.99	
CA1-6		-2.26	32.50	
CA2-6		-2.24	41.94	
CA3-6		87.77	58.25	
CA4-6		221.16	225.52	224.39
CA5-6		-2.27	117.64	
~~CA1-7~~ ICV-546			38.07	
CCB		NEG	-2.24	
CA1-7			33.57	
CA2-7			34.13	
CA3-7			66.76	
CA4-7			238.36	
CA5-7			120.74	
CA1R-1			-2.29	
CA2R-1			-2.44	
CA3R-1			-2.41	
CA4R-1			207.16	
CA5R-1			106.82	
ICV-546			36.67	
CCB			-2.18	
CA1R-2			-2.45	
CA2R-2			-2.28	
CA3R-2			-2.31	
CA4R-2			207.52	
CA5R-2			110.18	
ICV-546			37.35	
CCB			-2.20	

JUL 31 '90 15:28 SWR 5125223649 3848 P.5

20-3704-103
July 27, 1990

Jul27a	K	µg/ml	#			µg/ml
ICV-546		57.32	38	K4-6		10.25
CCB		0.79	39	K5-6		37.48
1-MET-BLK-1		0.15	40	K1-7		12.21
K1-1		45.57	41	K2-7		25.38
K2-1		50.28	42	K3-7		28.91
K3-1		50.52	43	K4-7		10.81
K4-1		13.90	44	K5-7		37.37
K5-1		66.82	45	K1R-1		76.45
K1-2		36.06	46	K2R-1		77.65
K2-2		44.16	47	ICV-546		62.24
~~K6-1~~ ICV 546		59.12	48	CCB		3.11
~~K6-2~~ CCB		1.91	49	Not a Sample		
K3-2		46.93				
K4-2		12.44				
K5-2		61.30				
K1-3		28.79				
K2-3		38.65				
K3-3		36.99				
K4-3		81.56				
K5-3		48.58				
K1-4		19.26				
K2-4		32.74				
ICV-546		61.77				
CCB		1.91				
K3-4		32.46				
K4-4		10.75				
K5-4		40.37				
K1-5		15.56				
K2-5		29.56				
K3-5		23.04				
K4-5		10.96				
K5-5		34.96				
K1-6		11.59				
K2-6		23.59				
ICV-546		38.86				
CCB		2.52				
K3-6		27.76				

JUL 31 '90 15:28 SWR 5125223649 3848 P.6

20-3704-103
July 27, 1990

Jul27b	K	µg/ml
ICV-546		56.16
CCB		-0.50
K3R-1		74.76
K4R-1		14.50
K5R-1		93.72
K1R-2		76.26
K2R-2		77.38
K3R-2		78.63
K4R-2		14.24
K5R-2		95.88
ICV-546		56.95
CCB		-0.70
NOT A SAMPLE		

JUL 31 '90 15:29 SWR 5125223649 3848 P.7

20-3704-013

Jul27a	Ca					µg/ml
ICV-546		29.30	38	CCB		-0.64
CCB		-0.69	39	CA5-6		35.51
MET BLK		-1.09	40	CA1-7		8.32
CA1-1		33.13	41	CA2-7		18.36
CA2-1		41.60	42	CA3-7		38.27
CA3-1		78.06	43	CA4-7		20.18
CA4-1		23.90	44	CA5-7		55.15
CA5-1		144.20	45	CA1R-1		29.80
CA1-2		30.81	46	CA2R-1		42.48
CA2-2		39.48	47	CA3R-1		81.53
CA3-2		78.14	48	CA4R-1		33.37
CA4-2		33.06	49	ICV 546		26.67
ICV-546		29.27	50	CCB		-0.7
CCB		-0.61				
CA5-2		43.25				
CA1-3		27.85				
CA2-3		36.92				
CA3-3		72.03				
CA4-3		31.11				
CA5-3		42.37				
CA1-4		22.97				
CA2-4		33.29				
CA3-4		66.00				
CA4-4		29.04				
ICV-546		30.19				
CCB		-0.67				
CA5-4		41.81				
CA1-5		19.62				
CA2-5		29.27				
CA3-5		59.62				
CA4-5		29.41				
CA5-5		40.63				
CA1-6		9.76				
CA2-6		20.19				
CA3-6		42.02				
CA4-6		22.31				
ICV 546		27.10				

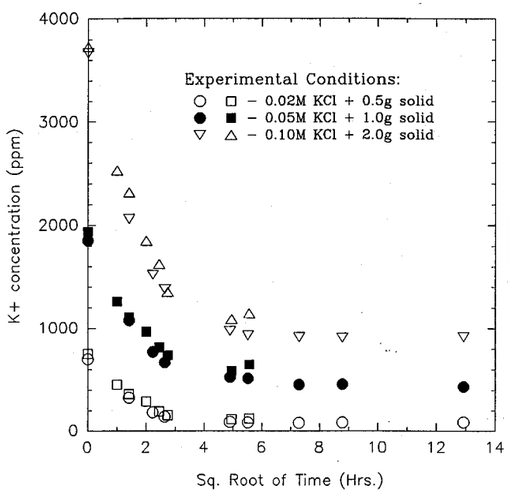
JUL 31 '90 15:29 SWR 5125223649 3848 P.8

