CENTER FOR NUCLEAR WASTE
REGULATORY ANALYSES

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9/11/00 PB — Copy made for QA archives.

3/7/01 AJ — copy made to page 53 for QA archives.

9/21/01 BAW — Copy made to page 123 for QA archives

BW 2-12-02

2/12/02

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2/12/02

AJ. Preparation of Clinoptilolite for Ion-Exchange Experiments

10/23/00

Clinoptilolite, CDV-2000 = 385 g. + 96 g.
= 481 g.

Materials Required and Equipments: AJ 2/15/02

① Clinoptilolite (Death Valley Junction, California)
② 8" diameter Stailess Steel Sieves [100, 60, 200 mesh]
③ RO-Tap Sieve Shaker
④ Ultrasonic Cleaner FS-28
⑤ Water Bath
⑥ Nano pure H_2O.
⑦ Graduated glass beakers (several sizes).
⑧ Magnetic stirrers.
⑨ Corning Stir/Hot Plate.
⑩ Centrifuge tubes (50 ml).
⑪ Glass & rubber tip stirrers.
⑫ Filter paper (FS glass fiber diameter porosity)
⑬ Glass funnels
⑭ Seperator funnels for density separation
⑮ Spatulas
⑯ Weighing balance (Mettler PM 4600 Delta range)
⑰ Microscope
⑱ Table top centrifuge (by Fischer Scientific Marathon 21K)
⑲ Drying oven (Stabil-Therm C-4850-A)
⑳ PH electrode
㉑ Plastic bottles to store solutions (several sizes)

(22) sodium bicarbonate, $NaHCO_3$ (FS, lot # 006275)
(23) sodium acetate trihydrate, $CH_3COONa \cdot 3H_2O$ (FS, lot # 937077)
(24) Glacial acetic acid (FS lot # ~~971799~~ 971798) AJ 2/15/02
(25) Sodium citrate dihydrate, $Na_3C_6H_5O_7 \cdot 2H_2O$
(26) Sodium dithionite, $Na_2S_2O_4$ (FS lot # 912722)
(27) Sodium chloride, NaCl
(28) Acetone (FS, lot # 993009)
(29) NN-dimethyl formamide (FS lot # 881639)
(30) Tetrabromoethane (Mallinckrodth lot # 1957 KBSH)

BM 2/12/02

PROCEDURE :- Reference to notebook # 266, pages 3 to 5 as follows :-

PROCEDURE :-

① Crush Clinoptilolite using mortar & pestle. Seive thru 8" diameter stainless steel 100-200 mesh seives using Sieve shaker.
② Wash Clinoptilolite with Nano-pure water several times till supernatant is clear. Use ultrasonic cleaner.
③ Removal of Carbonates :-
a) Transfer ~~5.0~~ 5.0 g AJ 2/15/02 of Clinoptilolite in several 100ml centrifuge tubes.
b) Add 50 ml of NaOAc buffer (at PH = 5) to each tube
c) The suspension is digested in a ≈ 95°C water bath for 30 minutes. The suspension is stirred with a rubber tipped rod before putting in wate bath.
d) The suspension is then centrifuged at 6000 rpm for 5 min. The supernatant is decanted.
e) The suspension is then washed with Nanopure water and centrifuged at 6000 rpm for 5 min two times.
④ Removal of Iron-Oxides :-
a) Transfer 10.0 g of clinoptilolite in several 100 ml centrifuge tubes.
b) Add 40 ml of 0.3 M Na-citrate solution and 5 ml of 1 M $NaHCO_3$ solution (these solutions can be added ahead of time). AJ 2/15/02
c) The temperature is brought to 75°-80°C (not >80°C) in a water bath. The suspension is transferred to the water bath and 1 gm of ~~Solid~~ $Na_2S_2O_4$ is added to each tube. The AJ 2/15/02 solid

mixture is stirred for 1 minute and digested for 5 min.

d) A second 1 gm portion of $Na_2S_2O_4$ (sodium dithionite) is added with stirring and digested for 5 min.

e) A third 1 gm portion of $Na_2S_2O_4$ is added with stirring & digested for another 5 min.

f) The suspension is then centrifuged at 6000 rpm for 5 min. The supernatant is decanted.

g) The suspension is then mixed with 10 ml of saturated NaCl solution and warmed in water bath for 5 min. The suspension is then centrifuged at 6000 rpm for 5 min. The supernatant is decanted.

h) The suspension is then washed with nanopure water and centrifuged at 6000 rpm for 5 min two times.

i) The suspension is filtered & dried in oven.

(5) Heavy Liquid Mineral Separation :-

a) A mixture of heavy liquid, Bromoform ($\rho_{20^\circ C}$ = 2.8899 g/cc) AJ 2/15/02 and diluent, NN-dimethyl formamide ($\rho_{20^\circ C}$ = ~~0.93~~ 0.9445 g/cc) is prepared using the following formula for volume ratio. 0.9345 g/cc

$$V_l = V_h \frac{G_h - G_x}{G_x - G_l}$$

where G_x = density of the mixture desired AJ 2/15/02

G_h = density of heavy liquid, bromoform 2.8899 g/cc

G_l = density of diluent

V_h = volume of heavy liquid

V_l = volume of diluent.

b) Transfer heavy liquid mixture to seperatory funnel clamped on metal stand. Add some clinoptilolite, cover with cork & shake it to mix well. Wait for about 30 minutes for complete density separation. Seperation is done under exhaust hood.

c) Open the stopper of separating funnel & trap the settled heavy impurities in a flask covered with filter & funnel. Pass most of the liquid AJ 2/15/02 thru it.

d) Change the flask and trap clean clinoptilolite in filter & funnel. Rinse the seperatory funnel with acet.

e) Repeat the whole procedure several times till all the clinoptilolite is cleared.

f) Remove the filter & let it dry under exhaust.

(6) Rinse the clean clinoptilolite several times with DI H_2O & dry it in oven.

AJ 2/15/02

AJ 2/12/02

10/24/00 AJ — Started with CDV = 60 g thru 60, 100 & 200 mesh size sieving.

Following fraction sizes were obtained

CDV < 200 mesh = 34.39 g

100 - 200 mesh = 25.52 g

60 - 100 mesh = 0.92 g

60 g CDV yield ~ 26 g of 100-20 mesh CDV ≈ 42%

Wash CDV < 200 mesh = 24.0 g with distilled water several times (~ 25-30 times) till supernatant is clear. This is necessary to remove finer particles.

10/25/00 AJ — After washing & drying CDV < 200 obtained = ~~8.69~~ 8.69 g. AJ 2/15/02

≈ 36 %.

Started with 481 g of CDV & seived thru 60, 100 & 200 mesh sizes sieves.

Total 100 - 200 mesh size fraction obtained = 203.90 g

10/26/00 Total CDV < 200 mesh size fraction obtained = 266.34 g

~~10/26/00~~ 10/27/00 AJ 2/15/02 Wash 203.90 g of CDV (100 - 200 mesh) several times with nanopure H_2O until supernatant is clear. Dry in oven at 65°C.

10/30/00 AJ Heavy liquid seperation started

10/31/00 wt. of CDV (100-200 mesh) after washing ≈ 180 g + 11 g ≈ 191 g.

Heavy Liquid Mineral Seperation :- Following the procedure # ⑤ on page 8, impurities were removed from clinoptilolite, 100-200 mesh size fraction.

$\rho_{clinoptilolite}$ = 2.16 g/cc, $\rho_{desired}$ = 2.3 g/cc

Purpose :- To remove impurities from 100 - 200 mesh size clinoptilolite utilizing heavy liquid density seperation

Materials or Supplies :- 2, 250 ml separatory funnels
#4 whatmen filter paper (18.5 cm dia.
2, 500 ml conical flasks
2, metal stands with clamps

Reagents :- NN-dimethyl formamide (FS Lot # 881639)
Tetrabromoethane (Mallinckrodt Lot # 1957 KBSH)
Acetone (FS Lot # 993009)

Procedure :-

① A mixture of heavy liquid tetrabromoethane and diluent NN-dimethylformamide was prepared using same procedure AJ 2/15/02 #5 on page 8. {150 ml of tetrabromoethane + {73.29 ml of NN-dimethyl formamide, Procedure

② Transfer HL mixture to seperatory ~~funnel~~ funnel AJ 2/15/02 clamped on metal stand. Add ~5 g of CDV (100-200 mesh), cover with cork, shake & mix well. Wait for 30 min. for complete density seperation. Perform the procedure under the exhaust hood.

③ Open stopper of separatory funnel and release settled heavy impurities onto filter paper and funnel over a conical flask.

④ Capture clean CDV onto another filter paper & funnel Rinse the remaining CDV in separatory funnel with acetone and capture on same filter paper.

⑤ Repeat the same procedure until all CDV is clean.

⑥ Remove filter paper from funnel & dry CDV under the hood.

⑦ Rinse CDV with DI water several times & dry in oven.

11/1/00
AJ Heavy liquid density separation continued.

11/3/00
AJ Following procedure #⑤ on page 8, a mixture of heavy liquid tetrabromoethane & diluent NN-dimethylformamide was prepared using formula on page 8.

V_{HL} (tetrabromoethane) = 150 ml
$V_{diluent}$ (NN-dimethylformamide) = 73.29 ml

Tetrabromoethane (Kodak CAS # 79-27-6, lot # 0250201274
NN-dimethylformamide (FS lot # 881639)

11/7/00
~~11/7/00~~ AJ 2/15/02
AJ Heavy liquid density separation continued and finished. Rinse CDV with acetone several times & dried under exhaust hood.

11/8/00 Removal of Carbonates :-
AJ Following procedure ③ (for removal of carbonates) the removal of carbonates started for CDV * 100-200 mesh * HL.
(FS lot # 937077)

NaOAc buffer :- 272.0 gm of $CH_3COONa \cdot 3H_2O$ AJ 2/15/02 (~~lot #~~ FS ~~937077~~) was dissolved in 2 L of DI H_2O. Adjust the PH to 5.0 with glacial CH_3COOH (# 971798 FS) $CH_3COOH \simeq 60$ ml. Store NaOAc buffer in plastic bottles.

CDV * HL was rinsed with DI H_2O several times & dried in oven at 65°C.
CDV * HL * 100-200 mesh = 176 gms.

11/9/00 Start removal of carbonates & continue
11/10/00 until all CDV is cleaned.

Another batch of NaOAc buffer was prepared for carbonate removal.

136 gms of $CH_3COONa \cdot 3H_2O$ dissolved in 1L of nanopure H_2O.

11/14/00 AJ Carborate removal continued (CDV*HL*100/200)

11/15/00 AJ carborate removal continued

11/17/00 AJ wt. of CDV (100/200) * HL * RC ≈ 176 gms.

Preparation of saturated NaCl & $NaHCO_3$ Solns.

NaCl (FS Lot # 984321) = 146 g + 73 g = 229 g

Nanopure H_2O ≈ 550 ml. + 275 ml = 825 g

1M $NaHCO_3$ Soln. (250 ml)

$NaHCO_3$ (CFS Lot # 936883) = 21.0 g & dilute it to 250ml with DI H_2O.

11/28/00 AJ Removal of Iron Oxides :-
Following the procedure of page 7 for removal of iron oxides, 20 ml of 0.3M Na citrate solution & 2.5 ml of 1M $NaHCO_3$ were mixed with 5g of CDV (100-200 mesh) * HL * RC ~~in~~ AJ 2/15/02 in 50 ml centrifuge tubes.
Total of 12 tubes

11/29/00 AJ Iron oxide removal from CDV * HL * RC (100/200) continued.

11/30/00 AJ Iron oxide removal from CDV * HL * RC (100-200 mesh) Continued.

12/1/00 AJ Iron oxide removal from CDV * HL * RC (100-200) Continued.

12/4/00 AJ CDV * 100-200 * HL * RC - removal of iron oxides continued.

12/6/00 AJ Removal of iron oxides from CDV * 100-200 * HL * RC continued.
wt. of cleaned & dried CDV * 100/200 * HL * RC * RFe = 141.02 g

12/7/00 AJ Removal of iron oxides from CDV * 100/200 * HL * RC * Continued. Wash with DI H_2O several times & dry it in oven at 65°C.

12/8/00 AJ Removal of iron oxides from CDV completed.
Total wt. of CDV * 100/200 * HL * RC * RFe = 172.78 g.

12/12/00 AJ 1100 hrs. Preparation of Na-enriched form of Clinoptilolite in equilibrium with NaCl.

objective :- To obtain Na-enriched form of CDV* 100-200* HL* RC* RFe with 3M NaCl solution for ion exchange experiments.

Method :- Purified CDV is reacted with 3M NaCl solution, and cation exchange sites are filled with Na^+.

Equipments & Materials :-

- Fisher Versabath Model
- 3M NaCl (lot # 986412)
- 500 ml PP bottles
- Blue M drying ove

Procedure :- Followed from Notebook #266, page 9 as follows:

① 25 g. of purified CDV was placed in 500 ml PP bottle. 2 bottles were prepared.

② 250 ml of 3M NaCl was added to the bottles.

③ The bottles were placed in water shaker bath set at 70°C and 60 rpm. The bottles were

continously agitated for about 14-15 days (2 weeks)

④ Replace 3M NaCl solution every 2 days.

Preparation of K-enriched form of Clinoptilolite

objective :- To obtain K-enriched form of CDV by reacting the solid with 3M KCl for ion exchange expts.

Method :- Purified CDV is reacted with 3M KCl solution and cation exchange sites are filled with K^+.

Equipments & Materials :- Same as on page 16 except
- 3M KCl (lot # 885967)

① 2 batches of 35g of CDV* 100/200 *HL* RC* RFe were reacted with 3M KCl. Water bath set at 70°C & 50 rpm.

② 25 g of CDV* 100/200 * HL* RC* RFe was reacted with 250 g of 3M NaCl. 2 batches of 25 g. of CDV were done. Water bath started at 1400 hrs.

Preparation of 3M NaCl :-

175.32 g of NaCl diluted with DI H_2O & transferred to 1000 ml flask. Fill with water upto 1000 ml mark.

Preparation of 3M KCl :- KCl (lot # 885967)

223.68 g of KCl diluted with

DI H_2O & filled upto 1000 ml mark.

12/13/00 AJ
1030 hrs. Preparation of 3M NaCl & 3M KCl for CDV.

87.66 g of NaCl (lot # 986412) was dissolved in DI H_2O & filled upto 500 ml mark.

111.84 g of KCl (lot # 885967) was dissolved in DI H_2O & filled up to 500 ml mark.

12/14/00 AJ
1030 hrs. 3M NaCl (250 ml) solution was replaced for both bottles containing 25g CDV each and placed back in water bath at 70°C.

3M KCl solution (350 ml) was replaced for both bottles ~~containing~~ containing 35g of CDV each & placed back in shaker AJ 2/15/02 water bath at 70°C.

2/15/00 AJ
Preparation of 3M NaCl & 3M KCl :-

① Dissolve 87.66 g of NaCl in DI H_2O & diluted it with DI H_2O to 1000 ml.

② 223.68 g ~~dilute~~ AJ 4/10/01 AJ of KCl was diluted with DI water to 1000 ml solution.

12/16/00 AJ
1350 hrs. 3M KCl & 3M NaCl solutions replaced.

12/18/00 Replace 3M KCl & 3M NaCl solutions.
1030 hrs. Prepare 500 ml 3M KCl & 3M NaCl each.

12/20/00 Prepare 1000 ml. 3M NaCl (175.32g/lt)
1000 hrs. Prepare 1000 ml 3M KCl (223.68g/lt)
Replace 3M KCl & 3M NaCl solutions.

12/21/00 Prepare 1000 ml 3M KCl. (223.68g diluted
1300 hrs. with DI H_2O to 1000 ml).
Replace the solutions (3M KCl & 3M NaCl) and decrease the temp. to 25°C on water bath.

01/09/01
AJ Water bath temperature set to 70°C and rpm set at 60 rpm.
Replace 3M KCl & 3M NaCl solutions.

01/10/01 Preparation of 3M NaCl & 3M KCl solutions
AJ
① Dissolve 175.32 g of NaCl in DI H_2O & dilute it to 1000 ml.

② Dissolve 223.68 g of KCl in DI H_2O & dilute it to 1000 ml.

AJ 2/15/02
~~01/12/01~~ Replace 3M KCl & 3M NaCl solutions AJ 2/15/02
AJ Prepare 500 ml of 3M NaCl &
(see page 18) 500 ml of 3M KCl solutions

01/14/01 AJ
2100 hrs. 3M KCl & 3M NaCl solutions replaced.

01/16/01 AJ Remove bottles from waterbath. The
1130 hrs. Clinoptilolite was washed several (~6 times) times with DI water. Place in waterbath at 25°C, 80 rpm to swirl Clinoptilolite in DI water.

01/17/01 AJ ① Remove clinoptilolite from water bath and
1000 hrs. wash it more with DI H_2O few more times. (~3-4 times).

② Preparation of 0.1M $AgNO_3$:-
1.7g of $AgNO_3$ (Lot # 976504 borrowed from Corrosion Lab)
diluted to 100 ml.

③ Decant 30-40 ml of DI H_2O from one of bottles containing Naf of Clinoptilolite and add 2-3 drops of 0.1M $AgNO_3$. No ppt. observed. This test was done to see presence of Cl^- ions.

④ Prepartion of double junction ~~Potass~~ AJ 2/15/02 reference electrode for K^+ ion-conc. measurements using

ISE (ion-selective electrodes).
Outer chamber solution :- Dilute 2ml ISA (ion-strength adjustor) for K^+ to 100 ml with AJ 2/15/02 ~~with~~ DI H_2O. Use this solut. to fill the outer chamber of double-junction reference electrode.

1/19/01 AJ
① Preparation of K^+ electrode for ISE measurements practice. Soak the electrode in DI H_2O for 15 min. & then in 10^{-2} M K^+ standard for 1 hr.
② Preparation of 10^{-2} M K^+ std. :-
Dilute 0.1 M K^+ standard (# 921906) (5ml) to 50 ml with DI H_2O.

③ Rinse Naf & kf Clinoptilolite several times & test for Cl^- ion using 0.1M $AgNO_3$. No white ppt or cloud ppt. observed. Transfer Naf CDV & kf CDV into two separate beakers and dry it overnight at ~~5~~ 50C. AJ 1/19/01

④ Check electrode operation following the instructions from orion model 93-19 potassium electrode instruction manual as follows:

1^{st} mV reading = ~~-41.8~~ AJ 2/15/02 -41.8
2^{nd} mV reading = 12.8
difference = $12.8 - (-41.8) = 54.6$ mV [54-60 mV O.K.]

1/22/01 AJ Prepare 0.5 M KCl using 3.728 g KCl (Lot # 901422, FS) AJ 2/15/02 & dilute it to 100 ml with DI H_2O.
Preparation of 0.001 (10^{-3}) M K^+ std. by diluting 2.5 ml of 0.01 (10^{-2}) M K^+ std. with DI H_2O to 25 ml.
Practice with K^+ electrode - calibration.

1/23/01 AJ

Practice with K^+ electrode - calibration using old sensing membrane (module). The values were not stable, constantly changing. Condition new sensing module. Also, slope with old sensing module didn't get ~~better~~ AJ 4/10/01 higher then 47 mv (should be between 54-60 mv).

1/24/01 AJ Ca^{2+} electrode /w single junction reference.

0.01 M (std)	Th. Value (400.8)		slope = 24 mv
0.1 M (std.	4008.0		

1/24/01 AJ Measure 0.05 M $CaCl_2$ 1480 PPm
Practice with K^+ electrode calibration & measurements using new sensing membrane. Also practice with Ca^{++} electrode calibration with old sensing module. Slope should be between ~~25~~ 25 AJ 2/15/02 – 30 mV) for Ca^{++}.

1/25/01 AJ

1/25/01 AJ 2/15/01 ① Recalibration with K^+ electrode & remeasure same samples to see reproducibility & accuracy.
The results are as follows.

2 point Calibration :-

	PPm (Measured)	Theoritical	slope
0.001 M (std.) K^+	37	39.1	
0.010 M (std.) AJ 2/15/02	370	391	
1/24/01 0.005 M (KCl)	270, 260, 260, 270, ~~200~~ 260 AJ 2/15/02		56.6 mv
1/25/01	266/267, 260/264		~~56.7~~ 56.7 mv AJ 2/15/02

3-point Calibration :-

	Measured PPm	Theorital Value	Slope
0.001 M (Std) K^+			
0.01 M			
0.1 M			57.2
1/24/01 0.005 M KCl	260/270 260		56
1/25/01 0.05 M KCl	2600/2700 ~~2600/2700~~		

② Remove samples ((CDV), converted to Naf & kf) from oven at 60°C.
wt. of Naf CDV = 48.94 g.
wt. of Kf CDV = 70.05 g

1/30/01 AJ

wt. of Kf CDV = ~~70.06~~ 70.06 g AJ 2/15/02

wt. of Naf CDV = 49.0 g

Practice with K^+ electrode calibration. Remeasure the same samples as on 1/25/01 & values are as follows:

2-point Calibration :-

	Measured PPM	Theoritical Value	Slope
0.001 M K^+ std.	37/38	39.1	57.9
0.01 M K^+ std.	~~37~~ 370 AJ 2/15/02	391.0	
0.005 M KCl	270		

3-point Calibration :-

	Measured ppm	Th. value	Slope
0.001 M K^+ std	38	39.1	
0.01 M K^+ std.	~~37~~ 370/380 AJ 2/15/02	391	55.1
0.1 M K^+ std.	3800	3910.00	55.6
0.005 M KCl	260/270, 260/270		
0.05 M KCl	~~2600~~ 2600, 2600 AJ 2/15/02		

Preparation of stock solution, 0.5 M KCl :-

~~Prepare~~ Prepare 0.5 M KCl using new KCl (Lot # 006242, FS). AJ 2/15/02

F.W = 74.56

Dilute 3.728 g of KCl with DI H_2O to 100 ml.

① ~~0.25~~ 0.25 M KCl :- Dilute 12.5 ml of 0.5 M KCl to 25 ml with DI H_2O. AJ 2/15/02

② 0.05 M KCl :- Dilute 2.5 ml of 0.5 KCl solution with

DI H_2O to 25 ml mark.

③ 0.005 M KCl :- Dilute 0.25 ml of 0.5 KCl soln. ~~EB~~ AJ 2/15/02 with DI water to 25 ml.

Calibration k^+ electrode (3 pt.) :-

0.001 M k^+ std.	39, 37
0.01 M	400, 370
0.1 M	400, 4000

1/31/01 AJ

	Resolution 0.00 (2-digit)	(3-digit resolution)
0.001 K^+ std	40	38.6, 38.8
0.01 M ↓	400	388, 391
0.1 M ↓	4000	3890, ~~3880~~ 3880 AJ 2/15/02
0.005 KCl	270	very unstable (242-252) 248 1270
0.025	1400	" 1250-1280
0.050	2700	" 2480-2520 2520
0.250	13,000	" 11700 11600-11900
0.500	out of range.	

2/2/01 AJ

	1 digit	(2-digit)	(3-digit resolution)
0.001 M k^+ std.	40	(37-40) 38	37.9-38.4, 37.7-38.2
0.01 M	400,	(400-410) 410	410
0.1 M	4000,	~~3900~~ (3800-3899) AJ 2/15/02	3880, 3970
0.005 KCl	260 AJ 2/15/02	(260-270)	(260-263) 261
0.025	1300	(1300-1400)	(1310-1340) 1330
0.05	2600	(2600-2700)	2550 (2550-2620)
0.25	12000		12000 (11900-12000)

Calibrated at 2-digit resolution

$CaCl_2$ (lot # 995698, FS) F.W. = 147.02

1M 147.02/lt , 0.5M 73.51/lt , 0.25M 36.755/lt.

Preparation of 0.5N (0.25M) $CaCl_2$:-

Dilute 1.8378 g of $CaCl_2$ with DI H_2O to 50 ml.

Dilute 0.5M KCl to 0.25 M (5ml 0.5M KCl + 5ml 0.5N $CaCl_2$)

0.025 M (0.5ml KCl + 9.5ml $CaCl_2$)

0.05 M (1 ml KCl + 9ml $CaCl_2$)

(0.1ml KCl + 9.9ml $CaCl_2$) 0.005 M with 0.5N $CaCl_2$.

Calibrate K^+ electrode using 0.1M, 0.01M & 0.001M K^+ std. and measure K^+ in KCl/$CaCl_2$ solution mixture. (Resolution - 2-digit)(Calibration - 3point at 0.1, 0.01 & 0.001M)

KCl/$CaCl_2$	PPm	Theoritical value
0.005	350	195.5
0.05	3600	1955
0.025	~~18,000~~ AJ 2/15/02 1800	977.5
0.25	15,000	AJ 2/15/02 9775

2/6/01 Continue measuring K^+ in KCl/$CaCl_2$ solutions

AJ	0.005	~~350~~ AJ 4/10/01 320
	0.05	~~3600~~ AJ 4/10/01 3500
	0.025	1800
	0.25	15,000
K^+ std.	0.001 M	39
	0.01 M	380/390
	0.1 M	3600/3700

Recalibrated (3-pt.)

0.001 M K^+ std.	39.1	37/38
0.01	391	370/380
0.1	3910	3800/3899

0.005 KCl/$CaCl_2$	320	K^+ Stds. after measuring KCl/$CaCl_2$
0.025	~~1800~~ AJ 2/15/02 1700	37
0.05	3500/3400	360
0.25	15,000	3600/3700

02/07/01 AJ — NO Calibration

K^+ Std.	PPM
0.001 M	44/45
0.01	430
0.1	4300

3-pt. Calibration & 2-digit resolution

K^+ ~~std~~ std. 2/15/02 after calibration

0.001 M	38/39
0.01 M	390
0.1 M	3800/3899

KCl/$CaCl_2$

0.005 M	330
0.025	1700
0.05	3400
0.25	14,000

2/13/01 Objective :- BET surface area analysis on Naf & AJ Kf of CDV * 100/200 * UC * HL * CPT using Coulter SA3100 surface area analyzer.

~~Equipments & Supplies :-~~ AJ 2/15/02

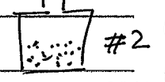
Equipments

Coulter SA3100
2 Sample holders with tube assembly.
He & N_2 gas tanks
Mettler AE240 weighing scale & weighing paper.

Procedure :- As per Coulter SA3100 product manual on page (5-1).

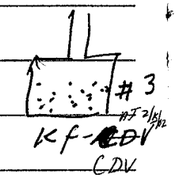
outgass temp. = 350°C , outgass time = 720 min

wt. of tube assembly #2 = 33.3319 g
wt. of tube assembly #2 + Naf CDV = 34.5434 g
Sample wt. = 1.2115 g



2/14/01
wt. of outgassed sample + tube assembly = 34.4030 g.
wt. of outgassed Naf-CDV = 34.4030 - 33.3319
= 1.0711 g

wt. of tube assembly #3 = 33.7029 g
+ Kf CDV = 34.9145 g
Sample wt. = 1.2116 g



2/14/01 wt. of outgassed sample + tube assembly #3 = 34.8071g
wt. of outgassed Kf CDV = 34.8071
- 33.7029
1.1042 g.