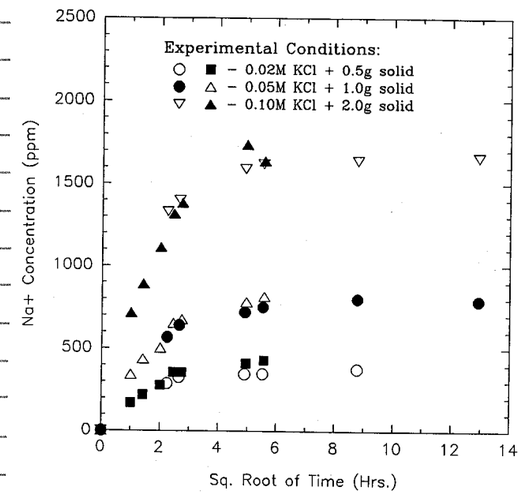
20-3704-013
July 27, 1990

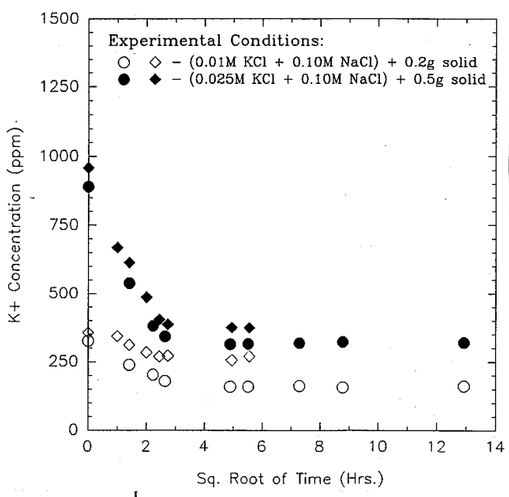
Jul27d	Ca	µg/ml
ICV-546		29.18
CCB		-0.93
CA5R-1		45.52
CA1R-2		37.57
~~CA2R-2~~ CA2R-2		95.28
~~CA3R-1~~ CA3R-2		86.00
~~CA4~~ CA4R-2		35.34
CA5R-2		49.52
ICV-546		26.79
CCB		-0.98

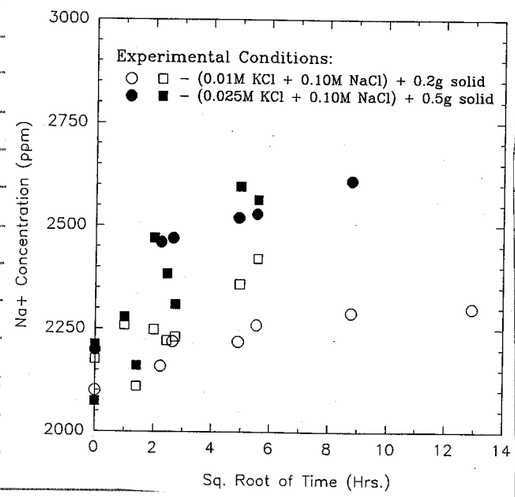
8/2/90 RDH

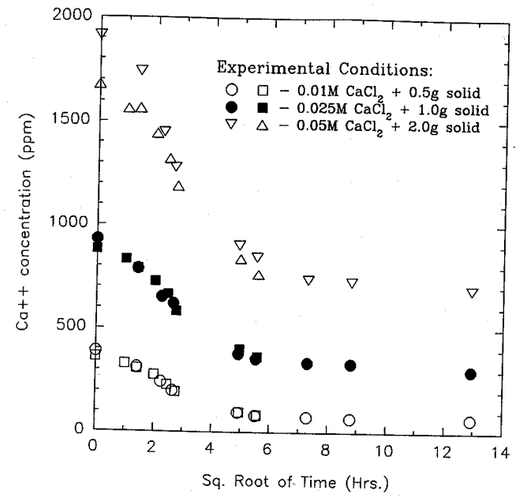
The following are plots of K^+, Ca^+, and Na^+ concentration as a function of time, based on results given in the tables on pages GC-02-127 and GC-02-129. Where more than one analysis was taken for a sample the average of the values are plotted.

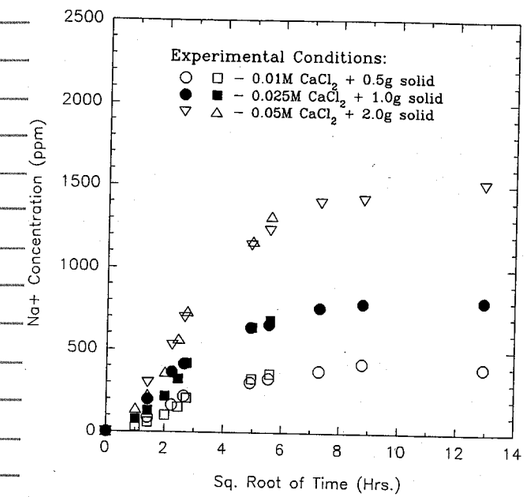


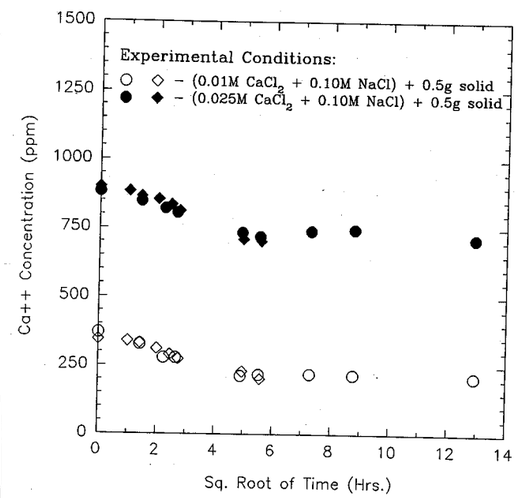


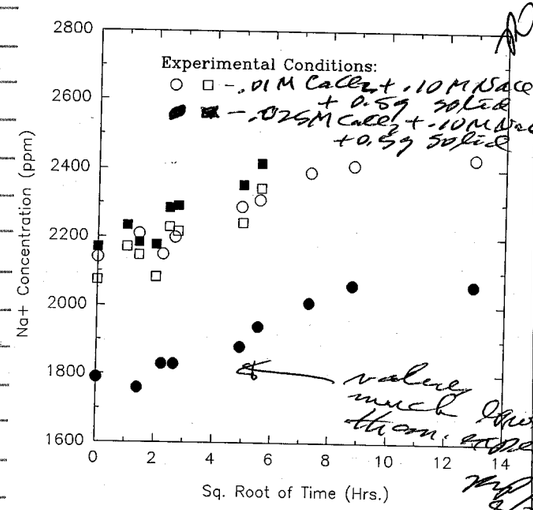












RDH 8/21/90

In the figures above, the first column of symbols are ~~experi~~ analytical data from ISE analysis, while the second column of symbols are based on ICP analysis. In general, the results from the two analytical techniques are in agreement within analytical uncertainty (±10% for

ICP. ± 2% for ISE). K^+ analytical results for the KCl–NaCl mixtures using ICP are higher than those for using ISE. Samples for these solutions will be reanalyzed by B Villaseñor of Div. 01. Na^+ analysis for the KCl solutions agree well for the two techniques. For the KCl–NaCl mixtures, ICP results for Na are fairly scattered, w/c is not unexpected due to the high concentrations of Na in solution. The ISE Na^+ results shown trends that mimick the results for K^+.