14 Feb 01 PB Several solutions of Sr^{2+}, Ca^{2+}, Ic^{-}, and Na^{+} will be made to check the operation of 2/14/01 and capabilities of capillary electrophoresis analyzer for use in the Ix expts.

Initially, calibration solutions for Sr^{2+} will be made by diluting a 1000 ppm Sr standard. sample solutions containing various quantities of Ic^{-} and Ca^{2+} will also be made. Finally, solutions with small amounts 1-10 ppm of Ic^{-} in 0.5 M background Ca^{2+} will be made to test ability to measure Ic in the strong Ca^{2+} matrix.

Sr standard 1000 µg/ml spex Lot # 7-123 SR exp 5/15/01

transferred 10ml of Sr std to 10 mL vol flask (after washing flask with ~ 3 mL std). made up to mark.
Quantitatively transferred 10 mL of std to a 1000 mL vol flask to make a 10 ppm Sr^{2+} solution. Made up to mark with nH_2O.
(10 mL * 1000 ppm) / 1000 mL = 10 ppm Sr^{2+}

To make 1 ppm std. transferred 10ml of 10 ppm solution using vol pipette (10 mL) into 100 mL vol flask. Made up to mark with nH_2O.

To make 0.5 ppm std. transfered 10 mL of 10 ppm solution using 10 mL vol. pipette in to 200 mL vol flask. Made up to mark using nH_2O.

14 Feb 01 To make 2 ppm std. Transferred 20 mL of 10 ppm solution into 100 mL vol. flask using 20 mL vol. pipette.

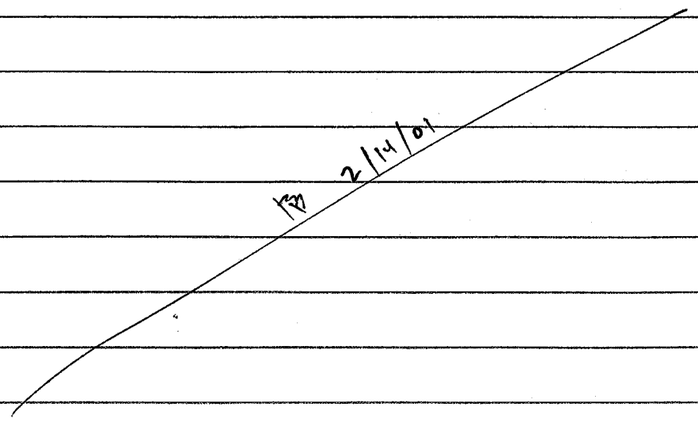
To make 5 ppm std. transferred 50 mL of 10 ppm solution using 50 mL vol. pipette into 100 mL vol. flask.

$\frac{(50\ mL) * 10\ ppm}{100\ mL} = 5\ ppm$ $\frac{(10\ mL) * 10\ ppm}{100\ mL} = 1\ ppm$

$\frac{(20\ mL) * 10\ ppm}{100\ mL} = 2\ ppm$ $\frac{(10\ mL) * 10\ ppm}{200\ mL} = 0.5\ ppm$ 2/14/01

=) have solutions of 10, 5, 2, 1, and 0.5 ppm Sr^{2+} in nH_2O

Transferred ~ 10 mL of each solution into a 50 mL PP beaker for temporary storage. Then transferred ~ 1 mL into labeled ~~capp~~ PP capillary vials using LDPE transfer pipette. 2/14/01



2/14/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NAP_CDV	Start Date	2/13/01 ~~10/11/90~~ AJ 4/10/01
Customer	UNKNOWN	Start Time	01:03:54
Operator	JAIN	Elapsed Time	39 min
Sample Wt	1.0711 g	Outgas Time	0 min
Profile	BET5	Outgas Temperature	0 C

Naf CDV * 100/200 * UC * HL * CPT

Summary

Surface Area Report

BET Surface area 32.288 sq.m/g
Correlation Coefficient 0.99997

Surface Area Report

BET Surface area 32.288 sq.m/g

Slope 0.133872
Intercept 0.000928
C_value 145.257
Monolayer Volume 7.4184 cc/g (STP)
Correlation Coefficient 0.99997

One Point BET Surface Area (Ps/Po=0.3) 31.777 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0483	0.007410	6.850
0.0552	0.008347	6.999
0.0623	0.009305	7.142
0.0674	0.009986	7.240
0.0702	0.010345	7.296
0.0799	0.011634	7.466
0.1046	0.014908	7.836
0.1187	0.016757	8.035
0.1363	0.019102	8.263
0.1403	0.019626	8.315
0.1595	0.022222	8.543
0.1827	0.025380	8.807
0.1984	0.027575	8.977
0.2016	0.027996	9.017

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.007622	6.906
0.0800	0.011638	7.472
0.1200	0.016993	8.025
0.1600	0.022347	8.523
0.2000	0.027702	9.025

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NAF_CDV	Start Date	2/13/01 ~~10/14/98~~ AJ 4/10/01
Customer	UNKNOWN	Start Time	01:03:54
Operator	JAIN	Elapsed Time	39 min
Sample Wt	1.0711 g	Outgas Time	0 min
Profile	BET5	Outgas Temperature	0 C

Naf CDV * 100/200 * UC * HL * CPT

Isotherm Data

Freespace Calculation

Slope	0.03515
Intercept	4.997e-005
Correlation Coefficient	0.99999

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	751.84
0.0000	0.417	0.001	752.10
0.0000	0.838	0.011	752.11
0.0000	1.326	0.019	751.91
0.0000	1.869	0.033	752.01
0.0001	2.472	0.057	752.43
0.0002	3.146	0.143	752.46
0.0007	3.880	0.539	752.63
0.0024	4.652	1.770	752.79
0.0056	5.194	4.244	752.43
0.0118	5.659	8.886	752.05
0.0207	6.060	15.539	751.84
0.0292	6.352	21.977	751.98
0.0350	6.521	26.343	752.22
0.0416	6.692	31.295	752.20
0.0483	6.850	36.346	752.43
0.0552	6.999	41.516	752.18
0.0623	7.142	46.847	751.85

Coulter SA 3100 Surface Area and Pore Size Analyzer

Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	KF_CDV	Start Date	2/13/01 ~~10/14/98~~ AJ 4/10/01
Customer	CNWRA	Start Time	02:53:52
Operator	JAIN	Elapsed Time	42 min
Sample Wt	1.1042 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	350 C

CDV * 100/200 * UC * HL * CPT * Kf

Summary

Surface Area Report

BET Surface area	31.582 sq.m/g
Correlation Coefficient	0.99998

Surface Area Report

BET Surface area	31.582 sq.m/g
Slope	0.136905
Intercept	0.000909
C_value	151.689
Monolayer Volume	7.2562 cc/g (STP)
Correlation Coefficient	0.99998

One Point BET Surface Area (Ps/Po=0.3) 31.103 sq.m/g

Analysis Data Ps/Po	BET Function	Vads cc/g(STP)
0.0532	0.008190	6.856
0.0603	0.009181	6.992
0.0655	0.009890	7.083
0.0700	0.010509	7.161
0.0801	0.011902	7.318
0.1010	0.014740	7.624
0.1112	0.016111	7.763
0.1199	0.017287	7.880
0.1400	0.020018	8.131
0.1607	0.022856	8.380
0.1787	0.025364	8.579
0.2001	0.028396	8.807

Interpolated Data Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.007754	6.788
0.0800	0.011861	7.331
0.1200	0.017337	7.965
0.1600	0.022813	8.349
0.2000	0.028290	8.837

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	KF_CDV	Start Date	2/13/01 ~~10/14/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	02:53:52
Operator	JAIN	Elapsed Time	42
Elapsed Time	42 min		
Sample Wt	1.1042 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	350 C

Operator JAIN

CDV * 100/200 * UC * HL * CPT * kf

Isotherm Data

Freespace Calculation

Slope	0.03534
Intercept	0.009164
Correlation Coefficient	0.99995

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	749.58
0.0001	0.395	0.092	749.44
0.0002	0.801	0.171	749.54
0.0003	1.271	0.240	749.38
0.0004	1.795	0.321	749.52
0.0006	2.374	0.424	749.41
0.0006	3.025	0.472	749.46
0.0007	3.598	0.516	749.54
0.0009	4.197	0.646	749.34
0.0019	4.803	1.391	749.38
0.0053	5.364	3.956	749.38
0.0126	5.822	9.472	749.28
0.0232	6.188	17.382	749.37
0.0326	6.430	24.432	749.32
0.0390	6.574	29.204	749.42
0.0460	6.721	34.469	749.24
0.0532	6.856	39.841	749.36
0.0603	6.992	45.199	749.30
0.0655	7.083	49.048	749.25
0.0700	7.161	52.443	749.26
0.0801	7.318	60.032	749.24
0.1010	7.624	75.692	749.23
0.1112	7.763	83.273	749.04
0.1199	7.880	89.802	749.07
0.1400	8.131	104.856	749.07
0.1607	8.380	120.433	749.22
0.1787	8.579	133.851	749.00
0.2001	8.807	149.834	748.96

2/16/01 AT The surface area values for Kf-CDV & Naf-CDV measured using BET SA as 31.582 m^2/g & 32.288 m^2/g seems unusually high compared to expected values between 9-12 m^2/g (as obtained previously). NIST-8005 ~~std.~~ 4/10/01 AJ std. will be analyzed using SA3100 to check the results.

Objective 1- To test the accuracy of Coulter SA3100 using standard NIST8005 (α-Al_2O_3)

outgass temp. = 350°C, outgas time = 180 min.

wt. of tube assembly #1 = 33.5819

" " " " " + NIST8005 = 35.3255

wt. of NIST8005 added = 1.7436 g.

wt. of tube assembly #1 + outgased NIST8005 = 35.3126 AJ 2/15/02

wt. of outgased NIST8005 = 35.3126 − 33.5819 = 1.7307 AJ 4/10/01

wt. of tube assembly #9 = 33.2608

" " " " " + NIST8005 = 34.8468

wt. of NIST8005 = 1.5860 g.

wt. of tube assembly #9 + outgased sample = 34.8359

wt. of outgased NIST 8005 = 34.8359 − 33.2608 = 1.5751 g. AJ 2/15/02

BW 2/13/02

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	N_ST8005	Start Date	2/16/01 ~~10/16/00~~ 4/10/01 AS
Customer	CNWRA	Start Time	08:18:39
Operator	JAIN	Elapsed Time	22 min
Sample Wt	1.5751 g	Outgas Time	180 min
Profile	BET5	Outgas Temperature	350 C

NIST 8005 (α-Al_2O_3)

Summary

Surface Area Report

BET Surface area	1.914 sq.m/g
Correlation Coefficient	0.99999

Surface Area Report

BET Surface area	1.914 sq.m/g
Slope	2.254063
Intercept	0.019616
C_value	115.911
Monolayer Volume	0.4398 cc/g (STP)
Correlation Coefficient	0.99999

One Point BET Surface Area (Ps/Po=0.3) 1.876 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0560	0.144860	0.410
0.0660	0.167982	0.421
0.0730	0.184021	0.428
0.0798	0.199595	0.434
0.0998	0.245286	0.452
0.1202	0.291462	0.469
0.1396	0.334834	0.485
0.1599	0.380419	0.500
0.1802	0.425490	0.516
0.1997	0.469224	0.532
0.2037	0.478326	0.535

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.132319	0.398
[illegible]	0.199941	0.435
0.1200	0.290103	0.470
0.1600	0.380266	0.501
0.2000	0.470428	0.531

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	N_ST8005	Start Date	2/16/01 ~~10/16/00~~ 4/10/01 AS
Customer	CNWRA	Start Time	08:18:39
Operator	JAIN	Elapsed Time	22 min
Sample Wt	1.5751 g	Outgas Time	180 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03521
Intercept	-0.004755
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	761.89
0.0020	0.243	1.557	762.15
0.0110	0.322	8.369	762.08
0.0213	0.355	16.235	761.98
0.0371	0.385	28.271	762.20
0.0457	0.397	34.848	762.31
0.0560	0.410	42.724	762.27
0.0660	0.421	50.297	762.34
0.0730	0.428	55.615	762.36
0.0798	0.434	60.805	762.40
0.0998	0.452	76.085	762.31
0.1202	0.469	91.630	762.16
0.1396	0.485	106.423	762.37
0.1599	0.500	121.883	762.23
0.1802	0.516	137.347	762.33
0.1997	0.532	152.282	762.49
0.2037	0.535	155.284	762.37

BWS 2/13/02

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NIST8005	Start Date	2/16/01 ~~10/16/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	07:43:31
Operator	JAIN	Elapsed Time	22 min
Sample Wt	1.7307 g	Outgas Time	180 min
Profile	BET5	Outgas Temperature	350 C

Summary

Surface Area Report

BET Surface area	1.909 sq.m/g
Correlation Coefficient	0.99999

Surface Area Report

BET Surface area	1.909 sq.m/g
Slope	2.261213
Intercept	0.018855
C_value	120.929
Monolayer Volume	0.4386 cc/g (STP)
Correlation Coefficient	0.99999

One Point BET Surface Area (Ps/Po=0.3) 1.873 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0550	0.142316	0.409
0.0651	0.165604	0.420
0.0721	0.181792	0.427
0.0797	0.199331	0.434
0.0998	0.245217	0.452
0.1199	0.290666	0.469
0.1396	0.335095	0.484
0.1599	0.380698	0.500
0.1800	0.425315	0.516
0.2000	0.470594	0.531
0.2039	0.479798	0.534

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.131915	0.399
0.0800	0.199752	0.435
0.1200	0.290200	0.470
0.1600	0.380649	0.500
0.2000	0.471097	0.531

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NIST8005	Start Date	2/16/01 ~~10/16/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	07:43:31
Operator	JAIN	Elapsed Time	22 min
Sample Wt	1.7307 g	Outgas Time	180 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03472
Intercept	-0.004744
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	760.00
0.0015	0.231	1.169	760.15
0.0098	0.317	7.455	760.33
0.0202	0.354	15.390	760.20
0.0360	0.384	27.377	760.18
0.0447	0.396	33.969	760.19
0.0550	0.409	41.838	760.13
0.0651	0.420	49.459	760.15
0.0721	0.427	54.808	760.47
0.0797	0.434	60.600	760.43
0.0998	0.452	75.903	760.63
0.1199	0.469	91.187	760.69
0.1396	0.484	106.200	760.80
0.1599	0.500	121.625	760.44
0.1800	0.516	136.825	760.21
0.2000	0.531	152.013	760.20
0.2039	0.534	155.073	760.42

BW 2/13/02

2/20/01 AJ

objective :- To determine the surface area of previously prepared CDV * 100/200 * UC * HL * CPT * Nf (and saturated wt.), CDV * 325 * UC * HL * CPT and to test the accuracy of SA3100 analyzer.

Equipment and Supplies :- Coulter SA3100
3 Sample tubes w/assembly
He & N_2 gas tanks
Liquid N_2.
AE Metler weighing balance
Weighing paper
γ-Al_2O_3 (Lot # C16F19, Alfa Aesar) & CDV*100/200 & CDV* 200/325

Procedure :- As per Coulter SA3100 product manual on page (5-1).

Initial Conditions :- outgas temp. = 350°C
outgas ~~temp~~ AJ 4/10/01 time = 330 min.

2/21/01 AJ

① wt. of tube assembly #1 = 33.6919 g
② wt. of tube assembly #2 = 33.3355 g
③ wt. of tube assembly #3 = 33.6744 g

① wt. of tube assembly #1 & γ-Al_2O_3 = 34.1070 g
② wt. of tube assembly #2 & CDV * 325/200 * UC * WA = 34.9095
③ wt. of tube assembly #3 + CDV * 100/200 * UC * HL * CPT = 35.2032

① wt. of γ-Al_2O_3 added = ~~35.2032 − 33.6744~~ AJ 2/21/01
~~= 1.5288 g.~~ AJ 2/21/01
= 34.1070 − 33.6919 g
= 0.4151 g.

wt. of outgassed γ-Al_2O_3 + tube assembly #1 = 34.0816
wt. of outgassed γ-Al_2O_3 = 34.0816 − 33.6919 g
= 0.3897 g.

② wt. of CDV * 325/200 * UC * WA added = 34.9095 − 33.3355
= 1.5740 g.

wt. of CDV * 325/200 * UC * WA outgased & tube assembly #2 = 34.7247
wt. of outgased CDV * 325/200 * UC * WA = 34.7247 − 33.3355 g.
= 1.3892 g

③ wt. of CDV * 100/200 * UC * HL * CPT ~~added~~ added AJ 2/15/02 = 35.2032 − 33.6744
= 1.5288 g.

wt. of outgased CDV * 100/200 * UC * HL * CPT + tube assembly #3
= 35.0218

wt. of outgassed CDV * 100/200 * UC * HL * CPT = 35.0218 − 33.6744
= 1.3474 g.

SW 2/13/02

2/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
			2/21/01 AJ
Sample ID	GAMMA-AL	Start Date	~~10/21/90~~ 4/10/01
Customer	CNWRA	Start Time	11:04:02
Operator	JAIN	Elapsed Time	46 min
Sample Wt	0.3897 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

γ-Al_2O_3

Summary

Surface Area Report

BET Surface area 87.003 sq.m/g
Correlation Coefficient 0.99998

Surface Area Report

BET Surface area 87.003 sq.m/g

Slope 0.049701
Intercept 0.000324
C_value 154.202
Monolayer Volume 19.9897 cc/g (STP)
Correlation Coefficient 0.99998

One Point BET Surface Area (Ps/Po=0.3) 85.706 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0499	0.002808	18.720
0.0572	0.003174	19.124
0.0647	0.003546	19.503
0.0702	0.003821	19.754
0.0799	0.004300	20.192
0.0978	0.005183	20.919
0.1222	0.006380	21.811
0.1405	0.007288	22.431
0.1593	0.008222	23.048
0.1795	0.009248	23.662
0.2005	0.010321	24.297

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.002809	18.734
0.0800	0.004301	20.220
0.1200	0.006289	21.684
0.1600	0.008277	23.014
0.2000	0.010265	24.355

2/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
			2/21/01
Sample ID	GAMMA-AL	Start Date	4/10/01 AJ ~~10/21/90~~
Customer	CNWRA	Start Time	11:04:02
Operator	JAIN	Elapsed Time	46 min
Sample Wt	0.3897 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope 0.03635
Intercept -0.005514
Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	717.85
0.0000	1.150	0.007	717.95
0.0000	2.306	0.021	718.36
0.0001	3.640	0.055	718.65
0.0002	5.131	0.149	718.55
0.0004	6.752	0.305	718.71
0.0007	8.557	0.495	718.68
0.0013	10.538	0.967	719.10
0.0026	12.115	1.848	719.15
0.0053	13.635	3.828	718.98
0.0105	15.025	7.564	719.24
0.0184	16.231	13.261	719.70
0.0269	17.098	19.356	719.76
0.0364	17.865	26.223	719.46
0.0428	18.288	30.849	720.18
0.0499	18.720	35.927	719.34
0.0572	19.124	41.171	719.45
0.0647	19.503	46.549	719.65
0.0702	19.754	50.504	719.65
0.0799	20.192	57.506	719.89
0.0978	20.919	70.384	719.54
0.1222	21.811	87.932	719.83
0.1405	22.431	101.138	719.85
0.1593	23.048	114.682	719.86
0.1795	23.662	129.243	719.86
0.2005	24.297	144.328	719.88

Note :- SA value for γ-Al_2O_3 is close to values obtained previously.

3/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-325	Start Date	2/21/01 ~~10/21/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	12:16:32
Operator	JAIN	Elapsed Time	25 min
Sample Wt	1.3892 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

CDV * 200/325 * UC * WA (old)

Summary

Surface Area Report

BET Surface area 9.037 sq.m/g
Correlation Coefficient 0.99999

Surface Area Report

BET Surface area 9.037 sq.m/g

Slope	0.478354
Intercept	0.003277
C_value	146.952
Monolayer Volume	2.0763 cc/g (STP)
Correlation Coefficient	0.99999

One Point BET Surface Area (Ps/Po=0.3) 8.896 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0504	0.027384	1.938
0.0594	0.031758	1.989
0.0658	0.034808	2.024
0.0730	0.038220	2.059
0.0802	0.041644	2.093
0.1000	0.051100	2.176
0.1202	0.060694	2.250
0.1392	0.069743	2.319
0.1597	0.079561	2.390
0.1803	0.089528	2.457
0.2002	0.099244	2.522

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.027195	1.935
0.0800	0.041546	2.093
0.1200	0.060680	2.247
0.1600	0.079814	2.386
0.2000	0.098948	2.527

3/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-325	Start Date	10/21/90
Customer	CNWRA	Start Time	12:16:32
Operator	JAIN	Elapsed Time	25 min
Sample Wt	1.3892 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03446
Intercept	0.005347
Correlation Coefficient	0.99997

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	751.70
0.0000	0.322	0.026	751.58
0.0001	0.644	0.088	751.75
0.0010	0.997	0.728	751.69
0.0042	1.324	3.174	751.66
0.0105	1.550	7.921	751.85
0.0202	1.698	15.200	751.84
0.0333	1.820	25.045	751.85
0.0412	1.879	30.957	751.80
0.0504	1.938	37.878	751.71
0.0594	1.989	44.664	751.63
0.0658	2.024	49.467	751.70
0.0730	2.059	54.844	751.66
0.0802	2.093	60.259	751.60
0.1000	2.176	75.199	751.63
0.1202	2.250	90.338	751.73
0.1392	2.319	104.646	751.80
0.1597	2.390	120.118	751.92
0.1803	2.457	135.610	752.00
0.2002	2.522	150.595	751.91

BWS 2/13/02

2/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-200	Start Date	2/21/01 AJ 4/10/01 ~~10/21/90~~
Customer	CNWRA	Start Time	13:01:44
Operator	JAIN	Elapsed Time	40 min
Sample Wt	1.3474 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

CDV * 100/200 * UC * RC * HL *

Summary

Surface Area Report

BET Surface area 29.243 sq.m/g
Correlation Coefficient 0.99999

Surface Area Report

BET Surface area 29.243 sq.m/g

Slope 0.147680
Intercept 0.001154
C_value 128.950
Monolayer Volume 6.7189 cc/g (STP)
Correlation Coefficient 0.99999

One Point BET Surface Area (Ps/Po=0.3) 28.723 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0528	0.008943	6.228
0.0596	0.009977	6.357
0.0646	0.010712	6.446
0.0700	0.011510	6.536
0.0800	0.012997	6.692
0.0989	0.015761	6.965
0.1200	0.018844	7.240
0.1418	0.022026	7.499
0.1597	0.024678	7.702
0.1791	0.027574	7.915
0.1996	0.030684	8.126
0.2027	0.031151	8.161

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.008538	6.164
0.0800	0.012969	6.705
0.1200	0.018876	7.224
0.1600	0.024783	7.686
0.2000	0.030690	8.146

2/21/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-200	Start Date	2/21/01 ~~10/21/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	13:01:44
Operator	JAIN	Elapsed Time	40 min
Sample Wt	1.3474 g	Outgas Time	330 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03393
Intercept	0.001903
Correlation Coefficient	0.99998

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	756.15
0.0000	0.335	0.007	756.07
0.0000	0.665	0.019	756.14
0.0000	1.052	0.035	756.18
0.0001	1.485	0.055	756.07
0.0001	1.967	0.103	756.15
0.0003	2.500	0.231	756.27
0.0008	3.088	0.636	756.28
0.0020	3.722	1.526	756.25
0.0038	4.196	2.885	756.24
0.0073	4.651	5.532	756.34
0.0133	5.062	10.075	756.13
0.0203	5.366	15.340	756.19
0.0284	5.635	21.470	756.11
0.0339	5.790	25.603	756.24
0.0399	5.946	30.155	755.93
0.0462	6.090	34.941	755.69
0.0528	6.228	39.865	755.66
0.0596	6.357	45.051	755.36
0.0646	6.446	48.782	755.24
0.0700	6.536	52.851	755.44
0.0800	6.692	60.448	755.47
0.0989	6.965	74.743	755.54
0.1200	7.240	90.687	755.44
0.1418	7.499	107.106	755.51
0.1597	7.702	120.635	755.36
0.1791	7.915	135.378	755.70
0.1996	8.126	150.725	755.26
0.2027	8.161	153.086	755.29

Note :-
Similar value of SA (32 & 31) m^2/g) were obtained on 2/14/01 (Pg 31 - 33)

2/27/01 AJ

Objective :- To determine the surface area of (CDV * 100/200 * CPT * HL * Naf & saturated over NaCl prepared earlier & stored on 11/2/99 by AJ), (old CDV * ~~1.50~~ AJ 2/27/01 * 200/325 * UC * WA) using Coulter SA 3100.

Procedure :- As per Coulter SA3100 product manual on page (5-1).

Initial conditions :- outgas temp. = 350°C
outgas time = 720 ~~min~~ AJ 2/15/02 min

① wt. of tube assembly #9 = ~~33.1988~~ 33.1988 AJ 2/13/02 g.
wt. of tube assembly #9 + CDV * 100/200 * CPT * HL * Naf (old) = 34.7437 g.
wt. of CDV * 100/200 * CPT * HL * Naf (old) added = 34.7437 − 33.1988
= 1.5449 g.

2/28/01 AJ
wt. of outgased sample with tube assembly = 34.5279
wt. of outgased sample = 34.5279 − 33.1988
= 1.3291 g.

② wt. of tube assembly #~~4~~ 1 = 33.5319 2/13/02
wt. of tube assembly #1 + CDV * 325/200 * UC * WA = ~~35.0610~~ 35.0610 AJ 2/15/02
wt. of sample added = 1.5291 g.

(No Liquid nitrogen available) couldn't do analysis.

3/2/01 AJ

Initial conditions, outgas temp. = 350°C
outgas time = 240 min.

① wt. of tube assembly #~~1~~ 2 = 33.3542 AJ 2/15/02 g.
wt. of tube assembly #2 + CDV * 200/~~325~~ 325 * ~~HL * CPT * Naf (old~~ AJ 3/1/01
CDV * 325/200 * UC * WA = 34.8679
wt. of sample added = 34.8679 − 33.3542
= 1.5137 g.
wt. of outgased sample + tube assembly #2
= 34.6930
wt. of outgased sample = 34.6930 − 33.3542
= 1.3388 g.

② wt. of outgased sample with tube assembly #9
= 34.5200 g.
wt. of outgased sample = 34.5200
CDV * 100/200 * CPT * HL * Naf (old 11/2/99 by AJ) − 33.1988
1.3212 g.