The ICP Ca^{2+} analyses are in good agreement w/ the ISE Ca^{2+} results, except for scattered points in the 0.05M $CaCl_2$ soln. Samples for this set will be reanalyzed for Ca^{2+} by ICP.

It appears that ICP analysis for the mixed KCl+NaCl and $CaCl_2$+NaCl are more uncertain than for the solutions w/c are initially pure KCl or pure $CaCl_2$. This could be due to matrix effects.

Comparisons of K^+ or Ca^{2+} sorbed into the solid with the Na^+ released into solution are tabulated below:

	Initial K^+ or Ca^{2+} conc.	Final K^+ or Ca^{2+} conc.	ΔK^+ or Ca^{2+}	ΔNa^+
0.02M KCl	0.0179 ±.0001	0.00216 ±.00013	0.0157 ±.0002	0.0156 ±.0008
0.05M KCl	0.0474 ±.0013	0.0119 ±.0009	0.0355 ±.0016	0.0340 ±.0011
0.10M KCl	0.0939 ±.0011	0.0235 ±.0002	0.0704 ±.0011	0.0713 ±.0009
0.01M KCl + 0.10M NaCl	0.00834 ~~±.00003~~	0.00410 ±.00006	0.00424 ±.00006	(0.0898 − 0.0913) = 0.00850
0.025M KCl + 0.10M NaCl	0.02279 ±.00013	0.00816 ±.00009	0.0146 ±.0002	(0.1118±.002 − 0.0957) = 0.0161 ±0.002
0.01M $CaCl_2$	0.00970 ±.00017	0.00175 ±.00012	0.00795 ±.00021	0.01731 ±.0010
0.025M $CaCl_2$	0.0231 ±.0005	0.00800 ±.00049	0.0151 ±0.0007	0.0340 ±.0011
0.05M $CaCl_2$	0.0477 ±.0011	0.0080 ±.0006	0.0397 ±.0012	0.0718 ±.0006
0.01M $CaCl_2$ + 0.10M NaCl	0.00919 ±.00024	0.00539 ±.00013	0.0038 ±.0003	(0.0933±.0006 − 0.0931±.0006) = 0.0122 ±.0006
0.025M $CaCl_2$ + 0.10M NaCl	0.0220 ±.0012	0.0181 ±.0004	0.0039 ±.0013	(.08888±.00126 − .07788) = 0.0110 ±0.0013

KA 8/26/90

8/10/90 RDP

Below is a summary of the reanalysis for K and Ca for samples K4, K5 and Ca1 (see p. GC-02-124) w/c gave initial results that disagreed with the ISE analysis (see p. GC-02-122 to GC-02-124). The original copy is kept by B Riddle (SwRI Div. 01) and xerox copies of the results transmitted by fax follows the table below. The values given in this table were recalculated to the original concentration using dilution factors used (see p. GC-02-119 to 123).

Date/Time and Sample Name	7/17 1000 +1	7/17 1100 +2	7/17 1300 +4	7/17 1500 +6	7/17 1630 +7.5	7/18 0930 +24.5	7/18 1600 +31
Potassium							
K4	260.3	220.3	216.3	190.0	182.3	168.5	157.3
K5	698.5	693.4	523.0	435.5	385.7	350.7	340.3
Calcium							
Ca1	326.1	315.5	272.9	232.7	189.2	98.8	78.7

Results for Ca1 are in good agreement with previous analysis. Ca3 should have been the samples analyzed, not Ca1. Request for reanalysis of Ca3+ to Ca3-7, and Ca3R-1 to Ca3R-2 was made to B Villarreal (SwRI Div 01). Will be performed next week.

AUG 07 '90 10:01 SWR 5.25223649 3840 P.1

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NOTE: results for K and Ca

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20-3704-013
July 05 1990

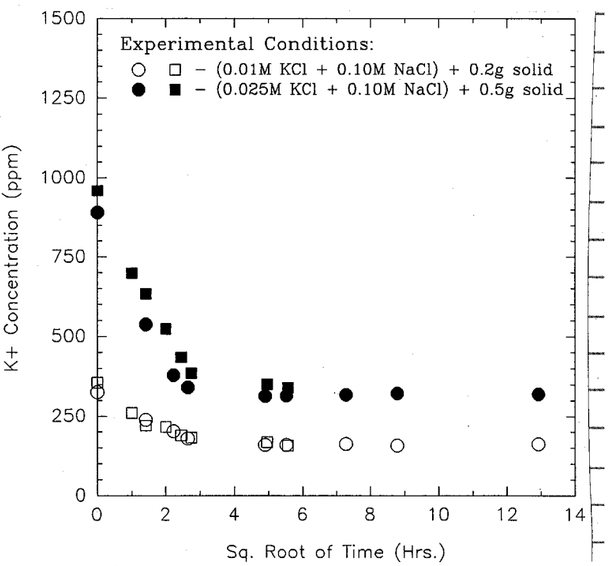
Jul03b	µg/ml
ICV-546	28.80
CCB	-0.52
CAI-1	32.61
CAI-2	31.55
CAI-3	27.29
CAI-4	23.27
CAI-5	18.92
CAI-6	9.68
ICV-546	28.53
CCB	-0.55
NO SAMPLE	—
NO SAMPLE	—
CAI-7	7.87
ICV-546	29.10
CCB	-0.60

AUG 07 '90 10:03 SWR 5.25223649 3840 P.3

20-3704-013
Aug 03, 1990

Aug03c	µg/ml
ICV-546	57.85
CCB	-1.39
K4-1	10.41
K4-2	8.81
K4-3	8.65
K4-4	7.60
K4-5	7.25
K4-6	6.74
K4-7	6.29
ICV-546	59.00
CCB	-0.62
K5-1	69.85
K5-2	63.34
K5-3	50.30
K5-4	48.55
K5-5	38.57
K5-6	35.07
K5-7	34.03
ICV-546	60.96
CCB	0.44

New results for K4 and K5 are in better agreement with ISE values than previous values. Comparison of new ICP values (□ and ■) with ISE results are shown on this figure →. ISE values are shown by ○ and ●. Compare figure on page GC-02-132 bottom left corner.