Bw
2/13/02

3/2/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NAF-OLD	Start Date	3/2/01 ~~10/30/98~~ 4/10/01 AJ
Customer	CNWRA	Start Time	05:25:42
Operator	JAIN	Elapsed Time	26 min
Sample Wt	1.3212 g	Outgas Time	240 min
Profile	BET5	Outgas Temperature	350 C

CDV* 100/200 * HL*CPT * Naf (old 11/2/99 by AJ)

Summary

Surface Area Report

BET Surface area	11.076 sq.m/g
Correlation Coefficient	0.99999

Surface Area Report

BET Surface area	11.076 sq.m/g
Slope	0.390342
Intercept	0.002601
C_value	151.088
Monolayer Volume	2.5449 cc/g (STP)
Correlation Coefficient	0.99999

One Point BET Surface Area (Ps/Po=0.3) 10.908 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0484	0.021510	2.367
0.0576	0.025128	2.431
0.0667	0.028708	2.491
0.0732	0.031226	2.530
0.0802	0.033911	2.570
0.1005	0.041820	2.672
0.1210	0.049717	2.768
0.1387	0.056623	2.845
0.1603	0.065050	2.935
0.1803	0.072965	3.014
0.2000	0.080873	3.091

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.022118	2.380
0.0800	0.033828	2.571
0.1200	0.049442	2.758
0.1600	0.065055	2.928
0.2000	0.080669	3.099

3/2/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	NAF-OLD	Start Date	3/2/01 ~~10/30/98~~ 4/10/01 AJ
Customer	CNWRA	Start Time	05:25:42
Operator	JAIN	Elapsed Time	26 min
Sample Wt	1.3212 g	Outgas Time	240 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03469
Intercept	0.002901
Correlation Coefficient	0.99998

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	726.88
0.0000	0.338	0.028	727.01
0.0001	0.677	0.073	727.30
0.0004	1.061	0.320	727.46
0.0023	1.451	1.661	727.28
0.0072	1.803	5.238	727.52
0.0153	2.015	11.148	727.65
0.0275	2.180	20.038	727.75
0.0403	2.302	29.346	727.78
0.0484	2.367	35.255	727.73
0.0576	2.431	41.914	727.93
0.0667	2.491	48.587	728.12
0.0732	2.530	53.300	727.95
0.0802	2.570	58.366	728.06
0.1005	2.672	73.194	728.10
0.1210	2.768	88.035	727.81
0.1387	2.845	100.982	727.84
0.1603	2.935	116.707	728.08
0.1803	3.014	131.228	727.93
0.2000	3.091	145.586	727.95

Note: SA of CDV* 100/200 * CPT * HL * Naf Cold prepared by AJ 11/2/99 yielded the same value of ~11.076 m^2/g as previous runs.

3/2/01

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-325	Start Date	3/2/01 ~~10/30/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	06:16:24
Operator	JAIN	Elapsed Time	25 min
Sample Wt	1.3388 g	Outgas Time	240 min
Profile	BET5	Outgas Temperature	350 C

CDV * 325/200 * WA * UC (old)

Summary

Surface Area Report

BET Surface area	9.194 sq.m/g
Correlation Coefficient	0.99998

Surface Area Report

BET Surface area	9.194 sq.m/g
Slope	0.470552
Intercept	0.002865
C_value	165.249
Monolayer Volume	2.1123 cc/g (STP)
Correlation Coefficient	0.99998

One Point BET Surface Area (Ps/Po=0.3) 9.066 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0506	0.026755	1.992
0.0598	0.031106	2.045
0.0662	0.034094	2.079
0.0732	0.037358	2.114
0.0799	0.040493	2.146
0.1005	0.050077	2.231
0.1207	0.059515	2.306
0.1388	0.067919	2.372
0.1597	0.077756	2.443
0.1810	0.087954	2.512
0.2000	0.097220	2.571
0.2035	0.098797	2.586

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.026392	1.994
0.0800	0.040509	2.147
0.1200	0.059331	2.298
0.1600	0.078153	2.437
0.2000	0.096975	2.578

3/2/01

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CDV-325	Start Date	3/2/01 ~~10/30/90~~ AJ 4/10/01
Customer	CNWRA	Start Time	06:16:24
Operator	JAIN	Elapsed Time	25 min
Sample Wt	1.3388 g	Outgas Time	240 min
Profile	BET5	Outgas Temperature	350 C

Isotherm Data

Freespace Calculation

Slope	0.03455
Intercept	0.005692
Correlation Coefficient	0.99997

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	747.80
0.0000	0.333	0.012	747.99
0.0001	0.665	0.077	748.02
0.0009	1.033	0.686	747.88
0.0043	1.371	3.204	748.02
0.0107	1.603	8.007	748.06
0.0206	1.754	15.402	748.15
0.0336	1.875	25.147	748.00
0.0415	1.934	31.028	748.10
0.0506	1.992	37.874	748.35
0.0598	2.045	44.751	748.25
0.0662	2.079	49.523	748.24
0.0732	2.114	54.784	748.45
0.0799	2.146	59.840	748.51
0.1005	2.231	75.174	748.14
0.1207	2.306	90.295	748.23
0.1388	2.372	103.858	748.46
0.1597	2.443	119.464	748.26
0.1810	2.512	135.469	748.60
0.2000	2.571	149.650	748.28
0.2035	2.586	152.311	748.49

SA value of 9.194 m^2/g of CDV * 325/200 * WA * UC
is consistent with SA value (on page 44) obtained on 2/21/01

3/7/01 Copy made to page 53 for QA archives.
AJ

3/20/01 wt. of CDV * 200/325 mesh * UC * WA = 266.79 gm.
AJ

Preparation of 1M NaOAc buffer at PH 5 for removal of Carbonates and other soluble salts:-

① Follow procedure #③ on page 7 of this book for removal of carbonates.

② Follow procedure on page 13 to prepare NaOAc buffer.

- $CH_3COONa \cdot 3H_2O$ (FS, lot # 937077) F.W. 136.08g
- DI H_2O
- CH_3COOH
- Orion 920A PH meter /w probe & PH standards 2, 4 & 7.
- Plastic bottle

① Dissolve ~~205.02~~ 136.08 AJ 3/20/01 g of $CH_3COONa \cdot 3H_2O$ with DI H_2O & dilute it to 1000 ml mark.

② Adjust PH to 5 using glacial CH_3COOH (FS, lot # 971798) vol. of CH_3COOH added ~ 25 ml.

3/21/01 AT ① Set water bath under the hood at 95°C.
② Add ~ 3g of CDV * 200/325 * UC * WA to 50ml centrifuge tubes (~~40~~ 40 AJ 2/15/02 tubes).
③ Add 30 ml of NaOAc buffer to each tube
④ Digest the mixture at ~95°C for about 30 min.

Prepare NaOAc buffer by dissolving 136.08 g of $CH_3COONa \cdot 3H_2O$ in DI H_2O & diluting it to 1000 ml Adjust the PH to 5 with CH_3COOH (~ 25 ml).

3/23/01 AJ Continue removal of Carbonates for CDV * 200/325 * UC * WA. Follow the procedure on page ~~13~~ 7 AJ 3/23/01 to prepare of this book.

24 centrifuge tubes (50 ml capacity) were used, with 3g of CDV * 200/325 * UC * WA and 30 ml of NaOAc buffer. The mixture was digested at 95°C in water bath for about 30 min.
Centrifuge the mixture at 6000 rpm for 5 min. & then 2 times with nanopure water at 6000 rpm for 5 min. Rinse CDV 3-4 times and dry it in oven at 55-60°C.

3/27/01
3/~~27~~28/01 AJ 2/15/02 AJ ① Prepare 250 ml of NaOAc buffer at PH ~5 following procedure on page 13 of this book.
② Continue removal of carbonates from CDV * 200/325 * UC * WA.
③ Remove CDV * 200/325 * WA * UC * RC from drying oven ~ 189 gms. Replace in drying oven at 55°C as ~~it~~ seemed slightly wet.

3/28/01 AJ Rinse CDV * 200/325 * WA * UC * RC several times with nanopure H_2O and place it in the oven for drying ~ 57°C

Remove a batch of CDV* 200/325 *UC*WA*RC from oven.

Heavy liquid Separation :- Sodium Polytungstate (SPT) in solid form will be dissolved with DI H_2O and used as a heavy liquid for density seperation

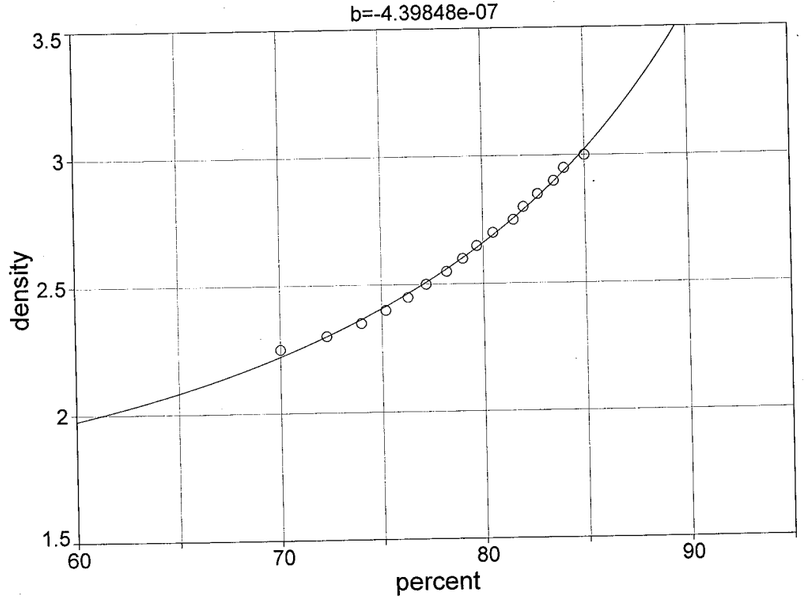
C:\Excel Data\SPT.xls

Rank 1 Eqn 49 $y^{-1}=a+bx^3$

r^2=0.996834 DF Adj r^2=0.996347 FitStdErr=0.0138651 Fstat=4407.55

a=0.601142

b=-4.39848e-07



Rank 1 Eqn 49 $y^{-1}=a+bx^3$

r^2 Coef Det	DF Adj r^2	Fit Std Err	F-value
0.9968336892	0.9963465645	0.0138650748	4407.5495291

Parm	Value	Std Error	t-value	95% Confidence Limits	
a	0.601142316	0.003573694	168.2131617	0.593477505	0.608807127
b	-4.3985e-07	6.80923e-09	-64.5957707	-4.5445e-07	-4.2524e-07

Date	Time	File Source
Mar 28, 2001	1:29:11 PM	c:\excel data\spt.xls

Density data range from 2.25 to 3.0 g/ml was used from the Mixing Chart (from Sometu-USA Product manual). This data was plotted as on page 56 and best fit equation was generated. The density values from 1.67 to 3.57 g/ml was obtained as on page 58-59.

Mixing Chart

D

Mass Percent = $(B/B+C)\times 100$

$(700/1000)\times 100 = 70\%$

72.3

74.0

75.2

76.3

77.2

78.2

79.0

79.7

80.5

81.5

82.0

82.7

83.5

84.0

85.0

Sodium Polytungstate $Na_6(H_2W_{12}O_{40})\cdot H_2O$ [Sometu-USA CAS # 12141-67-2]

Mar 28, 2001 Page 1

	Mass %	Density (g/ml)	
1.	20.00000000000	1.673294206987	Y=F(X)
2.	20.00000000000	1.673294206987	Y=F(X)
3.	21.00000000000	1.674848615390	Y=F(X)
4.	22.00000000000	1.676561679978	Y=F(X)
5.	23.00000000000	1.678441800886	Y=F(X)
6.	24.00000000000	1.680497544074	Y=F(X)
7.	25.00000000000	1.682737658442	Y=F(X)
8.	26.00000000000	1.685171094096	Y=F(X)
9.	27.00000000000	1.687807021885	Y=F(X)
10.	28.00000000000	1.690654854265	Y=F(X)
11.	29.00000000000	1.693724267614	Y=F(X)
12.	30.00000000000	1.697025226101	Y=F(X)
13.	31.00000000000	1.700568007221	Y=F(X)
14.	32.00000000000	1.704363229157	Y=F(X)
15.	33.00000000000	1.708421880104	Y=F(X)
16.	34.00000000000	1.712755349726	Y=F(X)
17.	35.00000000000	1.717375462952	Y=F(X)
18.	36.00000000000	1.722294516298	Y=F(X)
19.	37.00000000000	1.727525316963	Y=F(X)
20.	38.00000000000	1.733081224968	Y=F(X)
21.	39.00000000000	1.738976198606	Y=F(X)
22.	40.00000000000	1.745224843549	Y=F(X)
23.	41.00000000000	1.751842465967	Y=F(X)
24.	42.00000000000	1.758845130061	Y=F(X)
25.	43.00000000000	1.766249720473	Y=F(X)
26.	44.00000000000	1.774074010071	Y=F(X)
27.	45.00000000000	1.782336733694	Y=F(X)
28.	46.00000000000	1.791057668486	Y=F(X)
29.	47.00000000000	1.800257721551	Y=F(X)
30.	48.00000000000	1.809959025741	Y=F(X)
31.	49.00000000000	1.820185044498	Y=F(X)
32.	50.00000000000	1.830960686799	Y=F(X)
33.	51.00000000000	1.842312433375	Y=F(X)
34.	52.00000000000	1.854268475558	Y=F(X)
35.	53.00000000000	1.866858868270	Y=F(X)
36.	54.00000000000	1.880115698921	Y=F(X)
37.	55.00000000000	1.894073274176	Y=F(X)
38.	56.00000000000	1.908768326908	Y=F(X)
39.	57.00000000000	1.924240245930	Y=F(X)
40.	58.00000000000	1.940531331535	Y=F(X)
41.	59.00000000000	1.957687080333	Y=F(X)
42.	60.00000000000	1.975756503397	Y=F(X)
43.	61.00000000000	1.994792482400	Y=F(X)
44.	62.00000000000	2.014852169180	Y=F(X)
45.	63.00000000000	2.035997435073	Y=F(X)
46.	64.00000000000	2.058295377431	Y=F(X)
47.	65.00000000000	2.081818892057	Y=F(X)
48.	66.00000000000	2.106647321800	Y=F(X)
49.	67.00000000000	2.132867193452	Y=F(X)

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	Mass %	Density	
50.	68.00000000000	2.160573057343	Y=F(X)
51.	69.00000000000	2.189868446752	Y=F(X)
52.	70.00000000000	2.220866977622	Y=F(X)
53.	71.00000000000	2.253693613150	Y=F(X)
54.	72.00000000000	2.288486122876	Y=F(X)
55.	73.00000000000	2.325396772115	Y=F(X)
56.	74.00000000000	2.364594285368	Y=F(X)
57.	75.00000000000	2.406266137021	Y=F(X)
58.	76.00000000000	2.450621234832	Y=F(X)
59.	77.00000000000	2.497893077163	Y=F(X)
60.	78.00000000000	2.548343484517	Y=F(X)
61.	79.00000000000	2.602267031110	Y=F(X)
62.	80.00000000000	2.659996334708	Y=F(X)
63.	81.00000000000	2.721908405139	Y=F(X)
64.	82.00000000000	2.788432307268	Y=F(X)
65.	83.00000000000	2.860058467342	Y=F(X)
66.	84.00000000000	2.937350049177	Y=F(X)
67.	85.00000000000	3.020956957983	Y=F(X)
68.	86.00000000000	3.111633208320	Y=F(X)
69.	87.00000000000	3.210258638422	Y=F(X)
70.	88.00000000000	3.317866295141	Y=F(X)
71.	89.00000000000	3.435677295860	Y=F(X)
72.	90.00000000000	3.565145662683	Y=F(X)

3/30/01 AJ — Remove CDV* 200/325 * VC * WA * RC from drying oven · wt. ≈ 68 gm

Total wt. of CDV* 200/325 * VC * WA * RC = 257.00 gm

Preparation of Heavy liquid (Sodium Polytungstate) of density = 2.19 g/ml

① Dissolve 345.0 g of SPT in 155 ml of DI H_2O

0.1 ml SPT = 0.2208 g

② Dissolve another batch of 345.0 g of SPT in 155 ml of DI water and mix with previous batch.

0.1 ml SPT = 0.2207 g

SPT of ρ = 2.207 g/ml

Objective :- To remove impurities heavier than clinoptilolite such as iron oxide & silicate minerals from CDV * 200/325 mesh * UC * WA * RC to be used for ion exchange experiments.

Method :- Density separation using sodium polytungstate (SPT) with different specific gravity.

Equipment & Chemicals :

① Two separatory funnels - 250 ml capacity
② Filter paper (Fisherbrand P4)
③ volumetric flasks
④ pipettes & graduated cylinders.
⑤ Weighing balances (AE 240 accurate to 0.1 mg) & Metler).
⑥ Plastic bottle (500 ml) to store SPT.
⑦ SPT $Na_6(H_2W_{12}O_{40})\cdot H_2O$ (Sometu-USA, Cas # 12141-67-2)
⑧ DI H_2O
⑨ Magnetic stirrer

Procedure : ① Use heavy liquid of density, $\rho = 2.207$ g/ml as prepared on page 59. Pour this liquid (~250 ml) into two separatory funnel. Then add ~ 30 g of CDV * 200/325 * UC * WA * RC to the separatory funnel. Impurities with $\rho > 2.207$ were allowed to settle for about 30 minutes. Clinoptilolite with a density of ~2.16 & lighter impurities stay in suspension.

② Heavier impurities were run out of separatory funnel to a volumetric flask with funnel & filter paper & heavy liquid was recovered to be reused.

③ Clean clinoptilolite was caught on another filter paper & funnel over a volumetric flask. Clinoptilolite was rinsed with more DI water and also separatory funnel was rinsed with wate

④ Steps ①, ② & ③ were repeated until all of CDV * 200/325 * UC * WA * RC was treated.

⑤ Clinoptilolite collected is washed with DI H_2O several times, & then also cleaned ultrasonically.

⑥ Allow it to air dry.

4/3/01 AJ continue removal of heavier impurities from CDV * 200/325 * UC * WA * RC using heavy liquid density separation method.

wt. of container = 197.40 g.

wt. of cleaned CDV & also air dried = 125.87 g.

Prepare another batch of SPT by dissolving 345 g. of solid SPT in 155 ml of nanopure H_2O.

0.1 ml of HL = 0.2236 & 0.2233 g

4/4/01 AJ — Continue heavy liquid density seperation.
CDV dried & cleaned ≈ 130 g.
Total dried & cleaned CDV = 130 + ~~125.87~~ (AJ 4/4/01) + 125.87 (AJ 4/9/01)
= ~~255.87~~ g.
= 255.87 g.

Continue heavy liquid density seperation on previously treated CDV with heavy liquid once (2nd HL treatment).

4/6/01 AJ — Continue density separation method to remove heavier impurities for CDV (using SPT as HL) already cleaned once by the same method (2nd HL).

AJ 4/9/01
4/9/01 AJ — Rinse HL treated CDV with nanopure water several times & then divide it in 2 beakers. Partially fill the beakers with nanopure H_2O & place it in ultrasonic cleaner for about 7 min. Decant the supernatant and repeat the procedure until no turbidity is observed and supernatant is clear. Dry CDV* UC* WA * RC * HL * UC in drying oven at ~65°C.

CDV*UC*WA*RC*HL*UC = 142.50 g. + 93
- ~~235.50~~ (AJ 2/15/02) g
235.50

Removal of Iron-Oxides :- Follow the procedure on page 7 (AJ 4/10/01) of this book.

$NaHCO_3$ (FES lot # 006275)

1 M $NaHCO_3$ soln. :- Dissolve 21.0025 g. in nanopure H_2O & dilute it to 250 ml mark.

0.3 M Na-citrate soln :- Dissolve 88.23 g of $Na_3C_6H_5O_7 \cdot 2H_2O$ in nanopure H_2O & dilute it upto 1000 ml mark.

4/10/01 AJ — Start removal of iron-oxides from CDV using the procedure on page 7.
Water bath temp. ≈ 75-80°C (<80°C)
50 ml capacity centrifuge tubes were used.
5 g of CDV was mixed with 20 ml of 0.3 Na citrate solution and 2.5 ml of 1 M $NaHCO_3$ solution.

4/11/01 AJ — Continue iron-oxide removal from CDV * UC * WA * 200/325 * RC * HL

4/13/01 AJ — Continue iron-oxide removal from CDV * UC * WA * 200/325 * RC * HL.
Rinse CDV * UC * WA * 200/325 * RC * HL * RFe several times with nanopure H_2O & dried in oven at ~55°C overnight.

4/17/01 Continue removal of iron-oxides from CDV*
AJ 200/325 * UC * WA * RC * HL * ~~R~~ AJ 4/17/01 R+

Rinse CDV * 200/325 * UC * WA * RC * HL * R Fe with nanopure H_2O several times and dry it in oven at ≈ 55°C

CDV* 200/325 * UC x WA * RC * HL * RFe = 119.70 g.

objective :- To convert CDV * 200/325 * UC * WA * RC * HL * RFe
- to Naf] for ion exchange experiments using
- to kf]

3M KCl and 3M NaCl solutions.

Method :- Per page 16-17 of this book.

Use 2-500 ml PP (polypropylene) bottles.

~60 g of CDV was mixed with ≈ ~~60~~ AT 4/17/01 250 ml of 3M KCl

~60 g of CDV was mixed with ≈ 400 ml of 3M NaCl

Water bath set at 70°C and 100 rpm.

4/18/01 CDV * 200/325 * UC * WA * RC * HL * RFe = 61.579.
AJ

3M NaCl :-

Dissolve 175.32 g of NaCl (FS lot # ~~07-0198~~ AT 4/18/01 986412) in nanopure H_2O and dilute it to 1000 ml mark.

3M KCl :-

Dissolve 223.68 g of KCl (FS lot # 006242) in nanopure H_2O and dilute it to 1000 ml mark.

Rinse CDV * 200/325 * UC * WA * RC * HL * RFe was rinsed

with nanopure water several times and dry it oven at ≈ 55°C.

4/19/01 3M KCl & 3M NaCl solutions replaced.
AJ

4/20/01
AJ CDV * 200/325 * UC * WA * RC * HL * RFe = 57.45 g

Total CDV chemically pretreated for impurities
= 119.70 + 61.57 + 57.45
= 238.72 g.

3M NaCl :- Dissolve ~~H6.88~~ AJ 4/20/01 87.61 g of NaCl in nanopure H_2O and dilute it to 500 ml.

3M KCl :- Dissolve ~~H9.12~~ AJ 4/20/01 111.84 g. of KCl in nanopure H_2O and dilute it to 500 ml.

4/21/01 Replace 3M KCl & 3M NaCl solutions (water bath ~40°
4/23/01 3M KCl & 3M NaCl solutions replaced & CDV
AJ placed back in water bath at 70°C.

4/25/01 3M KCl & 3M NaCl solutions replaced. Place
AJ the bottles containing CDV & solutions back in water bath at 70°C.

3M NaCl :- Dissolve 87.61 g of NaCl in nanopure

H_2O and dilute it to 500 ml mark.

3M KCl :- Dissolve 111.84 g of KCl in nanopure H_2O and dilute it to 500 ml mark.

4/27/01 AJ — 3M NaCl & 3M KCl solutions replaced and place the bottles back in water bath at 40°C.

3M NaCl :- Dissolve 87.61 g of NaCl (lot #986412) in nanopure water and dilute it to 500 ml mark.

3M KCl :- Dissolve 111.84 g of KCl in nanopure water and dilute it to 500 ml mark.

4/29/01 AJ — Replace 3M NaCl & 3M KCl solutions and place bottles back in water bath at 40°C.

4/30/01 — The temperature of water bath was adjusted back to 70°C.

5/1/01 AJ — The bottles containing chemically pretreated CDV with 3M KCl and one with chemically pretreated CDV with 3M NaCl were removed from water bath. The Naf and Kf of CDV were transferred in two seperate beakers and washed with nanopure water several times. Test the presence of Cl^- in supernatant with 0.1M $AgNO_3$.

Add 1-2 drops of 0.1M $AgNO_3$ to the supernatant and if white precipitate is visible, rinse the powder with nanopure water again until no visible white ppt. is observed.

Note :- After several washing white ppt. was still observed. The powders were then rinsed with warm water (not boiling) twice. Test for Cl^- was negative.

Place Naf-CDV and Kf-CDV in the oven at ~65°C for drying.

5/2/01 AJ — Remove beakers containing Naf-CDV & Kf-CDV from the oven.

① wt. of container w/o lid = 196.60 g

CDV * ~~200~~/325 (AJ 2/15/02) * UC * WA * RC * HL * RFe * Naf = 60.80 g

② wt. of container w/o lid (5/2/01 AJ) = 197.42 g

CDV * 200/325 * UC * WA * RC * HL * RFe * ~~Na~~ Kf = 58.93 g

5/8/01 AJ — Surface Area Analysis on S1 & S2 $CaCO_3$ Samples (prepared by BW)

Objective :- To determine the surface area of S1 & S2 Calcite samples (prepared by BW (AJ 5/9/01) for Np sorption expts.) using Coulter SA 3100.

Equipment and supplies:- ① Coulter SA 3100
② 2 Sample tubes (9cc) w/assembly
③ He & N_2 gas tanks.
④ Liquid N_2
⑤ AE(Metler) 240 weighing Balance
⑥ Weighing paper
⑦ Sample funnel
⑧ S1 & S2 $CaCO_3$ samples.

Procedure:- As per Coulter SA 3100 product manual on page 5-1.

Initial Conditions:- outgas temperature = 150°C
outgas time = 720 min.

① wt. of tube assembly # 9 = 33.2902 g.
wt. of tube assembly + S1 calcite semple = 34.5585 g
wt. of S1- calcite semple added = 34.5585 − 33.2902
= 1.2683 g.

5/09/01 AJ wt. of tube assembly + outgased S1 = 34.5570 g.
wt. of outgased S1 calcite = 34.5570
−33.2902
1.2668 g.

SA = 0.190 m^2/g

② wt. of tube assembly # 2 = 33.4762 g
wt. of tube assembly + S2 calcite semple = ~~34.7250~~ AJ 2/15/02 34.7260 g.

wt. of S2 calcite sample added = 34.7260 − 33.4762
= 1.2498 g.

5/09/01 AJ wt. of tube assembly + outgased S2 $CaCO_3$ = 34.7260 g.
wt. of outgased S2 $CaCO_3$ = 34.7260
−33.4762
1.2498 g.

SA = 0.405 m^2/g

5/09/01
AJ ① wt. of tube assembly # 3 = 33.7495 g.
wt. of tube assembly + S1 $CaCO_3$ added = 36.6427 g
wt. of S1 $CaCO_3$ added = 2.8932 g.

5/11/01 AJ wt. of tube assembly & outgased S1 $CaCO_3$ = 36.6420 g.
wt. of outgased S1 $CaCO_3$ = 36.6420 − 33.7495
= 2.8925 g.

SA = 0.249 m^2/g

② wt. of tube assembly # 9 = ~~33.296~~ AJ 5/11/01 33.3024
wt. of tube assembly + S1 $CaCO_3$ = 36.2826
= 36.2826
−33.3024
2.9802

wt. of tube assembly & outgased S1 $CaCO_3$ = 36.2766
wt. of outgased S1 $CaCO_3$ = 36.2766 − 33.3024
= 2.9042 g.

SA = 0.210 m^2/g

③ wt. of tube assembly #2 = 33.4757
wt. of tube assembly and S2 $CaCO_3$ = 36.3323

wt. of S2 $CaCO_3$ added = 2.8566 g
wt. of tube assembly + outgased S2 $CaCO_3$ = 36.3205
wt. of outgased S2 $CaCO_3$ = 36.3205
− 33.4757
2.8448 g.

SA = ?? m^2/g

The sample was not analysed by SA, tried twice. Would be reanalysed. Couldn't measure freespace.

5/14/01
5/15/01
AJ 2/15/02

① wt. of tube assembly #3 = 33.6957 g.
wt. of tube assembly + S2 $CaCO_3$ added = 35.7047 g
wt. of S2 $CaCO_3$ added = 35.7047
− 33.6957
2.0090 g.

wt. of outgassed S2 $CaCO_3$ & tube assembly = 35.7033 g
wt. of outgassed $CaCO_3$ = 35.7033
− 33.6957
2.0076 g.

SA = 0.242 m^2/g

② wt. of tube assembly #9 = 29.3124 g
wt. of tube assembly + S2 $CaCO_3$ added = 30.4787 g
wt. of S2 $CaCO_3$ added = 30.4787
29.3124
1.1663 + 1663 g.
AJ 2/15/02

wt. of tube assembly + outgassed $CaCO_3$ = 30.4778 g.
AJ 2/15/02

wt. of $CaCO_3$ added = 30.4778 − 29.3124 g
= 1.1654 g.

SA = ??

③ wt. of S2 $CaCO_3$ added = 2.8566
Note!- Reanalysing sample from page 70 (#3)
wt. of tube assembly & outgassed S2 $CaCO_3$ = 36.3191 g
wt. of outgassed $CaCO_3$ = 36.3191
− 33.4757
2.8434 g.

SA = ??

Note!- The 2 samples couldn't be analysed. SA (Coulter 3100) couldn't measure freespace.

5/16/01 AJ The 2 samples of S2 $CaCO_3$ were resen for SA measurements. SA 3100 unable to measure freespace.

BW 2/13/02

5/8/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	S1-CALC	Start Date	~~01/05/91~~
Customer	CNWRA	Start Time	23:02:58
Operator	JAIN	Elapsed Time	22 min
Sample Wt	1.2668 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

AT 05/08/01
5/9/01

Summary

Surface Area Report

BET Surface area 0.190 sq.m/g
Correlation Coefficient 0.98447

Surface Area Report

BET Surface area 0.190 sq.m/g

Slope 22.017804
Intercept 0.884626
C_value 25.889
Monolayer Volume 0.0437 cc/g (STP)
Correlation Coefficient 0.98447

One Point BET Surface Area (Ps/Po=0.3) 0.174 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0502	1.781284	0.030
0.0668	2.295525	0.031
0.0832	2.774756	0.033
0.0998	3.187367	0.035
0.1164	3.615699	0.036
0.1329	3.991978	0.038
0.1822	4.650642	0.048

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.985516	0.027
0.0800	2.646050	0.033
0.1200	3.526762	0.039
0.1600	4.407474	0.043
0.2000	5.288187	0.047

Isotherm Data

Freespace Calculation

Slope 0.02496
Intercept 3.954e-005
Correlation Coefficient 0.99999

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	755.91
0.0119	0.022	9.004	755.96
0.0244	0.026	18.457	755.88
0.0412	0.029	31.121	755.88
0.0502	0.030	37.933	756.01
0.0668	0.031	50.479	755.96
0.0832	0.033	62.908	755.68
0.0998	0.035	75.418	755.51
0.1164	0.036	87.947	755.61
0.1329	0.038	100.422	755.73
0.1822	0.048	137.708	756.00
0.2312	0.061	174.833	756.06

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	S2-CALC	Start Date	~~5/7/01~~ ~~01/06/91~~ AT
Customer	CNWRA	Start Time	00:04:16
Operator	JAIN	Elapsed Time	38 min
Sample Wt	1.2498 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

5/08/01

Summary

Surface Area Report

BET Surface area 0.405 sq.m/g
Correlation Coefficient 0.93287

Surface Area Report

BET Surface area 0.405 sq.m/g

Slope	9.206561
Intercept	1.535386
C_value	6.996
Monolayer Volume	0.0931 cc/g (STP)
Correlation Coefficient	0.93287

One Point BET Surface Area (Ps/Po=0.3) 0.304 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0489	1.778645	0.029
0.0600	1.993075	0.032
0.0766	2.231323	0.037
0.0814	2.360843	0.038
0.0998	2.561748	0.043
0.1162	2.702238	0.049
0.1211	2.806254	0.049
0.1399	2.964120	0.055
0.1893	3.013387	0.077

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.995714	0.026
0.0800	2.271911	0.038
0.1200	2.640173	0.052
0.1600	3.008436	0.063
0.2000	3.376698	0.074

Isotherm Data

Freespace Calculation

Slope	0.03419
Intercept	0.0143
Correlation Coefficient	0.99992

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	756.46
0.0121	0.014	9.148	756.50
0.0227	0.020	17.164	756.53
0.0399	0.027	30.186	756.25
0.0489	0.029	36.981	756.35
0.0600	0.032	45.402	756.16
0.0766	0.037	57.966	756.27
0.0814	0.038	61.592	756.32
0.0998	0.043	75.479	756.33
0.1162	0.049	87.908	756.33
0.1211	0.049	91.600	756.38
0.1399	0.055	105.821	756.25
0.1893	0.077	143.188	756.42
0.2386	0.105	180.484	756.37

5/9/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
			5/09/01 AJ 5/11/01
Sample ID	S1-CAL#3	Start Date	~~01/07/91~~
Customer	CNWRA	Start Time	23:08:02
Operator	JAIN	Elapsed Time	25 min
Sample Wt	2.9042 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

Summary

Surface Area Report

BET Surface area	0.210 sq.m/g
Correlation Coefficient	0.99862

Surface Area Report

BET Surface area 0.210 sq.m/g

Slope	20.344821
Intercept	0.345188
C_value	59.938
Monolayer Volume	0.0483 cc/g (STP)
Correlation Coefficient	0.99862

One Point BET Surface Area (Ps/Po=0.3) 0.202 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0508	1.324723	0.040
0.0623	1.590266	0.042
0.0799	1.990259	0.044
0.0975	2.361962	0.046
0.1150	2.727950	0.048
0.1325	3.098921	0.049
0.1849	4.039340	0.056

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.362430	0.039
0.0800	1.972774	0.044
0.1200	2.786567	0.049
0.1600	3.600360	0.053
0.2000	4.414153	0.057

Isotherm Data

Freespace Calculation

Slope	0.03196
Intercept	0.00112
Correlation Coefficient	0.99999

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	757.05
0.0111	0.029	8.413	757.10
0.0240	0.035	18.154	757.10
0.0415	0.039	31.400	756.95
0.0508	0.040	38.477	757.17
0.0623	0.042	47.160	757.19
0.0799	0.044	60.477	756.73
0.0975	0.046	73.723	756.43
0.1150	0.048	86.988	756.59
0.1325	0.049	100.261	756.52
0.1849	0.056	139.982	756.49
0.2370	0.064	179.302	756.41

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
			5/09/01
Sample ID	S1-CAL#2	Start Date	~~5/10/01~~ AJ
Customer	CNWRA	Start Time	22:31:59 5/11/01
Operator	JAIN	Elapsed Time	19 min
Sample Wt	2.8925 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

Summary

Surface Area Report

BET Surface area	0.249 sq.m/g
Correlation Coefficient	0.99948

Surface Area Report

BET Surface area	0.249 sq.m/g
Slope	17.236763
Intercept	0.232342
C_value	75.187
Monolayer Volume	0.0572 cc/g (STP)
Correlation Coefficient	0.99948

One Point BET Surface Area (Ps/Po=0.3) 0.242 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0505	1.072169	0.050
0.0621	1.288250	0.051
0.0798	1.615347	0.054
0.0975	1.934194	0.056
0.1152	2.243921	0.058
0.1328	2.545635	0.060
0.1858	3.400754	0.067

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.094181	0.048
0.0800	1.611283	0.054
0.1200	2.300754	0.059
0.1600	2.990224	0.064
0.2000	3.679695	0.068

Isotherm Data

Freespace Calculation

Slope	0.03166
Intercept	-0.003761
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	753.05
0.0105	0.037	7.910	753.39
0.0236	0.044	17.743	753.26
0.0410	0.048	30.914	753.42
0.0505	0.050	38.004	753.19
0.0621	0.051	46.754	753.36
0.0798	0.054	60.115	753.48
0.0975	0.056	73.442	753.34
0.1152	0.058	86.809	753.37
0.1328	0.060	100.076	753.31
0.1858	0.067	139.962	753.24
0.2385	0.075	179.635	753.34

5/14/01
AJ

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	S2-CAL#1	Start Date	~~01/11/01~~ 5/14/01 AJ
Customer	CHWRA	Start Time	22:35:20 5/15/01
Operator	JAIN	Elapsed Time	18 min
Sample Wt	2.0076 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

Summary

Surface Area Report

BET Surface area	0.242 sq.m/g
Correlation Coefficient	0.99921

Surface Area Report

BET Surface area	0.242 sq.m/g
Slope	17.779207
Intercept	0.184347
C_value	97.444
Monolayer Volume	0.0557 cc/g (STP)
Correlation Coefficient	0.99921

One Point BET Surface Area (Ps/Po=0.3) 0.237 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.039341	0.051
0.0614	1.258824	0.052
0.0784	1.583934	0.054
0.0955	1.904603	0.055
0.1126	2.217977	0.057
0.1296	2.523301	0.059
0.1808	3.355061	0.066

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.073307	0.049
0.0800	1.606683	0.054
0.1200	2.317852	0.059
0.1600	3.029020	0.063
0.2000	3.740188	0.067

Isotherm Data

Freespace Calculation

Slope	0.0334
Intercept	-0.004231
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	752.10
0.0110	0.041	8.240	752.29
0.0236	0.046	17.791	752.37
0.0408	0.049	30.715	752.47
0.0500	0.051	37.596	752.28
0.0614	0.052	46.158	752.32
0.0784	0.054	59.008	752.32
0.0955	0.055	71.840	752.26
0.1126	0.057	84.696	752.26
0.1296	0.059	97.533	752.33
0.1808	0.066	135.994	752.25
0.2317	0.075	174.353	752.37

5/18/01 AJ Test Coulter SA3100 performance. BW investigate for possible leaks and fixed 3 leaks:-

① leak between N_2 tank regulator & delivery line.

② N_2 gas leak between delivery line and hook up to SA3100.

③ He gas leak between delivery line and hook up to SA3100.

Also, the O rings were coated with high vaccum grease (all O rings at the sample ports of outgas and analysis port).

① wt. of tube assembly #2 = 33.4757

wt. of tube assembly #2 w/S2 $CaCO_3$ = 36.3275g.

wt. of S2 $CaCO_3$ added = 36.3275
− 33.4757
2.8518 g.

wt. of tube assembly + outgased S2 $CaCO_3$
= 36.3275 g.

wt. of outgassed $CaCO_3$ AJ 5/18/01 S2 sample
= 2.8518 g.

Initial Conditions :-

outgas temperature = 150°C

outgas time = 45 min.

SA = 0.237 m^2/g

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	S2-CAL#2	Start Date	5/18/01 ~~01/15/91~~ AJ 5/18/01
Customer	CNWRA	Start Time	01:38:41
Operator	JAIN	Elapsed Time	19 min
Sample Wt	2.8518 g	Outgas Time	45 min
Profile	BET5	Outgas Temperature	150 C

Summary

Surface Area Report

BET Surface area	0.237 sq.m/g
Correlation Coefficient	0.99938

Surface Area Report

BET Surface area	0.237 sq.m/g
Slope	18.127007
Intercept	0.263573
C_value	69.774
Monolayer Volume	0.0544 cc/g (STP)
Correlation Coefficient	0.99938

One Point BET Surface Area (Ps/Po=0.3) 0.229 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0505	1.145223	0.046
0.0620	1.372075	0.048
0.0795	1.712217	0.050
0.0970	2.046844	0.053
0.1145	2.367765	0.055
0.1320	2.683691	0.057
0.1847	3.573094	0.063

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.169923	0.045
0.0800	1.713733	0.051
0.1200	2.438813	0.056
0.1600	3.163894	0.060
0.2000	3.888974	0.064

Isotherm Data

Freespace Calculation

Slope	0.03227
Intercept	-0.003879
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	748.98
0.0107	0.035	8.032	748.89
0.0236	0.041	17.653	748.88
0.0411	0.045	30.795	748.79
0.0505	0.046	37.849	748.93
0.0620	0.048	46.417	748.91
0.0795	0.050	59.512	749.02
0.0970	0.053	72.682	748.94
0.1145	0.055	85.749	749.05
0.1320	0.057	98.879	748.97
0.1847	0.063	138.351	748.93
0.2371	0.072	177.526	748.79

5/22/01 outgas temperature = 150°C, outgas time = 720 min.
AJ ① wt. of tube assembly #2 = 33.4757 g.
wt. of tube assembly + S2 $CaCO_3$ = 36.3281
wt. of S2 $CaCO_3$ added = 36.3281
− 33.4757
2.8524 g.

5/23/01 AJ wt. of tube assembly + outgassed S2 $CaCO_3$
= 36.3275 g.
wt. of outgassed S2 $CaCO_3$ = 36.3275
− 33.4757
2.8518

SA (S2 $CaCO_3$) = 0.232 m^2/g

② wt. of tube assembly #E = 29.3124 g.
wt. of tube assembly + S2 $CaCO_3$ added = 30.4499 g.
wt. of S2 $CaCO_3$ added = 30.4499
29.3124
1.1375 g.

5/23/01 AJ wt. of S2 $CaCO_3$ (outgassed) + tube assembly
= 30.4476 g.
wt. of of outgassed S2 $CaCO_3$ = 30.4476
− 29.3124
1.1352 g.

SA = couldn't be measured. due to ~~faulty~~ AJ 5/23/01

Note: 3 cc sample holder used for this run & ~~possibly~~ ~~Andy~~ AJ 5/23/01 inappropriate for small surface area measurements.

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	S2-CAL#4	Start Date	5/23/01 ~~01/19/01~~ AJ 5/23/01
Customer	CNWRA	Start Time	22:53:01
Operator	JAIN	Elapsed Time	19 min
Sample Wt	2.9513 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	150 C

Summary

Surface Area Report

BET Surface area	0.232 sq.m/g
Correlation Coefficient	0.99944

Surface Area Report

BET Surface area	0.232 sq.m/g
Slope	18.459775
Intercept	0.272853
C value	68.655
Monolayer Volume	0.0534 cc/g (STP)
Correlation Coefficient	0.99944

One Point BET Surface Area (Ps/Po=0.3) 0.225 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0507	1.175466	0.045
0.0621	1.404899	0.047
0.0795	1.747015	0.049
0.0970	2.084432	0.052
0.1145	2.415972	0.054
0.1319	2.734722	0.056
0.1843	3.637419	0.062

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	1.195842	0.044
0.0900	1.749635	0.050
0.1200	2.488026	0.055
0.1600	3.226417	0.059
0.2000	3.964808	0.063

Isotherm Data

Freespace Calculation

Slope	0.03218
Intercept	-0.003885
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	752.80
0.0108	0.033	8.149	752.78
0.0237	0.040	17.861	752.64
0.0412	0.044	30.997	752.60
0.0507	0.045	38.126	752.72
0.0621	0.047	46.720	752.65
0.0795	0.049	59.833	752.66
0.0970	0.052	73.035	752.77
0.1145	0.054	86.165	752.67
0.1319	0.056	99.273	752.75
0.1843	0.062	138.801	752.96
0.2365	0.070	178.053	752.92

Bus 2/13/02

5/23/01 Preparation of 0.05 N NaCl-KCl solutions for Ion-Exchange Experiments

Objective :- To prepare various KCl-NaCl solutions with a total normality of 0.05 N and a fixed K/Na ratio.

Equipment and Supplies :- 7-1L polypropylene Nalgene bottles
1000 ml volumetric flasks.
plastic dropper
magnetic stirrers & stir plate
250 ml beakers
NaCl (FS lot # 986412)
KCl (FS lot # 006242)
Mettler AE240 weighing balance
Nanopure water (>17 mΩ)

Procedure :-

1. Prepare 1000 ml of KCl-NaCl aqueous mixture with a total normality of 0.05 N and a fixed K/Na ratio by taring reagent grade KCl and NaCl in the amounts given in Table 1 onto a clean (acid-washed) 250-ml or 500-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

Decant the solution into a clean 1000 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 1000 ml polypropylene bottle. Label the bottle (e.g., KCl/NaCl*0.05N*0.1X_{Na}, plus Date and Initial).

Table 1

$E_{Na,i}$ (0.05 N) (KCl-NaCl soln.)	Wt. NaCl needed for 1000 ml	Wt. KCl needed for 1000 ml	Wt. NaCl used	Wt. KCl used
0.1	0.2922	3.3548	0.2923	3.3547
0.2	0.5844	2.9820	0.5845	2.9822
0.3	0.8766	2.6093	0.8767	2.6094
0.4	1.1689	2.2365	01.1688	2.2365
0.5	1.4611	1.8638	1.4611	1.8639
0.7	2.0455	1.1183	2.0456 ~~2.0456~~ AJ 2/15/02	1.1184
1	2.9222	0.0000	2.9224	0.0000

Zeolite to be used – CDV * 200/325 mesh * UC * WA * RC * HL * RFe * Kf

Procedure (Continued):-

2. Acid wash 1-15 ml, 16-25 ml and 3-50 ml capacity Nalgene polypropylene (PP) bottles and rinse them. After rinsing, dry ~~it~~ in oven @ 50°C. AJ 2/15/02

3. Place a clean bottle on a balance and tare it. Then add CDV * 200/325 * UC * WA * RC * HL * RFe * Kf to the bottle as given amount in Table 2.

4. Once weight has been recorded, tare again. Pipet KCl/NaCl solution as given volume in Table 2 and add to the bottle. Record the weight of solution.

AJ 5/30/01

EXPERIMENT KCl/NaCl-0.05N

Kf-CDV Table 2

Mixture # KNa05*	Mole fraction Na, XNa,ito use	Weight zeol. to use (gm)	Weight zeol. used (gm)	Volume soln. used (ml)	Weight soln. used (gm)	Calc.Na ppm,i	Calc.Na ppm,f	Calc.K ppm,i	Calc.K ppm,f
1	0.1	0.1103	0.1102	10	~~10.0760~~ 10.0760 AJ 5/17/02	115	11	1759	1935
2	0.1	0.1634	0.1633	25	25.0464	115	23	1759	1916
3	0.1	0.1072	0.1074	25	25.0370	115	34	1759	1896
4	0.2	0.1802	0.1803	25	25.0217	230	57	1564	1857
5	0.2	0.1277	~~0.1278~~ 0.1278 AJ 2/15/02	25	25.0282	230	86	1564	1808
6	0.2	0.1053	0.1054	25	25.0804	230	103	1564	1779
7	0.3	0.1871	0.1871	25	25.0397	345	115	1368	1759
8	0.3	0.1488	0.1488	25	25.0547	345	149	1368	1701
9	0.3	0.1259	0.1260	25	25.0583	345	172	1368	1662
10	0.4	0.1797	0.1798	25	25.0777	460	207	1173	1603
11	0.4	0.1592	0.1592	25	25.0565	460	230	1173	1564
12	0.4	0.1225	0.1228	25	25.0302	460	276	1173	1486
13	0.5	0.1485	0.1485	25	25.0568	575	345	977	1368
14	0.5	0.1081	0.1084	25	25.0526	575	402	977	1271
15	0.5	0.1409	0.1410	50	50.0262	575	460	977	1173
16	0.7	0.1369	0.1370	25	25.0244	805	575	586	977
17	0.7	0.1332	0.1334	50	50.0374	805	690	586	782
18	1	0.1956	0.1958	25	25.0153	1150	805	0	586
19	1	0.1277	0.1280	25	25.0415	1150	920	0	391
20	1	0.1251	0.1253	50	50.0272	1150	1035	0	195

Cover the samples with caps and place on gyratory shaker for equilibrium.

X_{Na} represents mole fraction Na $= \frac{m_{Na}}{m_{Na} + m_K}$

JW 2/13/02

6/1/01 Cole-Palmer Potassium Combination Electrode
AJ (27502-~~38~~ 39) AJ 6/11/01

Objective:- Test the operation of combination K^+ electrode for measurements of ion-exchange experiments.

Supplies:- ① Combination K^+ electrode (Cole-Parmer)
② 0.1M NaCl electrode filling solution
③ syringe to fill the electrode (Plastic)
④ 10^{-2} M K^+ std. to condition the electrode and for storage.
⑤ 0.1M K^+ std. (Orion ionplus 921906)
⑥ Orion 920A pH/mV/ISE/°C meter.

Procedure:-

1. Fill the electrode filling solution (0.1M NaCl) shipped with the electrode to a level just below the fill hole. Use a plastic syringe shipped with electrode.

2. Immerse the potassium electrode in 10^{-2} M K^+ std. for about 30 minutes to condition the electrode before first use.

3. Using procedure for checking slope on page 22 of this lab book.

1^{st} mV reading = -46.0 mV
2^{nd} mV reading = 11.8 mV

Difference = 11.8 − (−46.0) mV
= 57.8 mV [between 54-60 mV]
O.K.

4. Dilute 0.1M K^+ std. to 0.01M K^+ std. by adding 1ml of 0.1M K^+ std. to ~~dist~~ AJ 6/11/01 nanopure water & dilute it to 10 ml.
Add 0.2 ml ISA to the sample. (2ml ISA / 100 ml sample)

3 point Calibration (1^{st})

K^+ std.	Theo. Value	Measured	slope
0.001M	39.1 PPm	~~38~~ 99 AJ 6/11/01 36	62.4 mV
0.01M	391 PPm	~~38~~ 0 AJ 6/11/01 380	
0.1M	3910 PPm	~~3~~ 6 AJ 6/11/01 3899	

3 Point Calibration (2^{nd})

K^+ std.	Theo. Value	Measured	slope
0.001M	39.1 PPm	~~38~~ 99 AJ 6/11/01 36	62.4 mV
0.01M	391	380	
0.1M	3910	~~3~~ 6 AJ 6/11/01 3899	

6/5/01 3 Point Calibration
AJ

K^+ std.	The. Value	Meas. Value	
0.001 M	39.1 PPm	37	slope
0.01 M	391	390	63.1 mV
0.1 M	3910	3899	

Objective!- To test the electrode (Orion Ross Sure-Flow Na^+ electrode model 86-11) operation for Na^+ ion concentration measurements required for ion-exchange experiments.

Supplies!-
1. Orion Ross sodium electrode 86-11
2. 2M NH_4Cl reference electrode filling solution
3. sodium ionic strength adjustor (ISA)
4. Sodium Electrode storage solution
5. Orion Model 920A pH/mV/ISE/°C meter
6. ~~AJ 6/5/01~~ magnetic stir bars
7. Na standard solutions

Procedure!-

1. Electrode Rinse solution: Add 5 ml of ISA to a 500 ml squeeze bottle and filling it with nanopure water. Use this solution to rinse Na electrode between measurements.

2. Prepare the electrode as follows:

USING THE ELECTRODE

Figure 2
Filling The ROSS SURE-FLOW Sodium Electrode

ISA: To keep constant background ionic strength and adjust pH, add 10ml of ISA per 100 ml of sample.

3. Follow the following instructions to check the electrode operation after conditioning the electrode membrane.

6/6/01 using 1000 ppm Na^+ std. (Orion 841108)
AJ

1st mV reading = -119.8 mV (1 ml)
2nd mV reading = -70.1 (10 ml)
49.7 mV

slope doesn't lie between 54 and 60 mV.
Recondition the electrode in reconditioning sodium solution (Orion # 841113) for 30 sec. and then soak the electrode in storage solution for 15 min.
Repeat the above procedure:

1st mV reading = -125.8 mV
2nd mV reading = -71.5
slope = difference = 54.3 mV.

3 point Calibration: Na^+

	measured Value
10 PPM	10
100	99
1000	1000

slope = 58.9 mV

6/6/01 6/6/01

Mixture # KNa05*	XNa,i to use	Calc.Na ppm,i	Meas. Na ppm,l	Calc.K ppm,i	Meas. K ppm,l
1	0.1	115	120	1759	2500
2	0.1	115		1759	
3	0.1	115		1759	
4	0.2	230	230	1564	2200
5	0.2	230		1564	
6	0.2	230		1564	
7	0.3	345	350	1368	1900
8	0.3	345		1368	
9	0.3	345		1368	
10	0.4	460	AJ 2/15/02 ~~460~~ 460	1173	1600
11	0.4	460		1173	
12	0.4	460		1173	
13	0.5	575	AJ 2/15/02 ~~580~~ 580	977	1300
14	0.5	575		977	
15	0.5	575		977	
16	0.7	805	810	586	AJ 2/15/02 ~~810~~/820
17	0.7	805		586	PB 2/15/02 ~~810~~/820
18	1	1150	1200	0	1.1
19	1	1150		0	
20	1	1150		0	

Calibration for K^+ :- 0.1M
0.05M 1950 ppm
0.025M 975 ppm
0.01M 390 ppm

Calibration for Na^+ :- 100 ppm
500 ppm
1000 ppm

K^+ std. solutions :

0.05M or 1950 ppm : $\frac{0.1M \times 50ml}{100\ ml}$

0.025M or 975 ppm : $\frac{0.1M \times 25\ ml}{100\ ml}$

0.01M or 390 ppm : $\frac{0.1M \times 10\ ml}{100\ ml}$

2-13-02 MW

entries were made on 2 dates shown { AJ 6/12/01 / AJ 6/8/01 }

6/15/01 AJ
6/13/01 AJ

Mixture # KNa05*	XNa,i to use	Calc.Na ppm,i	Meas.Na ppm,f diluted	Meas. Na ppm,f	Calc.K ppm,i	Meas. K ppm,f diluted	Meas. K ppm,f
1	0.1	115	10	100	1759	174	1740
2	0.1	115	10	100	1759	172	1720
3	0.1	115	10	100	1759	172	1720
4	0.2	230	23	230	1564	208	2080
5	0.2	230	24	240	1564	207	2070
6	0.2	230	23	230	1564	205	2050
7	0.3	345	34	340	1368	177	1770
8	0.3	345	35	350	1368	177	1770
9	0.3	345	35	350	1368	175	1750
10	0.4	460	44	440	1173	152	1520
11	0.4	460	45	450	1173	152	1520
12	0.4	460	45	450	1173	154	1540
13	0.5	575	55	550	977	128	1280
14	0.5	575	54	540	977	126	1260
15	0.5	575	54	540	977	127	1270
16	0.7	805	73	730	586	82.5	825
17	0.7	805	77	770	586	96.5	965
18	1	1150	100	1000	0	23.8	238
19	1	1150	100	1000	0	17.4	174
20	1	1150	110	1100	0	9.86	98.6

Calibration :- Na^+
10 ppm
25
50

Meas.
11 ppm (6/12/01)
25
50

6/13/01 AJ — K^+ calibration :-

	Theor. Value	meas.
0.005 M	195.5 ppm	~~196.0~~ (AJ 6/15/01) 196
0.0025 M	97.7 ppm	196 (AJ 6/13/01) 98.6

6/15/01 AJ — K^+ :-

Calibration :-

	Th. Value	Measured
0.005M	97.7 PPm	97.7
0.0025 M	195.5	196
0.01 M	391	391
0.001 M	39.1	39.1
0.0001 M	3.91	4.02

Measurements on K^+ conc. for samples # 7 to 20.