13 Aug 90 Paul Bertetti

Values for resorption experiment sample data

	Sample #	2 Aug / 1430	10 Aug / 0930	13 Aug / 0830
Samples read 900 - 1400 13 Aug	Ca1	26.8 / 27.6 26.0	29.7 / 30.4 29.2	26.6 / 29.1 26.2
	Ca2	95.3 / 98.8 93.7	105 / 109 100	103 / 105 101
	Ca3	200 / 211 200	221 / 226 226	226 / ~~232~~ 234 226
	Ca4	3760 / 3950 3850	3760 / 3850 3820	3950 / 3920 3950
	Ca5	3820 / 3950 3850	3700 / 3880 3820	3950 / 4010 ~~4010~~ 3980
Samples read 13 Aug 500 - 1030	K1	49.9 / 52.2 52.3	48.3 / 50.1 49.6	48.7 / 50.2 ~~52.6~~ 52.7
	K2	164 / 172 175	179 / 185 182	171 / 172 179
	K3	351 / 378 378	374 / 382 385	360 / 363 375
	K4	3710 / 3820 3710	3760 / 3840 3770	3700 / 3710 3780
	K5	3680 / 3710 3670	3610 / 3620 3620	3590 / 3610 3620
Samples read 14 Aug 945 - 0930	CaNa1	389 / 399 397	400 / 404 397	400 / 409 409
	CaNa2	992 / 1000 1000	980 / 978 980	~~975~~ 984 / 983 989
	CaNa3	2010 / 2040 2020	1970 / 2000 2000	1990 / 2030 2030
	CaNa4	616 / 629 626	638 / 651 640	632 / 648 637
	CaNa5	1190 / 1200 1210	1200 / 1220 1210	1220 / 1240 1230
	KNa1	373 / 390 391	363 / 377 381	① 367 / 382 386
	KNa2	908 / 935 940	908 / 939 940	901 / 931 938
	KNa3	1960 / 2020 2030	1950 / 2000 2020	1900 / 1960 1970
	KNa4	263 / 282 279	267 / 278 270	261 / 280 273
	KNa5	687 / 694 700	669 / 694 699	669 / 691 700

* numbers indicated are meter reading x 10, and are in ~~ppm~~

① meter drift on recalibration indicated there 1st values may be low

8/15/90 NP

Aqueous solutions of NaCl - CsCl at ionic strength of 0.1 N was prepared by P. Bertetti, by weight of reagent grade NaCl (Lot No. 901163) and CsCl (Lot No. 880480 ~~901163~~ ~~880480~~). Volume = 100 ml each.

	X_{Cs} = Mole fraction Cs $= \frac{m_{Cs}}{m_{Cs}+m_{Na}}$	Actual Wt. CsCl per 100 ml soln	Actual Wt NaCl per 100 ml soln
1	0	0	0.5847
2	0.01	0.01686	0.5780
3	0.025	0.04210	0.5705
4	0.050	0.08570	0.5556
5	0.075	0.1263	0.5402
6	0.10	0.1682	0.5261
7	0.20	0.3370	0.4677
8	0.30	0.5048	0.4094
9	0.40	0.6734	0.3507
10	0.50	0.8422	0.2920
11	0.60	1.0106	0.2338
12	0.70	1.1790	0.1751
13	0.80	1.3472	0.1170
14	0.90	1.5154	0.05871
15	0.925	1.5573	0.04389
16	0.950	1.5991	0.02928
17	0.975	1.6430	0.01471
18	0.99	1.6667	0.00587
19	1.00	1.6842	0

NaCl and CsCl reagent dissolved in ultrapure H_2O. Stored in 125 ml polyethylene bottles.

8/16/80 TWM

Prepare aq. solns. of NaCl-CsCl at ionic strength of 0.05 N, from the solutions prepared yesterday. Take 50 ml of I = 0.1 N soln, dilute to 100 ml with ultrapure water. Rinse 50 ml vol. pipette once with a few ml. of the solution before pipetting in the full 50 ml volume. Use 100 ml volumetric flasks.

Store the aq. solutions in 125 ml polyethylene bottles.

Calculations were done to determine the optimum solid/solution ratio for the isotherm experiments of (NaCl-CsCl) solution + zeolite exchanges. The criterion used is there should be enough change in aq. solution concentration such that the change could be resolved with ICP analysis of the solutions. The total concentrations must also be w/in the limits of ICP analysis, considering the dilution factors used in the sampling.

The following table gives the optimum solid weight to solution volume, assuming total exchange takes place. The actual amount exchanged will be less. The calculations assumed the cation exchange capacity per gram zeolite

is 1.7×10^{-3} equivs. Soln I = 0.05N.

X_{Cs}	Solid wt (g.) / Soln vol. (ml)	Cs ppm initial	Cs ppm final	% D
0	~~0.1/100~~	—	—	—
0.01	~~pH 8/17/80~~	—	—	—
✓ 0.025	~~0.05/500 0.1/100~~	166	[−60]	136
✓ 0.05	~~0.05/500 0.1/100~~	332	106	68
✓ 0.075	~~0.1/500 0.2/100~~	498	47	91
✓ 0.10	~~0.1/500 0.2/100~~	665	213	68
0.20	~~0.1/50 0.2/100~~	1329	877	34
0.30	0.2/25	1994	186	91
0.40	0.2/25	2658	851	68
0.50	0.2/25	3323	1515	54
0.60	0.2/25	3987	2180	45
✓ 0.70	0.2/10	4652	133	97
0.80	0.2/10	5316	788	85
✓ 0.90	0.2/10	5981	1462	76
✓ 0.925	0.2/10	6147	1628	74
0.95	0.2/10	6313	1794	72
✓ 0.975	0.2/10	6479	1960	70
0.99	0.2/10	6579	2060	69
✓ 1.0	0.2/10	6646	2127	68

8/17/80 TWM

1000 hrs – measure (tare) weights of clinoptilolite given in column (2) above, into polyethylene bottles. Use batch (A) of Na-enriched clinoptilolite which has been kept in a desiccator over sat'd NaCl solution (see pag. GC-02-80).

Wt of batch A (including beaker + foil cover) = 86.9441 g.
Compare with last measured value of 86.9245 g, measured on 6/20/91 (see page ~~EC-02-85~~).

X_{Cs}	Wt clinopt.
1.0	0.2008
0.975	0.2003
0.925	0.2016
0.90	0.2013
0.80	0.2029
0.70	0.2017
0.60	0.2019
0.50	0.2013
0.40	0.2013
0.30	~~0.2017~~ ~~0.1030~~ 0.2017
0.20	~~0.2043~~ 0.1012
0.10	~~0.2025~~ 0.1030
0.075	~~0.2106~~ 0.1008
0.050	~~0.1025~~ 0.0511
0.025	~~0.1102~~ 0.0501
Total	2.8449

Reweigh batch A clinoptilolite beaker = 84.0212. Compare (86.9441 − 2.8449) = 84.0992; loss in transfer = 0.078 g.

1440 hrs — Reweigh clinoptilolite for X_{Cs} = 0.2, 0.1, 0.075, 0.050, 0.025 using revised table (p. EC-02-141) solid/soln ratio.

Weight batch (A) clinopt (+ beaker + foil) = 84.0260 g. (compare 84.0212 on page 142)

Reweigh = 83.6020

1500 hrs — Pipet required volume of Na-Cs solutions into respective plastic bottles. Rinse pipet 10X with ultrapure H_2O between samples, and twice with a few mL of the solution.

1530 hrs — Place all plastic bottles in shaker water bath set at 80 rpm, 25°C. Turn on shaker.

8/20/92 Thu

0810 hrs — Check shaker water bath. Shaker running, but power interrupt light on. Temp reading = 25.2°C.

8/21/92 Fri

0810 hrs — Check shaker water bath. Running okay.

1530 hrs — Replace acid water bath w/ new batch RO water + HNO_3 to make 10% HNO_3 soln.

The following is a copy of the results of Ca analysis for samples Ca3-1 to Ca3-7, done by B Villa Emp (SwRI Div. 01).

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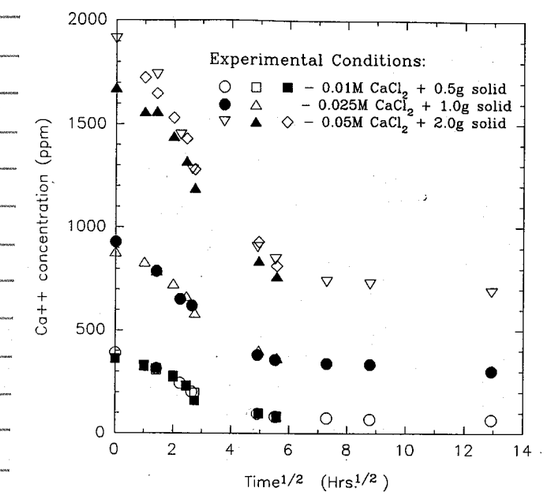
20-3704-103

Avg. 20 f	Ca µg/ml
ICV-Std	31.69
CCB	-6.56
CA3-1	86.23
CA3-2	82.46
CA3-3	76.59
CA3-4	71.54
CA3-5	63.98
CA3-6	46.54
CA3-7	40.74
CASR-1	50.89
CA2R-7	49.87
ICV-Std	31.21
CCB	-6.55

The above values, recalculated to the original solution concentration, are given below:

Ca3-1 = 1,725 ppm
Ca3-2 = 1,649
Ca3-3 = 1,532
Ca3-4 = 1,431
Ca3-5 = 1,280
Ca3-6 = 931
Ca3-7 = 815

The following is a plot of the Ca^{2+} concentration vs (time)$^{1/2}$ for samples Ca-1, Ca-2, and Ca-3. The new values given on page 144 are plotted as ◇ symbols. The previous ICP results for Ca-3 are shown as ▲. The recent results agree with the ISE analysis.



8/22/90 RDD
0830 hrs — The ~~ff.~~ are the dilution factors to be used in sampling the Na-Cs solutions from the isotherm experiments.

X_{Cs}	Dil. F. ICP	Dil. F. ISE	X_{Cs}	Dil. F. ICP	Dil. F. ISE
0.025				0.60	
0.050				0.70	
0.075				0.80	
0.10				0.90	
0.20				0.925	
0.30				0.975	
0.40				1.00	
0.50					

NaCl – CsCl (0.05 N)

X_{Cs}	~~ICP~~ Dil. F.	~~ESG~~ Dil. F.	X_{Cs}	~~ICP~~ Dil. F.	~~ESG~~ Dil. F.
0.025	10/100		0.60	1/100	
0.050	10/100		0.70	1/100	
0.075	10/100		0.80	1/100	
0.10	10/100		0.90	1/100	
0.20	5/100		0.925	1/100	
0.30	5/100		0.975	1/100	
0.40	1/100		1.00	1/100	
0.50	1/100				

The following are the expected concentrations of Na in the NaCl – CsCl solution before and after exchange. The values after exchange assume complete exchange with Cs, using a CEC value = 1.7×10^{-3} equiv/gm. The concentrations given below are Cs based on the initial and final ppm values on page GC-02-141. Dilution factors are not considered.

X_{Cs}	Na ppm initial	Na ppm final
.025	1,127	1,150
.05	1,092	~~1,150~~ 1,131
.075	1,063	~~1,150~~ 1,141
.1	1,035	~~1,150~~ 1,113
.2	920	~~1,150~~ 998
.3	805	1,118
.4	690	1,003
.5	575	888
.6	460	773
.7	345	1,127
.8	230	1,011
.9	115	897
.925	86	868
.975	29	811
1.0	0	782

Expected concentrations of sample solns. from NaCl – CsCl isotherm expts. using dilution factors on previous page:

Sample #	X_{Cs}	ICP Cs init	ICP Cs final	ICP Na init	ICP Na final
CS-1	.025	17	0	112	115
CS-2	.05	33	11	109	113
CS-3	.075	50	5	106	114
CS-4	.1	67	21	104	111
CS-5	.2	66	44	46	50
CS-6	.3	100	9	40	56
CS-7	.4	27	9	7	10
CS-8	.5	33	15	6	9
CS-9	.6	40	22	5	8
CS-10	.7	47	1.3	3.5	4
CS-11	.8	53	8	2.3	10
CS-12	.9	60	15	1.2	9
CS-13	.925	61	16	0.9	9
CS-14	.975	65	20	0.3	8
CS-15	1.0	66	21	0	8

1310 hrs – Samples of NaCl – CsCl solutions from isotherm expts. were taken. Pipettes used in taking the samples were rinse at least once with the sample and discarded. Dilution to required volume was done with 10% HCl + 2% HCl matrix.