6/19/01 AJ — K ppm values were not accurate as expected. Initial experimental solutions will be reanalyzed using 5-point calibration.

Std. solutions required :

0.001 M or 39.1 ppm : $\frac{0.1M \times 1ml}{100\ ml}$

0.01 M or 391 ppm : $\frac{0.1M \times 10\ ml}{100\ ml}$

0.02 M or 782 ppm : $\frac{0.1M \times 20ml}{100\ ml}$

0.05M or 1955 ppm : $\frac{0.1M \times 50\ ml}{100\ ml}$

Calibration : 5 point

K^+ Std. solution	Theor. ppm	measured ppm	
0.001 M	39.1	38.8	
0.01 M	391	388	slope = 55.6 mV
0.02 M	782	776	
0.05 M	1955	1940	
0.1 M	3910	3880	

2nd Calibration :- 5-point

K^+ Std. solution	Theor. ppm	measured ppm	
0.001 M	39.1	39.3	
0.01 M	391	388	slope =
0.02 M	782	776	55.9 mV
0.05 M	1955	1940	
0.1 M	3910	3890	

Note : slope is slightly low, possibly due to averaging over entire range. Slope is usually between (AJ 2/15/02) 58 ± (AJ 6/19/01) 2 mV

Mixture # KNa05*	XNa,i to use	Calc.Na ppm,i	Meas.Na ppm,i	Calc.K ppm,i	Meas. K ppm,i
1 to 3	0.1	115		1759	2440
4 to 6	0.2	230		1564	2150
7 to 9	0.3	345		1368	1879
10 to 12	0.4	460		1173	1630
13 to 15	0.5	575		977	1340
16 to 17	0.7	805		586	815
18 to 20	1	1150		0	0.000

Preparation of 0.05N CsCl/NaCl solutions for Ion Exchange Experiments

objective:- To prepare various CsCl - NaCl solutions with a total normality of 0.05N and a fixed Cs/Na ratio.

Equipments and Supplies:- ~~5~~ 5 AJ 2/15/02 - 1L polypropylene Nalgene bottles
1000 ml volumetric flasks
Plastic dropper
Magnetic stirrers & stir plate
~~5~~ 5 2/15/02 - 250 ml beakers
Mettler AE 240 weighing balance
Nanopure water (>17 mΩ)
NaCl (FS Lot # 986412)
CsCl (FS Lot # 010406)

Procedure:

1. Prepare 1000 ml of CsCl-NaCl aqueous mixture with a total normality of 0.05 N and a fixed Cs/Na ratio by taring reagent grade CsCl and NaCl in the amounts given in Table 1 onto a clean (acid-washed) 250-ml or 500-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

(Note: Instead of 250-mL or 500 mL beaker, one can use a weighing boat for taring the reagent. This procedure can be modified as needed).

Decant the solution into a clean 1000 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 1000 ml polypropylene bottle. Label the bottle (e.g., CsCl/NaCl*0.05N*0.1E_{Cs}, plus Date and Initial).

6/22/01 AJ

Table 1

ECs,i (0.05 N) (CsCl-NaCl soln.)	Wt. CsCl needed for 250 ml	Wt. NaCl needed for 250 ml	6/22/01 Wt. CsCl used (gm)	6/22/01 Wt. NaCl used (gm)
0.05	0.1052	0.6940	0.1054	0.6941
0.2	0.4209	0.5844	0.4210	0.5845
0.4	0.8418	0.4383	0.8419	0.4383
0.6	1.2627	0.2922	1.2628	0.2923
1	2.1045	0	2.1045	0

Procedure (continued):-

2. Acid wash 1 - 15ml, 12 - 25 ml and 6 - 50ml capacity Nalgene polypropylene (PP) bottles and rinse them. After rinsing, dry in the oven @ 50°C. until dry.

3. Place a clean bottle on a balance and tare it. Then add CDV * 200/325 * UC * WA * RC * HL * RFe * Naf to the bottle as given amount in Table 2

4. Once weight has been recorded, tare again. Pipet CsCl ~~CsCl~~ AJ 2/15/02/NaCl solution as given volume in Table 2 and add to the bottle. Record the weight of the solution.

BW 2/13/02

6/26/01

Table 2

Mixture # CsNa-05	ECs,I to use	Weight Na-zeol. to use (gm)	Weight Na-zeol. used (gm)	Volume soln. to use (ml)	Weight ~~Volume~~ of soln.used ~~(ml)~~ AJ 6/26/01	Weight of soln. used (gm)
1	0.05	0.122	0.1222	10	10.0276	
2	0.05	0.1502	0.1502	25	25.0215	
3	0.05	0.0964	0.~~0964~~ 0.0964 AJ 2/13/02	25	25.0118	
4	0.05	0.1323	0.1322	50	50.0208	
5	0.2	0.2266	0.2267	25	25.0228	
6	0.2	0.1714	0.1714	25	25.0176	
7	0.2	0.1499	0.1499	25	25.0308	
8	0.2	0.1152	0.1152	25	25.0464	
9	0.2	0.1769	0.1769	50	50.0160	
10	0.2	0.1131	0.1130	50	50.0398	
11	0.4	0.1661	0.1661	25	25.0611	
12	0.4	0.1066	0.1067	25	25.0420	
13	0.4	0.1404	0.1404	50	50.0492	
14	0.6	0.1637	0.1637	25	25.0855	
15	0.6	0.1102	0.1102	25	25.0570	
16	0.6	0.093	~~0.0934~~ AJ 2/15/02	0.0931 50	50.0568	
17	1	0.2245	0.2245	25	25.1384	
18	1	0.1242	0.1243	25	25.1461	
19	1	0.0674	0.0673	50	50.2660	

6/27/01

AJ Prepare 250 ml of CsCl-NaCl * 0.05N * 0.2 Ecs solution using Table 1 on page 99.

The solutions (initial CsCl/NaCl) were not spiked with Cs-137.

0.05 Ecs i, 0.~~0~~6 Ecs i, 0.4 Ecs i & 1.0 Ecs i — 0.25 ml of Cs 137 spike

0.20 Ecs i → 0.5 ml of Cs-137 spike [1.48 uCi/g, 369/029 PB]

6/27/01 6/29/01

Mixture # CsNa-05	ECs,I to use	Weight Na-zeol. to use (gm)	Weight Na-zeol. used (gm)	Volume soln. to use (ml)	Weight of soln. used (gm)
1	0.05	0.1220	0.1222	10	10.0218
2	0.05	0.1502	0.1504	25	25.0068
3	0.05	0.0964	0.0964	25	25.0190
4	0.05	0.1323	0.1323	50	~~49.9215~~ AJ 2/15/02 49.9216
5	0.2	0.2266	0.2268	25	25.0412
6	0.2	0.1714	0.1714	25	25.0518
7	0.2	0.1499	0.1500	25	25.0389
8	0.2	0.1152	0.1152	25	25.0398
9	0.2	0.1769	0.1770	50	50.0144
10	0.2	0.1131	0.1132	50	50.0128
11	0.4	0.1661	0.1662	25	25.0402
12	0.4	0.1066	0.1065	25	25.0416
13	0.4	0.1404	0.1403	50	50.0517
14	0.6	0.1637	0.1636	25	25.0717
15	0.6	0.1102	0.1102	25	25.0593
16	0.6	0.0930	0.0933	50	50.0749
17	1	0.2245	0.2245	25	25.1151
18	1	0.1242	0.1241	25	25.~~0418~~ 1236 AJ 6/29/01
19	1	0.0674	0.0675	50	50.2363

BAJ 2/13/02

0.5 ml of Vial + 0.1M HNO_3

	Initial wt.	Final wt.	wt. of sample added
0.05 Ecsia	7.8251	8.3222	
0.05 Ecsib	7.7652	8.2642	
0.02 ~~Ecsia~~ AJ 2/15/02 Ecsiaa	N/A	N/A	
0.02 Ecsiab	N/A	N/A	
0.02 Ecsiba	7.7910	8.2812	
0.02 Ecsibb	7.8147	8.3017	
0.4 Ecsia	7.7909	8.2878	
0.4 Ecsib	7.7549	8.2523	
0.6 Ecsia	7.8022	8.2991	
0.6 Ecsib	7.8034	8.2995	
1.0 ~~1.0~~ A.J 2/15/02 Ecsia	7.8181	8.3151	
1.0 Ecsib	7.8439	8.3425	

7/11/01 AJ

Na^+ Std. Solutions:

500 PPM : $\frac{100\ PPm \times \text{~~500ml~~}\ AJ\ 2/15/02\ 500ml}{100\ ml}$

250 PPM : $\frac{100\ PPm \times 250ml}{100\ ml.}$

750 PPM : $\frac{100\ PPm \times 750}{100\ ml}$

7/11/01

7/12/01

Mixture # CsNa-05	ECs,I to use	Calc. Na ppm,initial	Meas. Na ppm,initial (7/11/01)	Calc. Na ppm,f	diluted Meas. Na ppm,f
1	0.05	1092		1149	112
2	0.05	1092	1090	1148	112
3	0.05	1092		1146	108
4	0.05	1092		1142	110
5	0.2	920		1132	110
6	0.2	920		1113	AJ ~~109~~ 109 2/15/02
7	0.2	920		1100	108
8	0.2	920	924	1071	103
9	0.2	920		1042	101
10	0.2	920		1002	97.6
11	0.4	690		945	91.1
12	0.4	690	691	862	83.1
13	0.4	690		806	74.6
14	0.6	460		736	67.5
15	0.6	460	460	650	62.7
16	0.6	460		542	55.6
17	1	0		404	38.4
18	1	0		228	20.9
19	1	0		63	14.8

Following the procedure on page 92, check the Na^+ electrode operation. Use 1000 ppm std.

I^{st} mV reading = $-12\text{~~8~~}\ AJ\ 2/15/02\ mV$ -123.8 mV

2^{nd} mV reading $= -71.3$ mV

Difference $= 52.5$ mV ($< 54-60$ mV)

Soak the electrode for 30 sec. in Na reconditioning solution.

I^{st} mV reading $= -124.3$ mV

2nd mV reading = -71.0 mV

Difference = -124.3 + 71.0 = 53.3 mV

Note!- The above readings are not correct as meter ~~mater~~ AJ 2/15/02 was set at K^+ conc. The meter was changed for Na^+ and slope was remeasured as follows:

1st mV readings = -123.8 mV

2nd mV reading = AJ 2/15/02 -70.3 mV

Difference = 53.5 mV

3 pt calibration

Na^+ std.	Th. Value	Meas. Value
250 ppm		250 ppm
500		500
1000		996

7/12/01
AJ

Na^+ std solutions:

25 ppm : $\frac{100\ ppm \times 25\ ml}{100\ ml}$

50 ppm : $\frac{100\ ppm \times 50\ ml}{100\ ml}$

75 ppm : $\frac{100\ ppm \times 75\ ml}{100\ ml}$

125 ppm : $\frac{1000\ ppm \times 12.5\ ml}{100\ ml}$

7/17/01
~~7/16/01~~ AJ 2/15/02 check K^+ electrode operation (slope) following AJ the procedure on page 22 of this lab book.

0.1M K^+ std used.

1st mV reading = -52.9 mV

2nd mV reading = +4.1

Difference between 2nd & 1st mV readings = 57.0 mV (54-60 mV O.K.)

Objective:- To remeasure the K^+ ion concentrations for 0.05 N NaCl/KCl ion exchange experiments due to possible systematic errors introduced by a problem with K standard solution (as supplied

Supplies & Equipment:- ① New K^+ standard solution ~~received~~ on AJ 2/15/02

② Orion 920A PH/ISE meter.

③ K^+ ISE (combination)

④ stir plate

7/17/01 AJ

K^+ Std. Solutions :-

390

0.01 M or ~~390~~ PPM : 0.1 M x 10 ml / 100 ml
AJ 2/15/02

0.02 M or 780 PPM : 0.1 M x 20 ml / 100 ml

0.04 M or 1560 PPM : 0.1 M x 40 ml / 100 ml

0.03 M or 1170 PPM : 0.1 x 30 ml / 100 ml

1955

0.05 M or ~~1950~~ PPM : 0.1 x 50 ml / 100 ml
AJ 2/15/02

3 pt. Calibration :

K^+ Std.	Measured Value	
390 ~~390~~ PPM (AJ 2/15/02)	388, 390	
780 PPM	780, 780	56 mV
1170 PPM	1180, 1170	59.1 mV
1170 PPM	1160 ~~1170~~ (AJ 2/15/02), 1150	
1560 PPM	1550, 1550	57.2 mV
1950 PPM	1949, 1949	55.2 mV

Mixture # KNa05*	XNa,i to use	Calc.K ppm,i	Meas. K ppm,i	Meas. K ppm,i
1	0.1	1759	1729	1750
2	0.1	1759		
3	0.1	1759		
4	0.2	1564		
5	0.2	1564	1560	1570
6	0.2	1564		
7	0.3	1368		
8	0.3	1368		
9	0.3	1368	1360	1370
10	0.4	1173		
11	0.4	1173	1200	1200
12	0.4	1173		
13	0.5	977		
14	0.5	977	997	997
15	0.5	977		
16	0.7	586	596	598
17	0.7	586		
18	1	0		
19	1	0		
20	1	0		

7/18/01 AJ

K^+ std solutions :-

0.005

~~0.005~~ M or 195.5 PPM : 0.1M x 5 ml / 100 ml
AJ 2/15/02

156.4 PPM

0.004 M or ~~156 PPM~~ : 0.1 M x 4 ml / 100 ml
AJ 2/15/02

0.003 M or 117.3 PPM : $\frac{0.1\,M \times 3\,ml}{100\,ml}$

0.002 M or 78.2 PPM : $\frac{0.1\,M \times 2\,ml}{100\,ml}$

0.001 M or 39.1 PPM : $\frac{0.1\,M \times 1\,ml}{100\,ml}$

Mixture # KNa05*	XNa,i to use	Calc.Na ppm,i	Calc.K ppm,i	Calc.K ppm,f	Meas. K ppm,f diluted	Meas. K ppm,f
1	0.1	115	1759	1935	186	1860
2	0.1	115	1759	1916	182	1820
3	0.1	115	1759	1896	179	1790
4	0.2	230	1564	1857	153	1530
5	0.2	230	1564	1808	153	1530
6	0.2	230	1564	1779	157	1570
7	0.3	345	1368	1759	134	1340
8	0.3	345	1368	1701	136	1360
9	0.3	345	1368	1662	136	1360
10	0.4	460	1173	1603	116	1160
11	0.4	460	1173	1564	116	1160
12	0.4	460	1173	1486	116	1160
13	0.5	575	977	1368	97.9	979
14	0.5	575	977	1271	97.1	971
15	0.5	575	977	1173	97.9	979
16	0.7	805	586	977	65.0	650
17	0.7	805	586	782	62.1	621
18	1	1150	0	586	18.5	185
19	1	1150	0	391	14.2	142
20	1	1150	0	195	8.72	87.2

these measurements were done on 7/20/01

PH of 1L $UO_2(NO_3)_2$ + 0.97 CDV (Prepared by RP) adjusted to 6.37 by adding 1M $NaHCO_3$ (≈ 75ul).

7/20/01

~~7/20/01~~ AJ 2/15/02 PH of 1L $UO_2(NO_3)_2$ + 0.97 CDV (Prepared by RP) measured as 6.64. No need to adjust.

7/24/01 PH of 1L $UO_2(NO_3)_2$ + 0.97 CDV (prepared by RP) measured as 6.72. No PH adjustment.

AJ 2/13/02

7/25/01 Preparation of 0.05N CsCl/KCl solutions for Ion-Exchange Experiments
AJ

objective:- To prepare several CsCl-KCl solutions with a total Normality of 0.05 and a fixed Cs/K ratio.

Equipment & Supplies: 6-~~6~~ AJ 2/19/02 - 500ml polypropylene Nalgene bottles
250 ml Volumetric flasks
Plastic dropper
Nanopure water (>17 mΩ resistivity)
Magnetic stirrers & stir plate
6 - 150 ml beakers
Mettle AE240 weighing balance
Weighing boats or paper
~~Aldrich~~ AJ 7/25/01 KCl (FS lot #)
CsCl CFS lot #)

Procedure

1. Prepare 250 ml of CsCl-KCl aqueous mixture with a total normality of 0.05 N and a fixed Cs/K ratio by taring reagent grade CsCl and KCl in the amounts given in Table 1 onto a clean (acid-washed) 100-ml or 150-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

(Note: Instead of 100-mL or 250 mL beaker, one can use a weighing boat for taring the reagent. This procedure can be modified as needed).

Decant the solution into a clean 250-ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water drowse up to the mark; remix.

Transfer the solution into a clean 250 ml polypropylene bottle. Label the bottle (e.g., CsCl/KCl*0.05N*0.1E_{Cs}, plus Date and Initial).

Table 1

ECs,i (0.05 N) (CsCl-KCl soln.)	Wt. CsCl needed for 250 ml	Wt. KCl needed for 250 ml	Wt. CsCl used (gm)	Wt. KCl used (gm)
0.05	0.1052	0.8853	0.1053	0.8854
0.1	0.2105	0.8387	0.2105	0.8387
0.2	0.4209	0.7455	0.4211	0.7455
0.4	0.8418	0.5591	0.8419	0.5592
0.7	1.47315	0.2796	01.4732	0.2797
1	2.1045	0	2.1045	0

Measured on 7/27/01

27 Jul 01 Sampling of 0.05N CsCl/NaCl experimental solutions for analyses of Cs-137 by LSA. Analysis of Nat using ISE was previously accomplished (see pg. 103).
PB

Spiked experimental solutions will be sampled by withdrawing ~0.5mL of each solution and transferring the solution to a pre-weighed and labeled liquid scintillation vial. The LS vials (7mL) are preloaded with 0.5 mL of 0.1 M HNO_3. ~5mL of Ultima Gold AB LS cocktail is then added and the vials are mixed.

Analysis is accomplished using the LSA in cpm mode with a protocol designed to count Cs-137 and Ba-137m photons.

27 Jul 01

PD	Mixture # $CsNa-05N$	Sample number	wt vial (+ 0.5mL HNO_3)	wt. vial + sample
CsNa spkd	1	05CsNa 1 a	7.5695	8.0682
		05CsNa 1 b	7.6403	8.1442
"	2	05 CsNa 2a	7.7164	8.2106
		05 CsNa 2b	7.6259	8.1228
"	3	05 CsNa 3a	7.6425	8.1390
		05 CsNa 3b	7.6063	8.1023
"	4	05 CsNa 4a	7.6150	8.1105
		05 CsNa 4b	7.6729	8.1685
"	5	05 CsNa 5a	7.6921	8.1862
		05 CsNa 5b	7.6499	8.1460
"	6	05 CsNa 6a	7.6542	8.1454
		05 CsNa 6b	7.6444	8.1403
"	7	05 CsNa 7a	7.6647	8.1584
		05 CsNa 7b	7.6623	8.1583
"	8	05 CsNa 8a	7.6517	8.1455
		05 CsNa 8b @ 7/27/01	7.6252	8.1207
"	9	05 CsNa 9a	7.6365	8.1328
		05 CsNa 9b	7.6213	8.1170
"	10	05 CsNa 10a	7.6303	8.1283
		05 CsNa 10b	7.6522	8.1512
"	11	05 CsNa 11a	7.6337	8.1304
		05 CsNa 11b	7.5946	8.0934
"	12	05 CsNa 12a	7.6763	8.1738
		05 CsNa 12b	7.6896	8.1885
"	13	05 CsNa 13a	7.7279	8.2239
		05 CsNa 13b	7.6506	8.1473
"	14	05 CsNa 14a	7.6557	8.1513
		05 CsNa 14b	7.7346	8.2328
"	15	05 CsNa 15a	7.6495	8.1458
		05 CsNa 15b	7.6480	8.1466
"	16	05 CsNa 16a	7.6434	8.1398
		05 CsNa 16b	7.6601	8.1572
"	17	05 CsNa 17a	7.6962	8.1939
		05 CsNa 17b	7.7162	8.2130
"	18	05 CsNa 18 a	7.6426	8.1426
		05 CsNa 18 b	7.6542	8.1536
"	19	05 CsNa 19 a	7.7856	8.2870
		05 CsNa 19 b	7.7871	8.2874

BW 2/13/02

7/31/01
AJ

Mixture # CsK-05*	ECs,i to use	Weight K-zeol. to use (gm)	Weight K-zeol. used (gm)	Volume soln. to use (ml)	Weight of soln. used (gm)
1	0.05	0.2271	0.2270	25	25.0309
2	0.05	0.1174	0.1175	25	25.0438
3	0.1	0.1660	0.1661	25	25.0647
4	0.1	0.1709	0.1709	50	50.0604
5	0.2	0.2357	0.2358	25	25.0674
6	0.2	0.1469	0.1468	25	25.0713
7	0.2	0.1371	0.1370	50	50.0696
8	0.4	0.1217	0.1220	10	10.0923
9	0.4	0.1285	0.1284	25	25.9303
10	0.4	0.1077	0.1076	50	50.0672
11	0.7	0.2328	0.2328	25	25.0872
12	0.7	0.1600	0.1601	25	25.0453
13	0.7	0.0957	0.0957	50	50.0843
14	1	0.2321	0.2321	25	25.1321
15	1	0.1776	0.1777	25	25.1457
16	1	0.1794	0.1796	50	50.2925
17	1	0.1096	0.1095	50	50.2798
18	1	0.1005	0.1006	AJ 98+2 7/31/01 ~~100~~	98.0599 +2.0210

8/7/01
AJ

K^+ std. solutions required :-

0.01 M or 391 PPm : $\frac{0.1\ M \times 10\ ml}{100\ ml}$

0.02 M or 782 PPm : $\frac{0.1\ M \times 20\ ml}{100\ ml}$

0.005 M or 195.5 PPm : $\frac{0.1\ M \times 5\ ml}{100\ ml}$

8/7/01 AJ

0.004 M or 156.4 ppm : $\frac{0.1M \times 4ml}{100ml}$

0.003 M or 117.3 ppm : $\frac{0.1M \times 3ml}{100ml}$

0.05 M or 1955 ppm : $\frac{0.1M \times 50ml}{100ml}$ 40

0.04 M or 1564 ppm : $\frac{0.1M \times 40ml}{100ml}$ AJ 2/15/02

0.03 M or 1173 ppm : $\frac{0.1M \times 30ml}{100ml}$

Mixture # CsK-05*	ECs,i to use	Calc. K ppm,initial	Meas. K ppm,initial 8/7/01, 9/4/01	Calc. K ppm,f	Meas. K ppm,f 9/4/01
1	0.05	1857	1940, 1920	1930	1940/1950
2	0.05	1857		1910	1930
3	0.1	1759	1879, 1850	1865	1890
4	0.1	1759		1825	1850
5	0.2	1564	1790, 1729	1775	1840/1850
6	0.2	1564		1714	1840
7	0.2	1564		1643	
8	0.4	1173	1500, 1370	1561	
9	0.4	1173		1370	
10	0.4	1173		1262	
11	0.7	586	1220, 1110	1032	
12	0.7	586		913	
13	0.7	586		794	
14	1	0		563	
15	1	0		453	
16	1	0		252	
17	1	0		161	
18	1	0		77	

0.015 M or 586.5 ppm: $\frac{0.1M \times 15ml}{100ml}$

K^+ electrode slope check: Following procedure from page 22. (0.1M K^+ std)

1st mV reading = −73.2 mV
2nd mV reading = +3.4 mV [> 56±2mV]

1st mV reading = −52.2 mV
2nd mV reading = +4.9 mV [Difference should be between 56±2mV]

Difference = 4.9 + 52.2 mV
= 57.1 mV (between 56±2m

3 pt. Calibration :- 3 times before measurements

1955 ~~1955~~ AJ 2/15/02 PPm — 1949, 1900
1564 PPm — 1560, 1570
1173 PPm — 1160, 1140

8/8/01 AJ

3 pt. Calibration :

1955 PPm — 1949, 1949 — 56.6 mV
1564 PPm — 1550, 1550
1173 PPm — 1170, 1160

BJS 2/14/02

8/10/01

AJ	Mixture # CsK - 0.05	Sample Number	wt. of vial + 0.5ml HNO_3	wt. of vial + sample
CsK Stkd	1	0.05CsK 1a	7.6103	8.1099
		1b	7.6396	8.1421
"	2	0.05CsK 2a	7.6333	8.1316
		2b	7.6870	8.1871
"	3	0.05CsK 3a	7.6135	8.1130
		3b	7.6206	8.1214
"	4	0.05CsK 4a	7.6179	8.1199
		4b	7.6521	8.1535
"	5	0.05CsK 5a	7.6109	8.1099
		5b	7.6731	8.1746
"	6	0.05CsK 6a	7.5988	8.0987
		6b	7.6357	8.1368
"	7	0.05CsK 7a	7.6528	8.1529
		7b	7.6010	8.1034
"	8	0.05CsK 8a	7.5738	8.0740
		8b	7.7019	8.2030
"	9	0.05CsK 9a	7.6312	8.1317
		9b	7.6082	8.1104
"	10	0.05CsK 10a	7.6482	8.1463
		10b	7.7243	8.2257
"	11	0.05CsK 11a	7.6212	8.1204
		11b	7.6127	8.1148
"	12	0.05CsK 12a	7.6581	8.1580
		12b	7.6518	8.1535
"	13	0.05CsK 13a	7.6450	8.1450
		13b	7.6809	8.1825
"	14	0.05CsK 14a	7.7734	8.2734
		14b	7.5985	8.0995
"	15	0.05CsK 15a	7.6940	8.1933
		15b	7.5738	8.0751
"	16	0.05CsK 16a	7.6619	8.1620
		16b	7.6640	8.1654
"	17	0.05CsK 17a	7.6293	8.1297
		17b	7.6640	8.1662
"	18	0.05CsK 18a	7.6764	8.1793
		18b	7.6459	8.1491

BW 2/14/02

8/14/01

AJ	Ecs,i Mixture # Initial	Sample Number	wt. of vial + 0.5ml of 0.1N HNO_3	wt. of vial + 0.5ml of Sample
	0.05	0.05 Ecs,i 1a	7.6236	8.1231
		0.05 Ecs,i 1b	7.6766	8.1775
	0.1	0.1 Ecs,i 1a	7.6567	8.1541
		0.1 Ecs,i 1b	7.5841	8.0817
	0.2	0.2 Ecs,i 1a	7.7362	8.2360
		0.2 Ecs,i 1b	7.6712	8.1720
	0.4	0.4 Ecs,i 1a	7.6726	8.1690
		0.4 Ecs,i 1b	7.6471	8.1448
	0.7	0.7 Ecs,i 1a	7.7779	8.2747
		0.7 Ecs,i 1b	7.6512	8.1482
	1.0	1.0 Ecs,i 1a	7.7016	8.2014
		1.0 Ecs,i 1b	7.6681	8.1679

9/4/01 K^+ stds prepared using self prepared 0.1 M KCl as follows:

1.8640 gm of KCl (lot # 006242) was dissolved in deionized H_2O and diluted with more water to 250 ml mark. Store in plastic bottle.

0.05 ~~ppm~~ M (AJ 9/4/01) or 1955 ppm : $\frac{0.1\,M \times 50\,ml}{100\,ml}$

0.04 M or 1564 PPm : $\frac{0.1\,M \times 40\,ml}{100\,ml}$

0.03 M or 1173 PPm : $\frac{0.1\,M \times 30\,ml}{100\,ml}$

0.005 M or 195.5 ppm : $\frac{0.1\,M \times 5\,ml}{100\,ml}$

0.004 M or 156.4 ppm : $\frac{0.1\,M \times 4\,ml}{100\,ml}$

0.003 M or 117.3 ppm : $\frac{0.1\,M \times 3\,ml}{100\,ml}$

3 pt. calibration 2 times:

K* std	Measured Value
1173 ppm	1160
1564	1560
1955	1960

mixture (GK-0.05N)

Ecsi to use	Measured K ppm initial
0.05	1920
0.1	1850
0.2	1729
0.4	1370
0.7	1110

0.1M NaCl for K^+ ~~reference~~ AJ 9/14/01 electrode as reference fill solution:

~~Bob~~ 2/14/02

9/5/01

AJ Purpose:- To practice sample preparation of core cylinders (≤3 x 5 cm) using 1¼" core drill bit and Black & Decker Core drill 748 for hydraulic conductivity measurements.

Equipment & supplies:

① Core drill 748 (Black & Decker. Ser. 1903)
② 1¼" core drill bit
③ any rock sample
④ garden hose & water supply
⑤ Ear plugs
⑥ Safety glasses.

Procedure:

① Secure the rock sample in the desired orientation using the vice on drill.

② Start the continuous smooth flow of water on the rock sample.

③ Lower the drill bit at the desired location on the rock sample.

④ Wear safety glasses, ear plugs and start the drill bit.

⑤ Move the drill very slowly & smoothly into the sample until finished.

9/7/01 AJ 9/5/01 ⑥ Move the drill bit ~~rapidly~~ AJ 9/5/01 away from the rock sample rapidly and turn it off.

⑦ Remove the core cylinder, dry it and store.

9/7/01
AJ Objective :- To obtain 3cm x 5cm core cylinders from BB1 rock sample using core drill bit ~~bits~~ AJ 9/7/01 (diameter - 1½") and core drill 748 (Black & Decker) for hydraulic conductivity measurements.

Equipment and supplies :-

① Core Drill (Black & Decker 748, ser. 1903)
② 1½" core drill bit (Blazer diamond Products, Inc.) AJ 2/15/02
③ BB1 rock sample
④ Garden hose & water supply
⑤ Ear plugs & safety glasses.

Method : Same as on page ~~118~~ 119 AJ 2/15/02 - 120.

Note : BB1 rock sample was unable to fit under core drill. To cut the rock sample BB1 into 2 parts, it was soaked in tap water for 3 days.

9/11/01
9/~~11~~/01 AJ 9/11/01 AJ BB1 rock sample was removed from tap water and placed in the vice of core drill. Hand ~~tooth~~ saw ~~tooth~~ AJ 9/11/01 was used to divide the sample into 2 parts. Both parts were marked with top surface and one part was used to ~~drill~~ drill AJ 2/15/02 three 3cm x 5cm core cylinders. The core cylinders were wrapped using plastic saran wrap along the surface only and exposing both ends. The purpose of exposed ends is to get rid of ~~excess~~ excess AJ 2/15/02 moisture which could cause bacterial growth. The cylinders were further secured with duct tape to ~~support~~ AJ 9/11/01 ~~prevent~~ AJ 2/15/02 prevent it from crumbling.

9/12/01
AJ Purpose :- To test ~~epoxy~~ AJ 9/12/01 the strength of epoxy #2300 (Asemco Products, Inc.) for sealing the surface of fresh drilled (3cm x 5cm) cores of BB1 rock sample.

Equipment & Supplies :

① BB1 rock core sample (3cm x 5cm or < 3x5)
② disposable plastic container to mix epoxy
③ Epoxy #2300 from Asemco Products Inc.
④ Spatula (plastic)

Procedure :- Mix resin and hardner in ratio 10:1 (by weight) in small plastic ~~container~~ container AJ 2/15/02 or weigh boat. Use plastic spatula to mix it well and place the sample at desired location. Let it cure at room temperature overnight.

9/14/01
AJ The freshly drilled samples of rock (BB1, vitric tuff) sealed strongly with epoxy #2300. The excess sample was cut using saw (marathon Electric Model # WVF48S17D2093H) and was flushed with epoxy.

Preparation of BB1 sample rock for x-ray diffraction:
Several small pieces of BB1 rock sample were crushed together in mortar and pestle (agate). The grinded powder sample was passed through 325 mesh sieve using ROTap shaker to obtain sample for x-ray diffraction analysis.

AJ 9/18 9/19/01 Thin layer of epoxy #2300 on fresh cored cylinders of BB1 rock:
Two (3cm x 5cm ~~3cm x 5cm~~ AJ 2/15/02) core cylinders were ~~drilled~~ drilled AJ 2/15/02 from rock sample BB1 ~~using~~ using AJ 9/15/02. These samples were slightly dried by soaking excess water on paper towel. A thin layer of epoxy #2300 (Aremco Inc.) was applied around the core cylinders with a plastic spatula.

The samples were stored exposed to air overnight to be cured at room temperature.

9/18/01 AJ ① Thin layer of epoxy #2300 didn't dry completely at ~~Room~~ Room 2/15/02 temperature. The possible cause may be the excess moisture absorbed inside the sample ~~from~~ during 9/18/01 drilling cores. The samples are kept in drying oven at 50°C overnight.
② Two dried samples were coated with epoxy #2300
9/19/01 AJ ① The samples cured at 50°C overnight seemed to have dry epoxy (#2300) layer around core sample
② Thin layer of epoxy #2300 on air dried rock samples did not appear to cure in air overnight. AJ 9/19/01
9/21/01 Copy made for QA archives
BAW

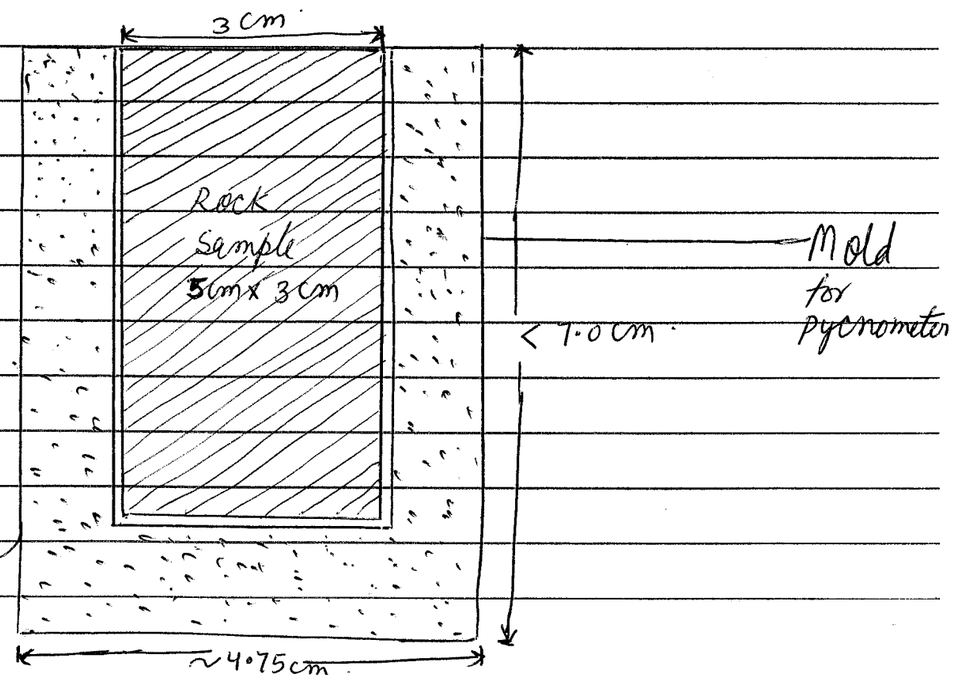
BW 2/14/02

9/21/01 AJ — Preparation of mold for air pycnometer porosity (air) measurements

Dimensions of large Volume Cell = 7cm x 4.9 cm.
Length x Diameter

Dimensions of rock BB1 sample = 5cm x 3cm.
Length x Diameter

The purpose of the mold is to minimize the air space between the volume cell and the sample. The material used for the pore has to be non-porous e.g. teflon.



The ~~request~~ request for mold is given to Don Benson for Div. 10. AJ 2/15/02

Note: Deairator hooked up to hydraulic conductivity measurement apparatus is non-functional. Alternate procedure will be investigated to prepare samples for measurements.

wt. of BB1 before putting it in oven @ 90°C
= 50.0834 gm & ~~B~~ AJ 2/15/02 wt. of beaker = 81.1972 g

BW 2/14/02

9/25/01 (Dilution - $\frac{1}{10}$)

AJ

Mixture # Csk 0.05*	Measured K ppm, final
1	194, ~~197~~ 197 (AJ 9/15/02)
2	194, 196
3	195, 196
4	191, 190
5	188, 189
6	186, 187
7	180, 182
8	173, 176
9	~~159~~ 159 (AJ 9/15/02), 153
10	150, ~~145~~ 145 (AJ 9/15/02)
11	145, 141
12	135, 131
13	121, 117
14	113, 109
15	107, ~~103~~ AJ 9/15/02 103
16	99.5, 95.3
17	97.3, 93.4
18	~~94.4~~ 94.4 (AJ 9/15/02), 90.0,

K^+ stds 1- 0.002 M or 78.2 PPM : $\frac{0.1M \times 2ml}{100ml}$

0.003 M or 117.3 ppm : $\frac{0.1M \times 3ml}{100ml}$

0.004 M or 156.4 PPM : $\frac{0.1 \times 4ml}{100ml}$

9/26/01 AJ

0.005 M or 195.5 PPM : $\frac{0.1M \times 05\ ml}{100\ ml}$ (AJ 9/15/02)

Mixture # Csk 0.05*	Measured K PPm Final (w/ dilution $\frac{1}{10}$)
1	198, 197
2	195, 196
3	193, 195
4	191, 193
5	187, 190
6	185, 187
7	179, 182
8	168, ~~EG~~ AJ 9/26/01 173
9	150, 153
10	144, 146
11	138, 139
12	130, 132
13	117, 120
14	107, 112
15	100, 105
16	~~90~~ AJ 9/26/01 91.4, 94.1
17	89.7, 87.4
18	85.8, 82.5

Check electrode operation ~~following~~ following (AJ 2/15/02) procedure on page 22.

I st mV reading = -59.6 mV

2nd mV reading = -2.5 mV

slope = -2.5 - (-59.6) = 57.1 mV (between 54-60 mV) O.k.

9/28/01 AJ — Preparation of KCl - NaCl (0.05 N) solutions from revised procedure for ion-exchange expts.

Objective :- To prepare several KCl - NaCl solutions with a normality 0.05 and a fixed K/Na ratio.

Equipment & Supplies :-

7 - 500 ml polypropylene Nalgene bottles
250 ml volumetric flasks
Plastic droppers
Magnetic stirrers and stir plate
Mettler AE240 weighing balance
Nanopure water (>17 MΩ)

KCl (FS lot # 006242)
NaCl (FS lot # 986412)

Procedure

1. Prepare 1000 ml of KCl-NaCl aqueous mixture with a total normality of 0.05 N and a fixed K/Na ratio by taring reagent grade KCl and NaCl in the amounts given in Table 1 onto a clean (acid-washed) 250-ml or 500-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

Decant the solution into a clean 1000 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 1000 ml polypropylene bottle. Label the bottle (e.g., KCl/NaCl*0.05N*0.1X_{Na}, plus Date and Initial).

$E_{Na,i}$ (0.05 N) KCl-NaCl Solns.	wt. NaCl needed for 250 ml	wt. of KCl used needed for 250 ml	wt. of NaCl added	wt. of KCl added
0.22	0.1607	0.7269	0.1608	~~0.727~~ AJ 2/15/02 0.727
0.45 ~~0.45~~ AJ 2/15/02	0.3286	0.5126	0.3287	0.5127
0.56	0.4091	0.4100	0.4093	0.4102
0.68	0.4968	0.2982	0.4968	0.2982
0.8	0.5844	0.1864	0.5845	0.1863
0.92	0.6721	0.0746	0.6723	0.746
1.0	0.7306	0	0.7308	0

10/2/01
AJ

AJ ~~Mixture~~ # 10/2/01 CsK - 0.05N $E_{Cs,i}$ to use	Measured K ppm Initial solution
0.05	1890, 1850, 1850
0.1	1840, 1830, 1800
0.2	1700, 1680, 1659
0.4	1390, 1380, 1340
0.7	1100, 1110, 1080

Calibration at 1173 ppm, 1563 ppm & 1955 ppm AJ 10/4/01 K^+ stds. prepared as per page 114.

EXPERIMENT KCl/NaCl-0.05N
(SET #2)

Mixture # KNa05 #2*	$X_{Na,i}$ to use	Weight zeol. to use (gm)	Weight zeol. used (gm	Volume soln. to use (ml)	Calc. Na ppm,i	Meas. Na ppm,i	Calc. K ppm,i	Meas. K ppm,i
1	0.22	1.4706		10	253		1525	1530
2	0.45	1.5319		10	517		1075	1070
3	0.56	1.0621		10	644		860	859
4	0.68	0.8578		10	782		626	629
5	0.8	0.7003		10	920		391	394
6	0.92	0.6033		10	1058		156	161
7	1.0	0.4202		10	1150		0	
8	1.0	0.2941		10	1150		0	
9	1.0	0.2131		10	1150		0	
10	1.0	0.1362		10	1150		0	
11	1.0	0.1776		25	1150		0	
12	1.0	0.1149		25	1150		0	
13	1.0	0.1251		50	1150		0	
14	1.0	0.1225		100	1150		0	
15	1.0	0.1751		250	1150		0	
16	1.0	0.0766		250	1150		0	

BW 2/14/02

10/3/01

Preparation 3M $CaCl_2$ for converting CDV to Calcium form to be used in ion exchange experiments.

$CaCl_2 \cdot 2H_2O$ (FS Lot # 995698)

441.06 g of $CaCl_2 \cdot 2H_2O$ was dissolved in water and transferred to 1000 ml Volumetric flask. Dilute it up-to mark and store in plastic bottle.

Two batches of ~ 20.0 g each of CDV * 100/200 * HL * RC * RFe were reacted with 3M $CaCl_2$ solution in two 250 ml plastic bottles. 200 ml of 3M ~~CaCl~~ $CaCl_2$ solution was used for each batch. Set up the water bath at 70°C and 60 rpm. Place both the bottles in water bath. BW 10/3/01

BW 2/14/02

40CT01

BAW

AA analysis for potassium of Cs/K binary solutions with zeolite

The potassium concentration of the cesium/potassium binary solutions with zeolite was to be determined using AA. Dilutions would also be required in order to bring the potassium concentration into the calibration curve range (0.1 to 2 ppm K). The solutions analyzed were 18 sample solutions (420/114) and 5 reference solutions (420/110). Only five of the six reference solutions (420/110) were analyzed for potassium. One of the six solutions (CsCl/KCl 1N) did not have KCl added to it. Therefore, this solution was not analyzed for potassium.

Matrix effects were a concern since the concentration of both cations varied significantly. The potassium and cesium target concentrations were known for each solution. I used the potassium concentration to determine what dilution factor would be required to result in a potassium concentration that would fall within the cal curve. I also wanted to know what the cesium concentration of the diluted samples would be in order to determine if potassium cal curves with different cesium concentrations would be required. Table 1 contains the target concentrations in the undiluted samples. Table 2 contains the concentrations of the diluted samples (DF = dilution factor) based on Table 1.

Table 1

Sample ID	Target Conc K (ppm)	Target Conc Cs (ppm)
1	1930	38.6
2	1910	38.2
3	1865	37.3
4	1825	36.5
5	1775	35.5
6	1714	34.28
7	1643	32.86
8	1561	31.22
9	1370	27.4
10	1262	25.24
11	1032	20.64
12	913	18.26
13	794	15.88
14	563	11.26
15	453	9.06
16	252	5.04
17	161	3.22
18	77	1.54
CsCl-KCl 0.05N	1857	37.14
CsCl-KCl 0.1N	1759	35.18
CsCl-KCl 0.2N	1564	31.28
CsCl-KCl 0.4N	1173	23.46
CsCl-KCl 0.7N	586	11.72

BAW 10/4/01

40CT01

CONT BAW

Table 2

Sample ID	DF 1500 Target Conc K (ppm)	DF 1500 Target Conc Cs (ppm)	DF 250 Target Conc K (ppm)	DF 250 Target Conc Cs (ppm)	DF 100 Target Conc K (ppm)	DF 100 Target Conc Cs (ppm)
1	1.286667	0.057333	7.72	0.344	19.3	0.86
2	1.273333	0.102667	7.64	0.616	19.1	1.54
3	1.243333	0.202933	7.46	1.2176	18.65	3.044
4	1.216667	0.294733	7.3	1.7684	18.25	4.421
5	1.183333	0.4088	7.1	2.4528	17.75	6.132
6	1.142667	0.5462	6.856	3.2772	17.14	8.193
7	1.095333	0.707533	6.572	4.2452	16.43	10.613
8	1.040667	0.8924	6.244	5.3544	15.61	13.386
9	0.913333	1.326067	5.48	7.9564	13.7	19.891
10	0.841333	1.5696	5.048	9.4176	12.62	23.544
11	0.688	2.091333	4.128	12.548	10.32	31.37
12	0.608667	2.360933	3.652	14.1656	9.13	35.414
13	0.529333	2.630667	3.176	15.784	7.94	39.46
14	0.375333	3.1548	2.252	18.9288	5.63	47.322
15	0.302	3.402933	1.812	20.4176	4.53	51.044
16	0.168	3.8596	1.008	23.1576	2.52	57.894
17	0.107333	4.065533	0.644	24.3932	1.61	60.983
18	0.051333	4.255733	0.308	25.5344	0.77	63.836
CsCl-KCl 0.05N	1.238	0.221333	7.428	1.328	18.57	3.32
CsCl-KCl 0.1N	1.172667	0.443333	7.036	2.66	17.59	6.65
CsCl-KCl 0.2N	1.042667	0.886	6.256	5.316	15.64	13.29
CsCl-KCl 0.4N	0.782	1.772	4.692	10.632	11.73	26.58
CsCl-KCl 0.7N	0.390667	3.101333	2.344	18.608	5.86	46.52

An initial AA sample prep scheme was based on the information in Table 2. Three different DFs would be used: 1500, 250, and 100. A few samples would be prepared at two different DFs. Three potassium cal curves would be prepared with various cesium matrix concentrations: 20ppm, 2ppm, and 0.2ppm.

Table 3 contains the samples initially prepared for DF 100 analysis. Table 4 contains the samples initially prepared for DF 250 analysis. All DF 100 and DF250 samples will be analyzed with a 20 ppm Cs potassium cal curve. Table 5 contains the samples initially prepared for DF 1500 analysis. Some DF 1500 samples will be analyzed with a 2 ppm Cs potassium curve and some DF 1500 samples will be analyzed with a 0.2 ppm Cs curve. Columns within Table 5 provide information indicating which cesium matrix curve would be used.

BAW 10/4/01

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CONT BAW

Table 3

Sample ID	DF 100 Target Conc K (ppm)	DF 100 Target Conc Cs (ppm)
17	1.61	60.983
18	0.77	63.836

Table 4

Sample ID	DF 250 Target Conc K (ppm)	DF 250 Target Conc Cs (ppm)
13	3.176	15.784
14	2.252	18.9288
15	1.812	20.4176
16	1.008	23.1576
17	0.644	24.3932
18	0.308	25.5344
CsCl-KCl 0.7N	2.344	18.608

Table 5

Sample ID	DF 1500 Target Conc K (ppm)	DF 1500 Target Conc Cs (ppm)	K cal curve with 0.2 ppm Cs	K cal curve with 2 ppm Cs
1	1.286667	0.057333	YES	
2	1.273333	0.102667	YES	
3	1.243333	0.202933	YES	
4	1.216667	0.294733	YES	
5	1.183333	0.4088	YES	
6	1.142667	0.5462	YES	
7	1.095333	0.707533	YES	
8	1.040667	0.8924	YES	
9	0.913333	1.326067		YES
10	0.841333	1.5696		YES
11	0.688	2.091333		YES
12	0.608667	2.360933		YES
13	0.529333	2.630667		YES
14	0.375333	3.1548		YES
CsCl-KCl 0.05N	1.238	0.221333	YES	
CsCl-KCl 0.1N	1.172667	0.443333	YES	
CsCl-KCl 0.2N	1.042667	0.886	YES	
CsCl-KCl 0.4N	0.782	1.772		YES
CsCl-KCl 0.7N	0.390667	3.101333		YES

4OCT01 BAW

5OCT01 Preparation of standards and samples for Aft analysis
BAW for potassium of K/Cs binary solns w/ zeolite

K Stock - Target 10 ppm K

reagents: Spex Certiprep 1000 ppm K in 2% HNO_3 cat # PLK2-2X, lot # 7-95K, rec 1/13/01, exp 1/31/02.

nanopure water

Added 1 mL (vol pipet) of 1000 ppm K to a 100 mL vol flask and diluted to mark with nanopure water.

Cs Hi - Target 100 ppm Cs made from solid CsCl into 100 mL

$$\frac{0.0127\text{ g CsCl}\left(\frac{132.9\text{ g Cs}}{168.36\text{ g CsCl}}\right)\left(\frac{10^6\text{ mg}}{\text{g}}\right)}{100\text{ mL}} \doteq 100.25\text{ mg/mL or ppm}$$

The appropriate mass of CsCl (Fisher BP210-500, lot # 010406, rec 5/14/01) was added to a ~~tare~~ BAW 10/5/01 pre weighed dish. The CsCl was transfered to a 100 mL vol flask. The dish was rinsed three times with nanopure water into the 100 mL vol flask. The 100 mL vol flask was diluted to mark with nanopure water.

Mass (g) of weighing dish	Mass (g) of dish + CsCl	Mass (g) of CsCl
0.5241	0.5369	~~0.0129~~ BAW 2/15/02 0.0128

Cs Low - Target 10 ppm Cs

Added 10 mL (vol pipet) of 100 ppm Cs (Cs Hi 420/135) to a 100 mL vol flask and diluted to mark with nanopure water

10/5/01 Preparation of CsCl/KCl - 0.05N solution
AJ with mole fraction $E_{Cs,i}$ = 1.0 per page 111

CsCl/KCl - 0.05N $E_{Cs,i}$	wt. of CsCl Needed (g)	wt. of KCl Needed	wt. of CsCl used (g)
1.0	2.1045	0	2.1044

0.25ml Spike #14A Cs-137 (1.48 uCi/g) on page 369/028 was added to 250 ml solution.