Samples of the starting solutions were also taken, using the same dilution factors. Sample numbers are suffixed w/ letter R (for reference).

8/27/90 [illegible]

0900 hrs — Samples taken yesterday were submitted to B Riddle (SWRD Div. 06) together w/ Cs standard stock solution purchased from SPEX. Sample #'s CS-1 to -15, and reference sample #'s CS-1R to -15R.

1430 hrs — Prepare NaCl-SrCl mixtures; total normality = 0.5. Use $SrCl_2 \cdot 6H_2O$ reagent lot # 884315 and NaCl reagent lot # 901163. Mettler AE240.

X_{Sr} (0.5N)	g $SrCl_2 \cdot 6H_2O$ per 250 ml soln.	g NaCl per 250 ml soln
0		
0.01		
0.025	.4964	7.1239
0.05	.8338	6.9395
0.075	2489	6.7592
0.1	1.6664	6.5754
0.2	3.3376	5.8465
0.3	5.0022	5.1143
0.4	6.6658	4.3864
0.5	8.3323	3.6590
0.6	9.9986	2.9233
0.7	11.664	2.1949
0.8	13.331	1.4615
0.9	14.997	0.7309
0.925	15.413	0.5484
0.975	16.246	0.1833
1.0	16.663	0

8/28/90 [illegible]

NaCl – $SrCl_2$ (0.05 N)

The following table gives the optimum solid weight to solution volume ratios for the Na-Sr isotherm experiments, assuming total exchange takes place and a CEC of 1.7×10^{-3} equiv/gm.

X_{Cs}	Solid wt (g) / soln vol. (ml)	Sr ppm initial	Sr ppm final	% Δ
0.025	.05/100	54.8	17.5	68
0.05	.05/50	102.5	35.1	68
0.075	0.2/100	164	15	91
0.1	0.2/100	219	70	68
0.2	0.2/50	438	140	68
0.3	0.2/25	657	61	91
0.4	0.2/25	876	280	68
0.5	0.2/25	1095	489	54
0.6	0.2/25	1314	719	45
0.7	0.2/10	1533	213.8	87
0.8	0.2/10	1752	263	85
0.9	0.2/10	1971	482	76
0.925	0.2/10	2026	557	74
0.975	0.2/10	2138	646	70
1.0	0.2/10	2191	701	68

0930 hrs – Actual weights of Na-enriched clinoptilolite used:

X_{Sr}	Wt.	X_{Sr}	Wt	X_{Sr}	Wt
0.025	0.05042	0.4	.2000	0.925	.2000
0.05	0.05067	0.5	.2006	0.975	.2000
0.075	.1999	0.6	.2001	1.0	.1995
0.1	.2002	0.7	.1998		
0.2	.2005	0.8	.2005		
0.3	.2002	0.9	.2004		

XH 8/28/90

Wt of batch of clinoptilolite (+ beaker + foil cover) (See pages GC-02-142 and 143) = 83.6408 g. Compare with 83.6020 g, page GC-02-143.

Wt after taking aliquots for isotherm experiments = 80.9132. Δwt = 2.6888 g.; Compare with total wt. used for isotherm expt. = 2.7028 g. Difference = −0.0139 g. Could be due to water adsorbed during weighing? Humidity reading 25% RH.

1350 hrs — Completed putting clinoptil. powder and aqueous solutions into plastic bottles, as per solid wt. to soln ratios given on page 249. Samples bottles placed in shaker water bath, set at T = 25°C and 80 rpm.

8/29/90 XH

0830 hrs — Prepare NaCl–CsCl aqueous solns. at a total normality equal to 0.5 N for isotherm expts. The following table gives the weights of CsCl and NaCl reagents used (lot #s 820500 and 901163, resp.).

clinop 'A'

NaCl–CsCl (0.5 N)

Wt. of solid (g)	X_{Cs}	Wt CsCl (g)	Wt NaCl (g)	Vol. soln (ml)	Solid wt. to soln. vol. ratio
0.0501	0.01	0.04216	1.4461	50	0.05 g/25 ml
0.1002	0.025	0.10528	1.4246	50	0.1 g/25 ml
0.1005	0.05	0.21036	1.3882	50	0.1 g/10 ml
0.2002	0.075	0.3157	1.3514	50	0.2 g/10 ml
0.2004	0.1	0.4211	1.3145	50	0.2 g/10 ml
0.2498	0.2	0.8416	1.1687	50	0.25 g/5 ml
0.2505	0.3	1.2629	1.0225	50	0.25 g/5 ml
0.5003	0.4	0.8418	0.4383	25	0.5 g/5 ml
0.4997	0.5	1.0516	0.3652	25	0.5 g/5 ml
0.5002	0.6	1.2625	0.2922	25	0.5 g/5 ml
0.5002	0.7	1.4730	0.2191	25	0.5 g/5 ml
0.5004	0.8	1.6837	0.1462	25	0.5 g/5 ml
0.9999	0.9	1.8939	0.07315	25	0.5 g/5 ml
0.9999	0.95	1.9996	0.03658	25	0.5 g/5 ml
1.0004	1.0	2.1048	0	25	0.5 g/5 ml

(1.0) 8/29/90

Fisher lot # 896658A 8/28/90

Dilution factors to be used in sampling for ICP analysis and expected concentrations of Cs and Na assuming complete exch.

X_{Cs}	Dilution factor	Cs ppm initial	Cs ppm final	Na ppm initial	Na ppm final
0.01	1/100	66	21	114	115
0.025	1/100	83	38	560	568
0.05	1/100	33	15	109	113
0.075	1/100	50	4.7	106	114
0.1	1/100	66	21	103	111
0.2	1/250	53	8	37	45
0.3	1/250	80	35	32	40
0.4	0.5/250	53	31	9	22
0.5	0.5/250	66	21	11	19
0.6	0.5/250	80	35	9	17
0.7	0.5/250	93	48	7	15
0.8	0.5/500	53	31	2	6
0.9	0.5/500	60	15	1	9
0.95	0.5/500	63	18	0.6	8
1.0	0.5/500	66	21	0	8

Wt of Batch A clinop. after weighing out isotherm samples = 74.2218 g.

1400 hrs – Error in plastic bottles used for X_{Cs} = 0.01, 0.025, 0.95 and 1.0. Initially, 15 ml btls were used for X_{Cs} = 0.01 and 0.025, while 30 ml btls. were used for X_{Cs} = 0.95 and 1.0. The clinoptilolite powder in X_{Cs} = 0.01 bottle was discarded because some NaCl-CsCl solutions were already added. A new 0.01 X_{Cs} soln was prepared, also a new bottle (30 ml.) with 0.05 g clinop. The clinop. powder in the other bottles were transfered to correct bottles and the weights were made up to the correct amounts. Measured wt of Batch "A" = 74.1574

1615 hrs – Preparation of isotherm mixtures (NaCl + CsCl + clinop) completed. Plastic bottles placed in shaker water bath

8/30/90 RJP

The following gives the solid weight to solution volume to be used in the isotherm experiments for the mixture NaCl – SrCl2 (0.5 N). Also given are the dilution factors to be used in sampling for ICP analysis and the expected solution concentrations for initial and final conditions (assuming complete exchange w/ Na initially in the clinoptilolite occurs)

NaCl – SrCl2 (0.5 N)

X_{Cs}	Solid wt. to soln. volume ratio (wt. solid / ml soln used)	Dilution factor for ICP	Dilution factor for ICP	Expected Initial Cs ppm	Expected Final Cs ppm	Expected Initial Na ppm	Expected Final Na ppm	
0.01	0.1g/50 ml	0.1006	1/500	13.3	4.3			
0.025	0.2g/50 ml	0.2004	1/100	16.6	7.6			
0.05	0.2g/25 ml	0.2000	1/100	33	15			
0.075	0.2g/20 ml	0.2002	1/100	50	4.7			
0.1	0.2g/10 ml	0.1999	1/100	66	21			
0.2	0.25g/5 ml	0.2498	1/250	53	8			
0.3	0.25g/5 ml	0.2499	1/250	80	35			
0.4	0.25g/5 ml	0.2501	1/250	106	61			
0.5	0.5g/5 ml	0.4997	0.5/250	66	21			
0.6	0.5g/5 ml	0.5000	0.5/250	80	35			
0.7	0.5g/5 ml	0.5005	0.5/250	93	48			
0.8	0.5g/5 ml	0.5000	0.5/250	106	61			
0.9	1.0g/5 ml	1.0002	0.5/500	66	15			
0.95	1.0g/5 ml	0.9999	0.5/500	63	18			
1.0	1.0g/5 ml	1.0004	0.5/500	66	21			

see page

Most of the solutions required above have been prepared on 8/23/90 (see page GC-02-148), except for X_{Sr} = 0.01 and 0.95. These solutions were prepared by weighing

using reagent grade $SrCl_2 \cdot 6H_2O$ (Fisher, Lot # 884315) and NaCl (Fisher, Lot # 801163). The weights of the reagents used are:

X_{Cs}	wt $SrCl_2 \cdot 6H_2O$	wt NaCl
0.01	0.1667	7.2324
0.95	15.8310	0.3652

to make 250 ml volume solution.

1300 hrs — Clinoptilolite powder were tared into appropriate plastic bottles (See table on previous page for actual weights). Initial wt. of batch 'A' clinoptilolite (+ beaker + foil cover) = 74.1793 g.

Note: After taring in sample X_{Cs} = 0.7, beaker containing the clinop. powder broke, spilling contents on weighing table. Clinoptilolite powder was transferred into another beaker. Calculated amount of clinoptilolite powder lost ~ 0.3 g.

New wt. of batch 'A' clinopt. (+ beaker + foil cover) = 71.6297. Then samples X_{Cs} = 0.8, 0.9, 0.95 & 1.0 were tared into appropriate bttls.

Final wt. batch 'A' clinop. = 68.1188 g.

1500 hrs — Completed preparation of isotherm mixtures (clinop. + soln.) for the NaCl — $SrCl_2$ (0.5N) system.

Bottles were placed in shaker water bath, set at 25°C and 80 rpm.

The following table gives the dilution factors to be used in sampling the NaCl — $SrCl_2$ (0.05 total normality). Also given are the expected concentrations of Na and Sr in the diluted ICP samples w/c are calculated assuming complete exchange takes place with the Na initially in the zeolite. See page GC-02-149 for total expected solution concentrations.

NaCl — $SrCl_2$ (0.05 N)

X_{Sr}	Dilution Factor for ICP Analysis	Expected initial Sr ppm	Expected final Sr ppm	Expected initial Na ppm	Expected final Na ppm
0.025	25/100	13.7	4.4	280	285
0.05	25/100	27.4	8.8	273	283
0.075	25/100	41	3.8	266	285
0.1	25/100	55	17.5	259	278
0.2	10/100	44	14	92	103
0.3	10/100	66	6.1	80	111 ~~81~~
0.4	10/100	88	28	69	77 ~~70~~
0.5	5/100	55	25	29	44 ~~29~~
0.6	5/100	66	36	23	39 ~~23~~
0.7	5/100	77	2.2	17	56 ~~18~~
0.8	5/100	87	13	11	51 ~~12~~
0.9	5/100	99	24	6	45 ~~6~~
0.925	2/100	41	11	2	17 ~~2~~
0.975	2/100	43	13	0.6	16 ~~0.7~~
1.0	2/100	44	14	0	16 ~~0~~

The following tables give the dilution factors to be used in sampling the NaCl – $SrCl_2$ (0.5N) solutions for DSP analysis, and the expected concentrations of Na and Sr in the DSP samples. The concentrations are based on assuming complete exchange takes place between Sr in solution and Na in the solid. Dilution is done using a 10% HCl + 2% HNO_3 acid mixture.