Restore ~~no old spiked solution~~ was used

Mixture # CsK - 0.05N (AJ 1/15/02)	$E_{Cs,i}$ to use	wt. of Kf zeolite to use	wt. of Kf zeolite used	Vol. of solution to use
# 14	1.0	0.2321	0.2319	25 ml
# 15	1.0	0.1776	0.1776	25
# 16	1.0	0.1794	0.1796	50
# 17	1.0	0.1096	0.1095	50
# 18	1.0	0.1005	0.1005	100

~~Actual~~ (10/5/01) (AJ 1/15/02) of vials	wt. of vials	(wt. of vials + sample) (AJ 10/5/01)
CsCl/KCl - 0.05 ~~ia~~ ia	7.3797	7.~~88~~ 8788
CsCl/KCl - 0.05 ib	7.3310	7.7714

Bw 2/14/02

EXPERIMENT KCl/NaCl-0.05N
(SET #2)

10/5/01

Mixture # KNa05 #2*	$X_{Na,i}$ to use	Weight zeol. to use (gm)	Weight zeol. used (gm	Volume soln. to use (ml)	Calc. Na ppm,i	~~Meas. Na ppm,i~~ (AJ 10/5/01) Vol. of soln used	Calc. K ppm,i	Meas. K ppm,i
1	0.22	1.4706	1.4706	10	253	10	1525	
2	0.45	1.5319	1.5318	10	517	10	1075	
3	0.56	1.0621	1.0619	10	644	10	860	
4	0.68	0.8578	~~0.8580~~ 0.8580 (AJ 1/15/02)	10	782	10	626	
5	0.8	0.7003	0.7004	10	920	10	391	
6	0.92	0.6033	0.603	10	1058	10	156	
7	1.0	0.4202	0.4203	10	1150	10	0	
8	1.0	0.2941	0.2940	10	1150	10	0	
9	1.0	0.2131	0.2130	10	1150	10	0	
10	1.0	0.1362	0.1361	10	1150	10	0	
11	1.0	0.1776	0.1777	25	1150	25	0	
12	1.0	0.1149	0.1148	25	1150	25	0	
13	1.0	0.1251	0.1252	50	1150	50	0	
14	1.0	0.1225	0.1227	100	1150	100	0	
15	1.0	0.1751	0.1753	250	1150	250	0	
16	1.0	0.0766	0.0765	250	1150	250	0	

CDV * 100/200 * HL * CP * Kf (Prepared by AJ 1/25/01)

5OCT01

CONT BAW

Potassium calibration curves

All final volumes were 50 mL (volumetric flasks)
Volumetric pipets were used to transfer all solutions in the 0.5 mL to 25 mL volume range
% LaCl is w/w
10 ppm K was K stock (420/135)
1% LaCl was Soln A (463/40)
100 ppm Cs was CsHi (420/135)
10 ppm Cs was CsLow (420/135)

Potassium calibration curve with 20 ppm Cs

Soln ID	Target Conc of k (ppm)	Target Conc of LaCl (%)	Target Conc of Cs (ppm)	Vol (mL) of 10 ppm K	Vol (ml) of (1% LaCl)	Vol (ml) of 100 ppm Cs
K1*A	2	0.1	20	10	5	10
K2 A	1	0.1	20	5	5	10
K3 A	0.6	0.1	20	3	5	10
K4 A	0.4	0.1	20	2	5	10
K5 A	0.2	0.1	20	1	5	10
K6 A	0.1	0.1	20	0.5	5	10

* AA sensitivity check

Potassium calibration curve with 2 ppm Cs

Soln ID	Target Conc of k (ppm)	Target Conc of LaCl (%)	Target Conc of Cs (ppm)	Vol (mL) of 10 ppm K	Vol (ml) of (1% LaCl)	Vol (ml) of 10 ppm Cs
K1* B	2	0.1	2	10	5	10
K2 B	1	0.1	2	5	5	10
K3 B	0.6	0.1	2	3	5	10
K4 B	0.4	0.1	2	2	5	10
K5 B	0.2	0.1	2	1	5	10
K6 B	0.1	0.1	2	0.5	5	10

Potassium calibration curve with 0.2 ppm Cs

Soln ID	Target Conc of k (ppm)	Target Conc of LaCl (%)	Target Conc of Cs (ppm)	Vol (mL) of 10 ppm K	Vol (ml) of (1% LaCl)	Vol (ml) of 10 ppm Cs
K1* C	2	0.1	0.2	10	5	1
K2 C	1	0.1	0.2	5	5	1
K3 C	0.6	0.1	0.2	3	5	1
K4 C	0.4	0.1	0.2	2	5	1
K5 C	0.2	0.1	0.2	1	5	1
K6 C	0.1	0.1	0.2	0.5	5	1

05OCT01

CONT BAW Preparation of DF100 samples

DF100 will be a two fold dilution: 1to10 and 1to10.
See 420/134 for DF100 Sample list (Table 3)
The initial (first dilution) will be performed as follows:
Added 1mL (eppendorf pipette) of sample to a 10mL vol flask and diluted to mark with nanopure water, labeled X i 100 where X= sample #, i= intermediate, 100= ultimate DF
Final (second) dilution step will be performed later.

Preparation of DF250 samples

DF250 will be a two fold dilution: 1to25 and 1to10.
See 420/134 for DF250 sample list (Table 4)
The initial (first dilution) will be performed as follows:
Added 1mL ~~(eppendorf~~ pipette) of sample to a 25mL vol falsk and diluted to mark with nanopure water.
Labeled X i 250 where X= sample #, i= intermediate, 250= ultimate DF.
Final (second) dilution step will be performed later

Preparation of DF1500 samples

DF1500 will be a two fold dilution: 0.333to50 and 1to10
See 420/134 for DF1500 sample list (Table 5)
The initial (first dilution) will be performed as follows:
Added 0.333mL (eppendorf pipette) of sample to a 50mL

05OCT01
CONT BAW vol flask and diluted to mark with nanopure water: Labeled Xi1500 where X = sample #, i = intermediate, 1500 = ultimate DF
Final (second) dilution will be performed later

8OCT01 BAW

Preparation of samples for AA analysis (Potassium)

The final dilution step for all dilutions (DF100, DF250, + DF1500) was the same - a 1 to 10 dilution.

Added 1mL (vol pipette) of 1% La (w/w) (Soln A 463/40) and 1mL (vol pipette) of sample to a 10mL vol flask and ~~dilute~~ BAW 10/3/01 diluted to mark with nanopure water.

Preparation of 0.2 N HNO_3 solution

To be used for cleaning AA.
Added 20mL (vol pipet) of 1.0N HNO_3 (309/254) to a 100mL vol flask and diluted to mark with nanopure water

Potassium AA analysis

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
K - hollow cathode, lamp 8mA Fisher 14-386-108H
λ = 766.5, slit = 0.7nm, high, Air-acetylene flame

08OCT01 Blank = 0.1% La (w/w) Soln B 463/41
CONT BAW Integration time for samples = 3 sec

Curve "C" - 0.2ppm K Cs 8/14/99 BAW

ID	Conc (ppm)	Trial 1	2	3	4	5
K1A BAW	2	0.214	0.217	0.215	0.215	0.217
K2A 10/3/01	1	0.106	0.105	0.103	0.105	0.105
K3A	0.6	0.064	0.063	0.063	0.061	0.062
K4A	0.4	0.042	0.043	0.042	0.041	0.042
K5C	0.2	0.020	0.020	0.019	0.020	0.020
K6C	0.1	0.009	0.009	0.009	0.011	0.009

DF1500 samples on Curve C (0.2ppm K)

1	0.128	0.129	0.130	0.130	0.128
2	0.132	0.131	0.132	0.132	0.131
3	0.129	0.129	0.128	0.130	0.130
4	0.123	0.124	0.124	0.123	0.125
5	0.125	0.124	0.124	0.126	0.124
6	0.118	0.116	0.117	0.117	0.117
7	0.112	0.111	0.112	0.112	0.111
8	0.106	0.105	0.105	0.107	0.105
RO.05	0.117	0.119	0.119	0.119	0.118
RO.1	0.119	0.118	0.119	0.119	0.117
RO2	0.105	0.105	0.104	0.105	0.105

080CT01

CONT BAW — Curve B – 2ppm Cs

ID	Conc	Trial				
	ppm	1	2	3	4	5
K1B	2	0.213	0.214	0.211	0.215	0.215
K2B	1	0.107	0.108	0.109	0.109	0.108
K3B	0.6	0.065	0.065	0.064	0.064	0.065
K4B	0.4	0.043	0.042	0.043	0.042	0.043
K5B	0.2	0.020	0.020	0.020	0.020	0.020
K6B	0.1	0.009	0.009	0.009	0.009	0.009

DF1500 Samples on Curve B – 2ppm Cs

9	–	0.090	0.089	0.090	0.090	0.090
10	–	0.083	0.082	0.082	0.082	0.082
11	–	0.073	0.072	0.073	0.073	0.073
12	–	0.064	0.063	0.063	0.064	0.063
13	–	0.048	0.047	0.048	0.047	~~0.048~~ BW 2/15/02 0.048
14	–	0.035	0.035	0.035	0.035	0.036
R0.4	–	0.076	0.075	0.075	0.076	0.076
R0.7	–	0.039	0.038	0.039	0.039	0.039
K3B	0.6	0.065	0.066	0.066	0.066	0.066

BW 080101

080CT01

CONT BAW — Curve A – 20ppm Cs

ID	Conc	Trial				
	ppm	1	2	3	4	5
K1A	2	0.237	0.236	0.237	0.238	0.236
K2B	1	0.121	0.120	0.120	0.120	0.121
K3A	0.6	0.073	0.073	0.073	0.072	0.072
K4A	0.4	0.048	0.048	0.048	0.048	0.048
K5A	0.2	0.024	0.023	0.024	0.023	0.023
K6A	0.1	0.011	0.011	0.010	0.011	0.011

DF900, 250 Samples on Curve A – 20ppm Cs

17DF100	–	0.184	0.184	0.183	0.184	0.183
18DF100	–	0.095	0.095	0.095	0.095	0.095
13DF250	–	0.304	—— skipped ——			
11DF250	–	0.236	0.237	0.237	0.237	0.237
15DF250	–	0.191	0.192	0.192	0.190	0.190
16DF250	–	0.101	0.102	0.100	0.102	0.101
17DF250	–	0.066	0.065	0.065	0.065	0.066
18DF250	–	0.032	0.032	0.032	0.032	0.033
R0.7DF250	–	0.263	—— skipped ——			

Max correction for blank > 0.001

Aspirated with 0.2N HNO_3 for 10 min after analysis completed (420/140)

10/9/01 CsK-0.05N (Rerun)
AJ

Mixture #	Ecs,i to use	wt. of kf zeolite used	Vol. of soln. used
14	1.0	0.2320	25
15	1.0	0.1778	25
16	1.0	0.1795	50
17	1.0	0.1097	50
18	1.0	0.1005	AJ 10/9/01 97.5

3M $CaCl_2$:- Dissolve 220.53 g of $CaCl_2 \cdot 2H_2O$ (FS lot # 947250) in nanopure water and dilute it with more nanopure water to fill upto mark of 500 ml volumetric flask.

Replace 3M $CaCl_2$ solution for both bottles containing CDV to be converted to Ca-form.

10OCT01 BAW
The following is the results of the AA analysis for potassium of the cesium-potassium solutions. The data was collected 8OCT01 (420/147-143).

Cal Curves were generated. Regression lines were used to determine the concentration of the dilute solutions. These numbers were multiplied by the dilution factor to determine the concentration of the original solutions.

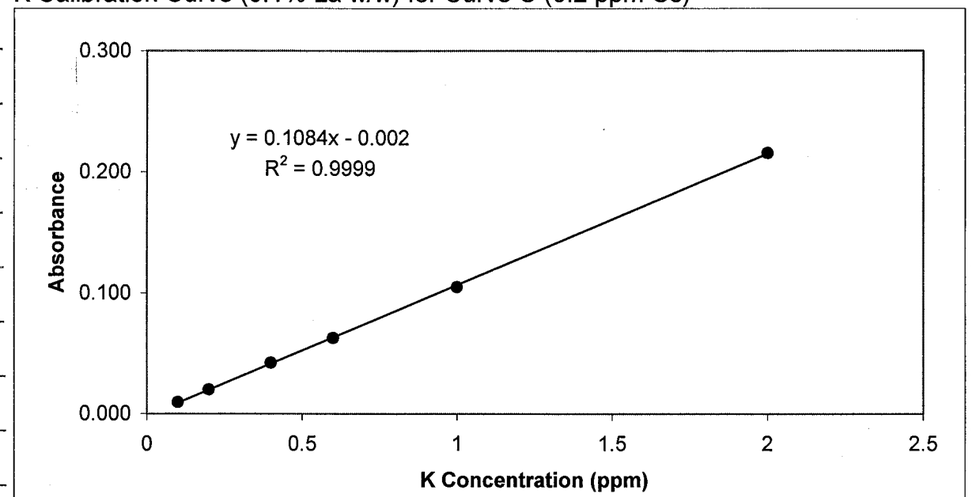
10OCT01 CONT BAW

Potassium cal curve C (0.2ppm Cs)

K Standard Data (0.1% La w/w) for Curve C (0.2 ppm Cs)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
K1C	2	0.214	0.217	0.215	0.215	0.217	0.2156
K2C	1	0.106	0.105	0.103	0.105	0.105	0.1048
K3C	0.6	0.064	0.063	0.063	0.061	0.062	0.0626
K4C	0.4	0.042	0.043	0.042	0.041	0.042	0.0420
K5C	0.2	0.020	0.020	0.019	0.020	0.020	0.0198
K6C	0.1	0.009	0.009	0.009	0.011	0.009	0.0094

K Calibration Curve (0.1% La w/w) for Curve C (0.2 ppm Cs)



K Data for samples analyzed on cal curve C (0.2 ppm Cs)

Solution ID	Dilution Factor	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
1	1500	0.128	0.129	0.130	0.130	0.128	0.1290
2	1500	0.132	0.131	0.132	0.132	0.131	0.1316
3	1500	0.129	0.129	0.128	0.13	0.13	0.1292
4	1500	0.123	0.124	0.124	0.123	0.125	0.1238
5	1500	0.125	0.124	0.124	0.126	0.124	0.1246
6	1500	0.118	0.116	0.117	0.117	0.117	0.1170
7	1500	0.112	0.111	0.112	0.112	0.111	0.1116
8	1500	0.106	0.105	0.105	0.107	0.105	0.1056
R0.05	1500	0.117	0.119	0.119	0.119	0.118	0.1184
R0.1	1500	0.119	0.118	0.119	0.119	0.117	0.1184
R0.2	1500	0.105	0.105	0.104	0.105	0.105	0.1048

10OCT01

CONT BW

K Data for samples analyzed on cal curve C (0.2 ppm Cs)

Solution ID	Dilution Factor	Average Absorbance	K Conc (ppm) dilute soln	K Conc (ppm) Orig soln*
1	1500	0.1290	1.208	1813
2	1500	0.1316	1.232	1849
3	1500	0.1292	1.210	1815
4	1500	0.1238	1.161	1741
5	1500	0.1246	1.168	1752
6	1500	0.1170	1.098	1647
7	1500	0.1116	1.048	1572
8	1500	0.1056	0.993	1489
R0.05	1500	0.1184	1.111	1666
R0.1	1500	0.1184	1.111	1666
R0.2	1500	0.1048	0.985	1478

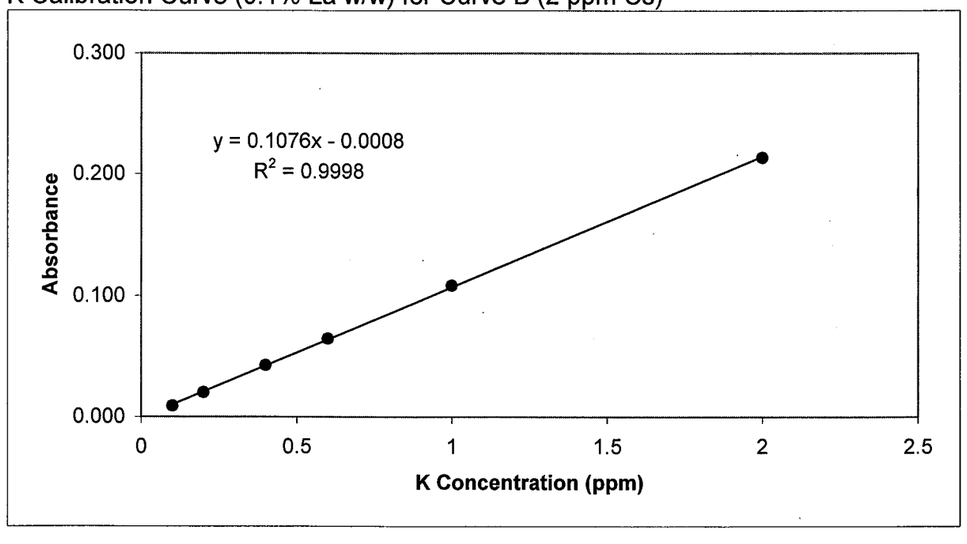
*Calculated by multiplying the concentration of dilute solution times the dilution factor

Potassium cal curve B (2ppm Cs)

K Standard Data (0.1% La w/w) for Curve B (2 ppm Cs)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
K1B	2	0.213	0.214	0.211	0.215	0.215	0.2136
K2B	1	0.107	0.108	0.109	0.109	0.108	0.1082
K3B	0.6	0.065	0.065	0.064	0.064	0.065	0.0646
K4B	0.4	0.043	0.042	0.043	0.042	0.043	0.0426
K5B	0.2	0.020	0.020	0.020	0.020	0.020	0.0200
K6B	0.1	0.009	0.009	0.009	0.009	0.009	0.0090

K Calibration Curve (0.1% La w/w) for Curve B (2 ppm Cs)



10OCT01

CONT BW

K Data for samples analyzed on cal curve B (2 ppm Cs)

Solution ID	Dilution Factor	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
9	1500	0.090	0.089	0.090	0.090	0.090	0.0898
10	1500	0.083	0.082	0.082	0.082	0.082	0.0822
11	1500	0.073	0.072	0.073	0.073	0.073	0.0728
12	1500	0.064	0.063	0.063	0.064	0.063	0.0634
13	1500	0.048	0.047	0.048	0.047	0.048	0.0476
14	1500	0.035	0.035	0.035	0.035	0.036	0.0352
R0.4	1500	0.076	0.075	0.075	0.076	0.076	0.0756
R0.7	1500	0.039	0.038	0.039	0.039	0.039	0.0388

K Data for samples analyzed on cal curve B (2 ppm Cs)

Solution ID	Dilution Factor	Average Absorbance	K Conc (ppm) dilute soln	K Conc (ppm) Orig soln*
9	1500	0.0898	0.8420	1263
10	1500	0.0822	0.7714	1157
11	1500	0.0728	0.6840	1026
12	1500	0.0634	0.5967	895
13	1500	0.0476	0.4498	675
14	1500	0.0352	0.3346	502
R0.4	1500	0.0756	0.7100	1065
R0.7	1500	0.0388	0.3680	552

*Calculated by multiplying the concentration of dilute solution times the dilution factor

Potassium cal curve A (20 ppm Cs)

K Standard Data (0.1% La w/w) for Curve A (20 ppm Cs)

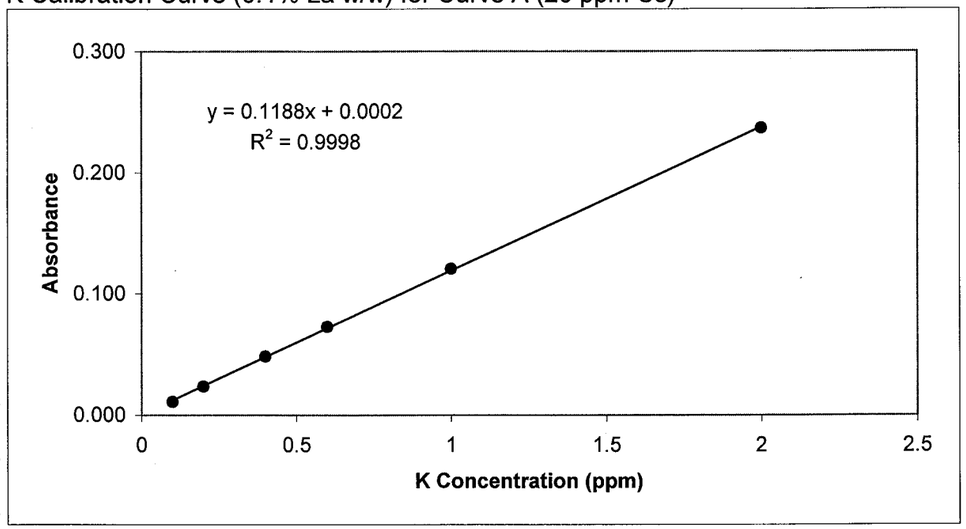
Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
K1A	2	0.237	0.236	0.237	0.238	0.236	0.2368
K2A	1	0.121	0.120	0.120	0.120	0.121	0.1204
K3A	0.6	0.073	0.073	0.073	0.072	0.072	0.0726
K4A	0.4	0.048	0.048	0.048	0.048	0.048	0.0480
K5A	0.2	0.024	0.023	0.024	0.023	0.023	0.0234
K6A	0.1	0.011	0.011	0.010	0.011	0.011	0.0108

BW 10OCT01

10 OCT 01

CONT FROM

K Calibration Curve (0.1% La w/w) for Curve A (20 ppm Cs)



K Data for samples analyzed on cal curve A (20 ppm Cs)

Solution ID	Dilution Factor	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
17	100	0.184	0.184	0.183	0.184	0.183	0.1836
18	100	0.095	0.095	0.095	0.095	0.095	0.0950
13	250	0.304	na	na	na	na	na
14	250	0.236	0.237	0.237	0.237	0.237	0.2368
15	250	0.191	0.192	0.192	0.190	0.190	0.1910
16	250	0.101	0.102	0.100	0.102	0.101	0.1012
17	250	0.066	0.065	0.065	0.065	0.066	0.0654
18	250	0.032	0.032	0.032	0.032	0.033	0.0322
R0.7	250	0.263	na	na	na	na	na

K Data for samples analyzed on cal curve A (20 ppm Cs)

Solution ID	Dilution Factor	Average Absorbance	K Conc (ppm) dilute soln	K Conc (ppm) Orig soln*
17	100	0.1836	1.5438	154
18	100	0.0950	0.7980	79.8
13	250	na	na	na
14	250	0.2368	1.9916	498
15	250	0.1910	1.6061	402
16	250	0.1012	0.8502	213
17	250	0.0654	0.5488	137
18	250	0.0322	0.2694	67.3
R0.7	250	na	na	na

*Calculated by multiplying the concentration of dilute solution times the dilution factor

10/10/01
AJ

Na^+ solutions required for measuring Na^+ conc. in initial solutions (Na/K - 0.05N 2nd set

200 PPm : $\frac{1000\ PPm \times 2\ ml}{10\ ml}$

400 PPm : $\frac{1000\ PPm \times 4\ ml}{10\ ml}$

600 PPm : $\frac{1000\ PPm \times 6\ ml}{10\ ml}$

800 PPm : $\frac{1000\ PPm \times 8\ ml}{10\ ml}$ 8 ml 2/15/02

KCl/NaCl-0.05N Set # 2 $X_{Na,i}$ to use	Calc Na PPm, i	Measured Na PPm, i
0.22	253	
0.45	517	
0.56	644	
0.68	782	787
0.8	920	921
0.92	1058	

10/11/01 AJ — Check electrode slope for Na^+ combination electrode following the procedure on page 92

I^{st} mV reading = −123.1 mV

2^{nd} mV reading = −75.3 mV

Difference = −75.3 − (−123.1)

= −75.3 + 123.1

= ~~47.8~~ 47.8 mV (slope low) AJ 2/15/02

Recondition the electrode for 30 sec. in reconditioning solution.

I^{st} mV reading = −130.3 mV

2^{nd} mV reading = −74.

KCl/NaCl-0.05N, 2^{nd} set

$X_{Na,i}$ to use	Cal. Na ppm i	Measured Na ppm i
0.22	253	252
0.45	517	514
0.56	644	639
0.68	787	
0.8	920	
0.92	1058	

10/12/01 AJ — 3M $CaCl_2$ solution replaced & bottles placed back in water bath.

Prepare 150 ml $CaCl_2$ solution (3M) [FS lot #947250]

10/16/01 AJ — 3M $CaCl_2$ solution was prepared by dissolving 220.53 g of $CaCl_2 \cdot 2H_2O$ (FS lot # 016231) in about 400 ml of water and dilute it to 500 ml mark.

Replace 3M $CaCl_2$ solution in bottles containing COV to convert to Ca-form. AJ 2/15/02

Na^+ std solutions needed:

20 ppm : $\frac{100\ ppm \times 2\ ml}{10\ ml}$

40 ppm : $\frac{100\ ppm \times 4\ ml}{10\ ml}$

60 ppm : $\frac{100\ ppm \times 6\ ml}{10\ ml}$

80 ppm : $\frac{100\ ppm \times 8\ ml}{10\ ml}$

The purpose is to remeasure the Na^+ conc. ~~for~~ AJ 2/15/02 CsCl/NaCl - 0.05N final experimental solutions

BW 2/14/02

Mixture # CsNa-05	ECs,I to use	Calc. Na ppm,initial	Meas. Na ppm,initial	Calc. Na ppm,f	Meas. diluted Na ppm,f	Meas. Na ppm,f
1	0.05	1092		1149	119, 118	
2	0.05	1092		1148	119, 117	
3	0.05	1092		1146	116, 116	
4	0.05	1092		1142	119, 119	
5	0.2	920		1132	115, 117	
6	0.2	920		1113	113, 114	
7	0.2	920		1100	113, ~~115~~ AS 21/5/02	114
8	0.2	920		1071	108, ~~110~~	110
9	0.2	920		1042	~~104~~ AS 10/16/01 105, AJ 21/5/02	105
10	0.2	920		1002	101, 101	
11	0.4	690		945	95.1, 95.1	
12	0.4	690		862	87.6, 88	
13	0.4	690		806	80.0, 81.1	
14	0.6	460		736	73.3, 73.1	
15	0.6	460		650	64.2, 63.3	
16	0.6	460		542	56.7, 57.8	
17	1	0		404	35.5, 36.7	
18	1	0		228	19.5, 20.9	
19	1	0		63	7.46, 8.76, 8.94	

10/17/01

AJ

Objective :- To prepare several KCl-$CaCl_2$ solutions with a total normality of 0.0005N and a fixed K/Ca ratio.

Equipment & Supplies :-

$CaCl_2 \cdot 2H_2O$ (FS lot # 016231)

3 - 2L Plastic bottles (PP or PC)

3 - 1L PP bottles, 1-25 ml PP bottles

2 - 500 ml PP bottles, 2 - 50 ml bottles

3 - 250 ml PP bottles, 2-100 ml PP bottles

AE-240 Mettler weighing balance

Stir plate & magnetic stirrers

Nanopure water

Procedure

1. Prepare 1000 ml of KCl-$CaCl_2$ aqueous mixture with a total normality of 0.0005 N and a fixed K/Ca ratio by taring reagent grade KCl and $CaCl_2 \bullet 2H_2O$ in the amounts given in Table 1 onto a clean (acid-washed) 250-ml or 500-ml beaker. Keep the beaker covered with parafilm between additions of reagents to minimize adsorption of water from the atmosphere. Add about 100 ml ultrapure water (>17 megaohm resistivity) to the beaker, add a stir bar and dissolve the salts completely on a stir plate (add more water if necessary). Retrieve the stir bar with a magnetic rod, then carefully rinse the rod and stir bar with ultrapure water, making sure to catch all the washings in the beaker.

(Note: Instead of 250-mL or 500 mL beaker, one can use a weighing boat for taring the reagent. This procedure can be modified as needed).

Decant the solution into a clean 1000 ml volumetric flask (with the help of a glass rod); rinse the beaker (and glass rod) with ultrapure water several times, carefully transferring all washings into the volumetric flask. Then fill the flask to about 2-3 inches below the mark with ultrapure water; swirl or shake the flask. Let stand for a few seconds, then add water dropwise up to the mark; remix.

Transfer the solution into a clean 1000 ml polypropylene bottle. Label the bottle (e.g., $KCl/CaCl_2 * 0.0005N * 0.1X_{Ca}$, plus Date and Initial, and lab notebook volume & page#).

Table 1.

$X_{i,Ca}$ (0.0005 N) (KCl-$CaCl_2$ soln.)	Wt. $CaCl_2.6H_2O$ needed for 1000 ml	Wt. KCl needed for 1000 ml	Wt. $CaCl_2.2H_2O$ used	Wt. KCl used
1.0 (several batches needed)	0.10954	0		

10/19/01 Replace 3M $CaCl_2$ solution in bottles containing CDV and placed in waterbath at 70°C to convert to Ca-form.