NaCl – $SrCl_2$ (0.5N)

X_{Sr}	Dilution factors for DSP analysis	Expected Sr ppm initial	Expected Sr ppm final	Expected Na ppm initial	Expected Na ppm final
0.01	10/100	22	7	1138	1146
0.025	10/100	55	25	1121	1136
0.05	5/100	55	25	546	562
0.075	5/100	82	37	532	571
0.1	1/100	22	7	103	111
0.2	1/100	44	66	92	112
0.3	1/100	66	28	80	100
0.4	1/100	88	50	69	89
0.5	0.5/100	55	18	29	48
0.6	0.5/100	66	28	23	43
0.7	0.5/100	77	39	17	37
0.8	0.5/100	88	50	11	31
0.9	0.5/100	99	24	6	45
0.95	1/250	83	24	2	34
1.0	1/250	88	28	0	31

9/5/90 RJP

Aqueous mixtures of NaCl and KCl were prepared by weight from reagent grade chemicals (Fisher Lot #s 901163 and 885967, respectively. The following table gives the actual weights corresponding to the mole fraction of K in solution (total normality equals 0.5 N)

0.5N NaCl – KCl			NaCl – KCl (0.05N) Isotherm Expts.	
X	Wt. KCl per 250 ml	Wt NaCl per 250 ml	Solid wt. to solution vol. ratio	Actual wt. ratio
0.01	0.09319	7.2371	—	—
0.025	0.2329	7.1225	0.09g/100ml	.0500
0.05	0.4663	6.9396	0.1g/100ml	.1006
0.075	0.6994	6.7583	0.1g/100ml	.0999
0.1	0.9311	6.5749	0.1g/50ml	.1009
0.2	1.8638	5.8443	0.2g/50ml	.1998
0.3	2.7950	5.1141	0.2g/50ml	.2003
0.4	3.7285	4.3872	0.2g/25ml	.1998
0.5	4.6593	3.6531	0.2g/25ml	.1999
0.6	5.5914	2.9230	0.2g/25ml	.2001
0.7	6.5222	2.1905	0.2g/25ml	.1997
0.8	7.4559	1.4604	0.2g/10ml	.2000
0.9	8.3870	0.7307	0.2g/10ml	.2000
0.925	~~8.6208~~	0.5490	0.2g/10ml	.1999
0.95	8.8523	0.3659	~~0.2g/10ml~~	~~.2000~~
0.975	9.0878	0.1826	0.2g/10ml	.2000
1.0	9.3188	0	0.2g/10ml	.2001

The above table also gives the ratio of solid to liquid to be used in the experiment, and the measured weights of the clinoptilolite powder used in the expts, for total normality of 0.05 N.

The following table gives the dilution factors to be used in sampling the NaCl–KCl (0.05 N) isotherm soln. for analysis using ion-selective electrodes (ISE). Also given are the expected concentrations of K and Na, assuming total exchange occurs.

NaCl–KCl (0.05N)

X_K	Dilution Factors	Expected K ppm initial	Expected K ppm final	Expected Na ppm initial	Expected Na ppm final
0.025	GC-03-01 1:10 dilution 9/14/90	49	16	1,121	1,140
0.05		98	31	1,092	1,131
0.075		147	80	1,064	1,102
0.1		196	63	1,035	1,113
0.2		391	125	920	1,078
0.3		587	321	805	961
0.4		782	250	690	1,002
0.5		978	446	575	887
0.6		1173	641	460	772
0.7		1368	837	345	658
0.8		1564	235	230	1,012
0.9		1759	430	115	827
0.925	See GC-03-01	1808	479	86	868
0.975		1906	578	29	810
1.0		1955	626	0	782

The solutions used for the 0.05N isotherm expts. were prepared by diluting 25 ml of the 0.5N mixtures to 250 ml volume with ultrapure H_2O.

(9/7/90 MP
Experiments started on 9/6/90 1830hrs for NaCl–KCl (0.05N)
See page 160.)

The following table gives the ratio of solid to liquid to be used in isotherm expts. in the system NaCl–KCl (total normality 0.5N), and the actual weights of the clinoptilolite powders used. The solutions were prepared by weight as shown on the table by page 157.

NaCl–KCl (0.5N)

X_K	Solid weight to soln. vol. ratio	Wt. of clinop. powder
	0.01 .1 g/50 ml	0.1002 g
Clinop A_0 65.6430 g	0.025 .2 g/50 ml	0.2005 g
	0.05 .25 g/25 ml	0.2498 g
Clin A_f 60.0997 g	0.075 .2 g/10 ml	0.1995 g
	0.1 .2 g/10 ml	0.2001 g
	0.2 .25 g/5 ml	0.2500 g
	0.3 .25 g/5 ml	0.2502 g
	0.4 .5 g/5 ml	0.5003 g
	0.5 .5 g/5 ml	~~0.4998~~ 0.4999 g
	0.6 .5 g/5 ml	0.5004 g
	0.7 .5 g/5 ml	0.4997 g
	0.8 .5 g/5 ml	0.5001 g
	0.9 .5 g/5 ml	0.4999 g
	0.925 .5 g/5 ml	0.4996 g
	1.0 .5 g/5 ml	0.4996 g

The table on the next page gives the dilution factors to be used in sampling for ISE analysis of Na and K. Also given are the expected concentrations of Na and K assuming total exchange occurs between Na initially in the zeolite structure and K in soln.

NaCl-KCl (0.5N)

X_K	Dilution factors	Expected K ppm initial	Expected K ppm final	Expected Na ppm initial	Expected Na ppm final
0.01	See page 4-03-05 for actual dilution factors used in all	195	63	11,380	11,458
0.025		489	223	11,208	11,364
0.05		977	313	10,920	11,311
0.075		1,466	137	10,633	11,415
0.1		1,955	626	10,346	11,127
0.2		3,910	586	9,186	11,150
0.3		5,865	2541	8,047	10,001
0.4		7,820	1173	6,897	10,805
0.5		9,775	3128	5,748	9,656
0.6		11,729	5083	4,598	8,506
0.7		13,684	7038	3,449	7,357
0.8		15,639	8993	2,299	6,207
0.9		17,594	10,947	1,150	5058
0.95		18,572	11,925	575	4,483
1.0		19,549	12,902	0	3,908

9/6/90 MSP

1830 – Experimental isotherm mixtures using NaCl-KCl (0.05N) solutions were completed. Plastic bottles were placed in shaker water bath set at 25°C and 80 rpm.

Initial wt of batch 5 clinoptil. powder (+ beaker + cover) = 68.2139 g. Final wt = 65.6256 g. Δ wt = 2.5883 g. Compare Δ wt w/ total wt powder in mixtures = 2.5510. Difference = 0.0373 g.

9/7/90 MSP

1330 hrs – Completed mixing 0.5N NaCl-KCl solutions + clinoptilolite for isotherm expts. Actual wts of solids given on p. 159. Plastic bottles placed in shaker water bath set at 25°C & 80 rpm.