3M $CaCl_2$:- 500 ml of 3M $CaCl_2$ was prepared by dissolving 220.53 g of $CaCl_2 \cdot 2H_2O$ (FS lot # 016231) in water and dilute it to 500 ml mark.

CDV * 100/200 * HL * CP * Kf (prepared by AJ 11/25/01

EXPERIMENT KCACL-.0005N

Mixture # CaK-0005*	$E_{Ca,i}$ to use	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc. Ca ppm,i	Calc. Ca ppm,f	Calc. K ppm,i	Calc. K ppm,f	10/19/01 weight of K-zeol. used (gm)
1	1.0	0.1507	25	10.0	0.16	0.0	19.23	0.1510
2	1.0	0.1473	50	10.0	0.39	0.0	18.80	0.1475
3	1.0	0.1162	50	10.0	0.52	0.0	18.53	0.1165
4	1.0	0.16020	100	10.0	0.85	0.0	17.89	0.1605
5	1.0	0.10428	100	10.0	1.49	0.0	16.64	0.1043
6	1.0	0.2039	250	10.0	2.02	0.0	15.61	0.2040
7	1.0	0.1447	250	10.0	2.92	0.0	13.85	0.1450
8	1.0	0.1158	250	10.0	3.58	0.0	12.56	0.1160 AT 7/15/02
9	1.0	0.1660	500	10.0	4.59	0.0	10.59	0.1663
10	1.0	0.1325	500	10.0	5.25	0.0	9.30	0.1326
11	1.0	0.1874	1000	10.0	6.19	0.0	7.47	0.1875
12	1.0	0.1478	1000	10.0	6.76	0.0	6.37	0.1482
13	1.0	0.1024	1000	10.0	7.51	0.0	4.90	0.1025
14	1.0	0.1591	2000	10.0	7.94	0.0	4.06	0.1593
15	1.0	0.1075	2000	10.0	8.48	0.0	3.00	0.1078
16	1.0	0.0819	2000	10.0	8.78	0.0	2.42	0.0820

10/21/01 AJ Replace 3M $CaCl_2$ solution in bottles containing CDV to be exchanged to Ca-form

10/22/01 AJ 3M $CaCl_2$: 110.26 g of $CaCl_2 \cdot H_2O$ (FS lot # 01623) was dissolved in nanopure water & filled up to 250 ml mark.

Sampled KCl/NaCl-0.05N * #2* set experimental solution from #1 to #16 to be analyzed for Na^+ conc.

10/22/01 CONT BAW

AA analysis for potassium of Cs/K binary solutions with zeolite

The potassium concentration of the cesium/potassium binary solutions with zeolite was to be determined using AA. Dilutions would also be required in order to bring the potassium concentration into the calibration curve range (0.1 to 2 ppm K). The solutions analyzed were 5 sample solutions (420/144). These were newly prepared solutions. Older solutions of similar make have already been analyzed on the AA (420/148). The reference solution for these 5 samples did not contain potassium so the reference solution was not analyzed for potassium. Information from the analysis of the older solutions (420/133-135 and 420/137-148) was used in deciding how to analyze the newly prepared solutions.

Based on the previous analysis of older solutions, I decided to use the 20 ppm cesium cal curve (matrix effect) and analyze DF250 of all solutions. I also made a DF500 of solution 14 since the previous analysis of the DF250 was at the high point of the cal curve.
Table 1 contains the target concentrations in the undiluted samples. Table 2 contains the concentrations of the diluted samples (DF = dilution factor) based on Table 1.

Table 1

Sample ID	Target Conc K (ppm)	Target Conc Cs (ppm)
14	563	11.26
15	453	9.06
16	252	5.04
17	161	3.22
18	77	1.54

Table 2

Sample ID	DF 500 Target Conc		DF 250 Target Conc	
	K (ppm)	Cs (ppm)	K (ppm)	Cs (ppm)
14	1.126	9.4644	2.252	18.9288
15	0.906	10.2088	1.812	20.4176
16	0.504	11.5788	1.008	23.1576
17	0.322	12.1966	0.644	24.3932
18	0.154	12.7672	0.308	25.5344

10/22/01 Preparation of DF500 samples
CONT BAW DF500 will be a two fold dilution 1 to 50 and 1 to 10
The initial (first dilution) will be performed as follows:
Added 1mL (eppendorf pipette) of sample to a 50mL vol flask and diluting to mark with nanopure water.
Labeled Xi500 where X = sample #, i = intermediate, 500 = ultimate DF. The final (second) dilution step will be performed later.

Preparation of DF250 samples
DF 250 will be a two fold dilution: 1 to 25 and 1 to 10
The initial (first dilution) will be performed as follows:
Added 1mL (eppendorff pipette) of sample to a 25mL vol flask and diluted to mark with nanopure water.
Labeled Xi250 where X = sample #, i = intermediate, 250 = ultimate DF. Final dilution step will be performed later.

Final Dilution step
The final dilution step was the same for all dilutions (DF500 + DF250) - a 1 to 10 dilution

Added 1mL of (vol pipette) 1% La (w/w) (Soln A 463/40) and 1mL (vol ~~pipette~~ BAW 10/22/01 eppendorf pipette) of sample to a 10mL vol flask and diloted to mark with nanopure water.

10/22/01 **Potassium AA Analysis**
CONT BAW

Perkin Elmer 3100 Atomic Absorption Spectrophotometer
K - hallow cathode lamp 8mA Fisher 14-386-106H
λ = 766.5, slit = 0.7nm, high, Air acetylene flame
Blank = 0.1% La (w/w) soln B 463/41
Integration time for samples = 3 sec

~~curve "C"~~ BW 10/22/01

Curve "A" Potassium - 20ppm Cesium (420/138)

ID	Conc (ppm)	Trial 1	2	3	4	5
K1A	2	0.250	0.248	0.248	0.249	0.248
K2A	1	0.125	0.126	0.125	0.125	0.125
K3A	0.6	0.074	0.075	0.075	0.075	0.075
K4A	0.4	0.049	0.048	0.048	0.048	0.049
K5A	0.2	0.023	0.023	0.023	0.023	0.023
K6A	0.1	0.011	0.012	0.012	0.015	0.011

Zero cal BAW

22OCT01 ID Trial

CONT BW	1	2	3	4	5
14DF500	0.115	0.117	0.116	0.116	0.116
14DF250	0.242	0.244	0.244	0.241	0.241
15DF250	0.192	0.190	0.191	0.192	0.190
16DF250	0.108	0.110	0.110	0.110	0.108
17DF250	0.069	0.069	0.069	0.069	0.069
18DF250	0.034	0.034	0.034	0.034	0.034
K3A	0.074	0.074	0.074	0.074	0.074

Max correction for A/Z was 0.001

AA cleaned by aspirating with 0.2N HNO_3 (420/140) for 5 minutes after analysis completed.

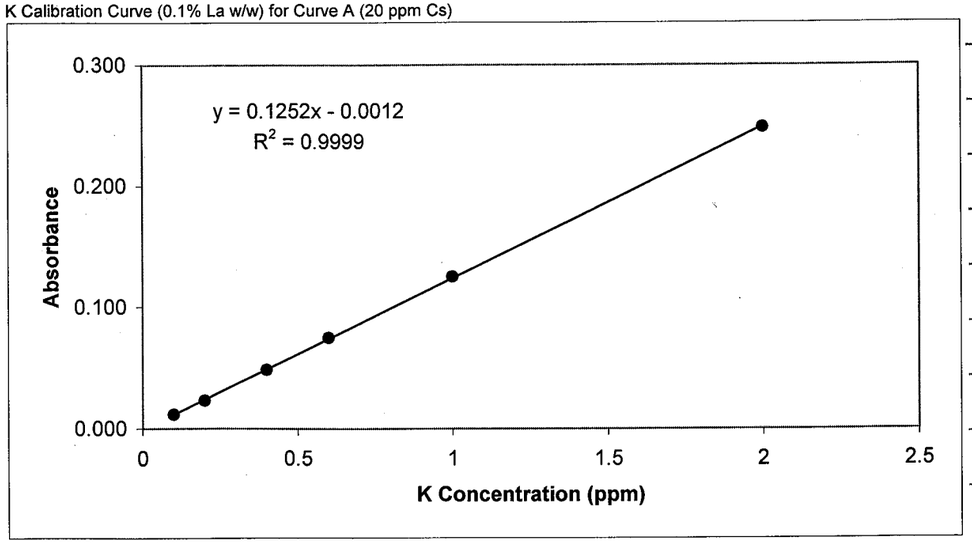
23OCT01 BAW

Potassium cal curve A (20 ppm Cs)

K Standard Data (0.1% La w/w) for Curve A (20 ppm Cs)

Solution ID	Ca Std (ppm)	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
K1A	2	0.250	0.248	0.248	0.249	0.248	0.2486
K2A	1	0.125	0.126	0.125	0.125	0.125	0.1252
K3A	0.6	0.074	0.075	0.075	0.075	0.075	0.0748
K4A	0.4	0.049	0.048	0.048	0.048	0.049	0.0484
K5A	0.2	0.023	0.023	0.023	0.023	0.023	0.0230
K6A	0.1	0.011	0.012	0.012	0.011	0.011	0.0114

K Calibration Curve (0.1% La w/w) for Curve A (20 ppm Cs)



23OCT01 CONT BAW

Potassium cal curve A (20ppm Cs)

K Data for samples analyzed on cal curve A (20 ppm Cs)

Solution ID	Dilution Factor	Absorbance Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average Absorbance
14	500	0.115	0.117	0.116	0.116	0.116	0.1160
14	250	0.242	0.244	0.244	0.241	0.241	0.2424
15	250	0.192	0.190	0.191	0.192	0.190	0.1910
16	250	0.108	0.110	0.110	0.110	0.108	0.1092
17	250	0.069	0.069	0.069	0.069	0.069	0.0690
18	250	0.034	0.034	0.034	0.034	0.034	0.0340

K Data for samples analyzed on cal curve A (20 ppm Cs)

Solution ID	Dilution Factor	Average Absorbance	K Conc (ppm) dilute soln	K Conc (ppm) Orig soln*
14	500	0.1160	0.9361	468
14	250	0.2424	1.9457	486
15	250	0.1910	1.5351	384
16	250	0.1092	0.8818	220
17	250	0.0690	0.5607	140
18	250	0.0340	0.2812	70

*Calculated by multiplying the concentration of dilute solution times the dilution factor

Comparison of 8 Oct 01 data (420/147+148) and 22 Oct 01 Data (420/159)

Summary data for DF250 samples

Solution ID	Potassium Conc (ppm) 8 Oct 01 data	Potassium Conc (ppm) 22 Oct 01 data	Percent Difference
14	498	486	2.3
15	402	384	4.4
16	213	220	-3.7
17	137	140	-2.2
18	67.3	70.3	-4.4

Summary data for all DFs. Values inside table are K conc in ppm.

Detail		Sample 14	15	16	17	18
DF1500	8 Oct	502				
DF250	8 Oct	498				
DF500	22 Oct	468				
DF250	22 Oct	486				
DF250	8 Oct		402			
DF250	22 Oct		384			
DF250	8 Oct			213		
DF250	22 Oct			220		
DF250	8 Oct				137	
DF100	8 Oct				154	
DF250	22 Oct				140	
DF250	8 Oct					67.3
DF100	8 Oct					79.8
DF250	22 Oct					70.3

10/23/01 Na^+ std solutions required :-
AJ

10 ppm : $\frac{100\ ppm \times 1\ ml}{10\ ml}$

30 ppm : $\frac{100\ ppm \times 3\ ml}{10\ ml}$

50 ppm : $\frac{100\ ppm \times 5\ ml}{10\ ml}$

75 ppm : $\frac{100\ ppm \times 7.5\ ml}{10\ ml}$

100 ppm : $\frac{100\ ppm \times 10\ ml}{10\ ml}$

125 ppm : $\frac{1000\ ppm \times 125\ ml}{10\ ml}$

Mixture # KNa05 #2*	$X_{Na,i}$ to use	Calc. Na PPm, f	Measured Na PPm, f diluted to 1/10	Measure Na PPm final
1	0.22	115	14.6, 15.3	146, 153
2	0.45	230	29.1, 29.9	291, 299
3	0.56	345	AJ 2/15/02 ~~400~~ 40.0, 40.4	400, 404
4	0.68	460	51.3, 51.2	513, 512
5	0.8	575	61.1, 61.6	611, 616
6	0.92	690	70.5, 72.9	705, 729
7	1	805	80.8, 80.0	808, 800
8	1	874	86.2, 86.6	862, 866

9	1	920	91.1, 91.1	911, 911
10	1	977	95.9, 98.6	959, 986
11	1	1035	103, 103	1030, 1030
12	1	1063	105, 107	1050, 1070
13	1	1092	107, 109	1070, 1090
14	1	1115	109, 111	1090, 1110
15	1	1127	111, 113	1110, 1130
16	1	1138	112, 113	1120, 1130

10/23/01
CONT BALS

Preparation of $SrCl_2$-KCl Solutions (0.0005N)

Prepare 7000mL of $SrCl_2$-KCl aqueous mixture with a total normality of 0.0005 N by starting with $SrCl_2 \cdot 6H_2O$ and no KCl. See table 1 for the mass of $SrCl_2 \cdot 6H_2O$ required for the solution. Parafilm was used to cover the weighing boat between additions of reagent to minimize adsorption of water from the atmosphere. The appropriate amount of $SrCl_2 \cdot 6H_2O$ was added. The weighing boat was removed from the balance and nanopure water was carefully added from a squirt bottle. The solution was left standing for about a minute and then decanted into a 2000mL volumetric flask. The weighing boat was washed with nanopure water several times, carefully transferring all washings into the volumetric flask. The volumetric flask is filled to about 2-3 inches below the mark with nanopure water and swirled for mixing. Then nanopure water was added dropwise up to the mark and remixed. The solution in the volumetric flask was then poured into a 2 gallon polypropylene bottle. The 2000mL volumetric flask was refilled to the mark with nanopure water and poured into the 2 gallon polypropylene bottle (solution total 4000mL). The 2000mL volumetric flask was again refilled to the mark with nanopure water and poured into the 2 gallon polypropylene bottle (solution total 6000mL). A 1000mL volumetric flask was filled to the mark with nanopure water and poured into the 2 gallon polypropylene bottle (solution total 7000mL). This bottle was labeled as follows: $SrCl_2$/KCl*0.0005N*1.0E_{Sr}.

Table 1.

$E_{Sr,i}$ (0.0005N) $SrCl_2$-KCl soln	Volume (mL)	Mass (g) of $SrCl_2 \cdot 6H_2O$ needed	Mass (g) of KCl needed	Mass (g) of $SrCl_2 \cdot 6H_2O$ used
1.0	7000	0.93317	0	0.93321

10mL aliquot of ↑ saved in pp bottle for Aft analysis.

10/23/01 Strontium Chloride - $SrCl_2 \cdot 6H_2O$ - Fisher S541-500, lot #
lot BAW 000052, rec 2/9/01, ~~exp~~ BW 10/23/01 opened 2/21/01
nanopure water
Mettler AE240 electronic balance

10/24/01
AJ K^+ std solutions required !-

39.1 ppm or 0.001 M : $\frac{0.1M \times 0.1 ml}{10 ml}$

78.2 ppm or 0.002 M : $\frac{0.1M \times 0.2 ml}{10 ml}$

117.3 ppm or 0.003 M : $\frac{0.1M \times 0.3 ml}{10 ml}$

156.4 ppm or 0.004 M : $\frac{0.1M \times 0.4 ml}{10 ml}$

195.5 ppm or 0.005 M : $\frac{0.1M \times 0.5 ml}{10 ml}$

19.55 ppm or 0.0005 M : $\frac{0.1M \times 0.05 ml}{10 ml}$ BW 2/15/02

9.78 ppm or 0.00025 M : $\frac{0.1M \times 0.025 ml}{10 ml}$

3.91 ppm or 0.0001 M : $\frac{0.1M \times 0.010 ml}{10 ml}$

BW 2/14/02

Mixture # KNa05 #2*	$X_{Na,i}$ to use	~~AJ 10/25/01~~ Calc. K PPm, f	Measured K PPm, f diluted	Measured K PPm, final
1	0.22	1759	173, 171, 172	1730, 1710, 1720
2	0.45	1564	148, 150, 151	1480, 1500, 1510
3	0.56	1368	129, 130, 131	1290, 1300, 1310
4	0.68	1173	~~108~~ 109, 110 AJ 2/15/02	1080, 1090, 1100
5	0.80	977	89.6, 89.9, 89.2	896, 899, 892
6	0.92	782	75.5, 75.8, 76.1	755, 758, 761
7	1.0	586	56.6, 57.0, 56.8	566, 570, 568
8	1.0	469	46.6, 46.8, 47.0	466, 468, 470
9	1.0	391	38.8, 39.1, 38.9	388, 391, 389
10	1.0	293	29.3, 29.2, 29.1	293, 292, 291
11	1.0	195	19.3, 19.4, 19.5	193, 194, 195
12	1.0	147	14.6, 14.5, 14.7	146, 145, 147
13	1.0	98	9.9, 9.7, 9.8	99, 97, 98
14	1.0	59	5.8, 5.9, 6.0	58, 59, 60
15	1.0	39	3.8, 3.9, 3.8	38, 39, 38
16	1.0	20	2.0, 2.1, 1.9	20, 21, 19

10/25/01 Preparation of $SrCl_2$-KCl Solutions (0.0005N) – Addition of zeolite
BAW

Table 1 provides a breakdown of the 17 solutions to be prepared. It contains the target amounts of zeolite and ESr1.0 solution used to make the solutions. It also contains initial and final target concentrations of Sr and K. The potassium form of the zeolite (k-zeo) will be used. The appropriate amount of k-zeo will be placed on a tared piece of weighing paper that has been folded in quarter sections. K-zeo masses were recorded in Table 2. The k-zeo is then carefully poured into an appropriately sized polypropylene bottle. A policeman (glass rod with rubber tip) was used to tap the outside of the paper or scrub the inside of the paper to completely transfer the solid from the paper to the bottle.

10/25/01
CONT BAW

TABLE 1.

EXPERIMENT $SrCl_2$/KCl-0.0005N

Mixture # SrK-0005*	$E_{Sr,i}$ to use	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc. Sr ppm,i	Calc. Sr ppm,f	Calc. K ppm,i	Calc. K ppm,f
1	1.0	0.1527	25	21.9	0.06	0.0	19.49
2	1.0	0.1521	50	21.9	0.15	0.0	19.41
3	1.0	0.1214	50	21.9	0.21	0.0	19.36
4	1.0	0.1723	100	21.9	0.35	0.0	19.23
5	1.0	0.1188	100	21.9	0.66	0.0	18.96
6	1.0	0.0976	100	21.9	0.96	0.0	18.69
7	1.0	0.1894	250	21.9	1.59	0.0	18.13
8	1.0	0.1624	250	21.9	2.17	0.0	17.61
9	1.0	0.1299	250	21.9	3.33	0.0	16.58
10	1.0	0.1119	250	21.9	4.31	0.0	15.71
11	1.0	0.1769	500	21.9	6.09	0.0	14.11
12	1.0	0.1496	500	21.9	7.46	0.0	12.89
13	1.0	0.1138	500	21.9	9.70	0.0	10.90
14	1.0	0.1866	1000	21.9	11.23	0.0	9.52
15	1.0	0.1344	1000	21.9	13.50	0.0	7.50
16	1.0	0.1057	1000	21.9	14.92	0.0	6.24
17	1.0	0.0706	1000	21.9	16.86	0.0	4.50

Zeolite: Potassium form
CDV*200/325*4C*WA*RC*ML*RFe* 420/54-67
by AJ 5/2/01
Mettler AE240 electronic balance
Weighing Paper - Fisher 09-898-12A 3"x3"

10/25/01
CONT BAW

Table 2.

Mixture # SrK-0.0005	$E_{Sr,I}$ to use	Volume (mL) of $E_{Sr,I}$ to use	Mass (g) of k-zeo
1	1.0	25	0.1529
2	1.0	50	0.1520
3	1.0	50	0.1214
4	1.0	100	0.1722
5	1.0	100	0.1189
6	1.0	100	0.0977
7	1.0	250	0.1894
8	1.0	250	0.1624
9	1.0	250	0.1299
10	1.0	250	0.1119
11	1.0	500	0.1769
12	1.0	500	0.1496
13	1.0	500	0.1138
14	1.0	1000	0.1866
15	1.0	1000	0.1344
16	1.0	1000	0.1057
17	1.0	1000	0.0705

10/26/01
AJ

Na^+ std solutions required :-

120 ppm : $\frac{1000\ ppm \times 1.2\ ml}{10\ ml}$

100 ppm : $\frac{100\ ppm \times 10\ ml}{10\ ml}$

KCl/NaCl - 0.05N Set #2 $X_{Na,i}$ to use	Cal. Na PPM, initial	Initial Measured Na PPM diluted	Measured Na PPM, initial
0.92	1058	106	1060

remove zeolite from water bath and wash it several times to obtain Ca-form COV.

10/30/01 AJ — Continue washing Ca-form COV and test the supernatant (NA AJ 2/15/02) liquid for (AJ 2/15/02) Cl^- ion with 0.1M ~~AgNO3~~ $AgNO_3$ (AJ 2/15/02). Wash until supernatant does (AJ 2/15/02) doesn't ~~form~~ (AJ 2/15/02) form white precipitate with few drops of 0.1M $AgNO_3$. Place the beaker in the oven at 55-60°C to dry. Cover the beaker partially with Kim wipe & rubber band.

10/31/01 AJ — Remove Ca form COV from the oven and let it cool. ~~weight~~ weigh (AJ 11/5/02) it and store

COV * 200/325 * UC * RC * HL * RFc * Caf = 39.14 g.

11-02-2001 BAW — Preparation of CsK solns for LSA analysis

The solns prepared for analysis were CsK-0.05N reruns for mixture #s 14-18 and the reference soln. Mixtures from 420/144 and reference soln already sampled with HNO_3 420/136

11-02-2001

CONT BAW — Cs blank from original analysis of 0.05CsK solns LSA analysis done in duplicate (lower case a/b). Second set (rerun) indicated by suffix "2". LSA vials labeled, 0.5mL of 0.1N HNO_3 (463/94) added (eppendorf pipettee) and vial weighed (Mettler AE240 balance). 0.5mL of sample added (eppendorff pipette) to LSA vial LSA vial reweighed. Added 5mL (bottle top dispenser) of Ultima Gold AB (Packard cat# 6013309, lot# 91,9031)

Challange mass (target 20.0001g)

start of analysis = 20.0001g end of analysis = 20.0001g

Date Measured →	11-02-2001	11-02-2001
Test Tube ID	Mass (g) of Vial and HNO3	Mass (g) after adding sample
0.05CsK14a2	7.7699	8.2657
0.05CsK14b2	7.8331	8.3291
0.05CsK15a2	7.8010	8.2942
0.05CsK15b2	7.8614	8.3545
0.05CsK16a2	7.9162	8.4076
0.05CsK16b2	7.8594	8.3481
0.05CsK17a2	7.7999	8.2907
0.05CsK17b2	7.8683	8.3532
0.05CsK18a2	7.8280	8.3194
0.05CsK18b2	7.9143	8.4073
ECs 1.0a *	7.3797	7.8788
ECs 1.0b *	7.3310	7.7714

* from 420/136 - 1mL soln in LSA vial. Mass measurements about 0.5g. Assume 0.5mL of 0.1N HNO_3 already added.

11/2/01 K^+ Stds. required:
AJ

19.55 ppm or 0.0005 M : $\frac{0.1M \times 0.5ml}{100\ ml}$

9.78 ppm or 0.00025 : $\frac{0.1M \times 0.25ml}{100\ ml}$

3.91 PPM or 0.0001 M : $\frac{0.1M \times 0.10ml}{100\ ml}$

1.96 ppm or 0.00005 M : $\frac{0.1M \times 0.05\ ml}{100\ ml}$

Note: Stds. were prepared in 100 ml ~~volumetric~~ volume for calibration. AJ 11/5/01

Mixture # CaK-0005*	ECa,i	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc. K ppm,i	Calc. K ppm,f	Meas. K ppm, f
1	1	0.1507	25	0	19.23	
2	1	0.1473	50	0	18.8	48.6
3	1	0.1162	50	0	18.53	44.6
4	1	0.1602	100	0	17.89	37.8
5	1	0.10428	100	0	16.64	30.6
6	1	0.2039	250	0	15.61	26.2
7	1	0.1447	250	0	13.85	21.8, 21.4, 21.4
8	1	0.1158	250	0	12.56	18.7, 18.5
9	1	0.166	500	0	10.59	14.5, 14.5
10	1	0.1325	500	0	9.3	12.4, 12.5
11	1	0.1874	1000	0	7.47	9.36
12	1	0.1478	1000	0	6.37	7.99
13	1	0.1024	1000	0	4.9	6.17
14	1	0.1591	2000	0	4.06	5.23
15	1	0.1075	2000	0	3	3.86
16	1	0.0819	2000	0	2.42	3.03

11/6/01 AJ

ppm Ca^{+2}	Molar Ca^{2+}
4.01	10^{-4} M
40.10	10^{-3} M
400.80	10^{-2} M
4008.0	10^{-1} M

10 ppm or 0.00025 M : $\frac{0.1M \times 0.25ml}{100\ ml}$

8 ppm or 0.0002 M : $\frac{0.1M \times 0.200ml}{100\ ml}$

6 PPM or 0.00015 M : $\frac{0.1M \times 0.150\ ml}{100\ ml}$

20 ppm or 0.0005 M : $\frac{0.1M \times 0.500\ ml}{100\ ml}$

30 ppm or 0.00075 M : $\frac{0.1M \times 0.750\ ml}{100\ ml}$

Note: 100 ppm or 0.0025 M : $\frac{0.1M \times 2.5\ ml}{100\ ml}$

100 ppm std was prepared and other dilutions were made using 100 ppm.

Electrode operation :- Follow procedure from Cole-Palmer® 27502-08,-09 operating instructions for Ca^{+2} electrodes.

I^{st} mV = 19.2 mV
2^{nd} mV = 42.7 mV
] difference = 23.5 mV
[← 25–29 mV

Mixture # CaK-0005*	ECa,i	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc.Ca ppm,i	Calc. Ca ppm,f	Calc. Ca ppm,f
1	1	0.1507	25	10	0.16	1.67
2	1	0.1473	50	10	0.39	4.87
3	1	0.1162	50	10	0.52	6.45
4	1	0.1602	100	10	0.85	10.1
5	1	0.10428	100	10	1.49	13.5
6	1	0.2039	250	10	2.02	15.1
7	1	0.1447	250	10	2.92	18.5
8	1	0.1158	250	10	3.58	20.0
9	1	0.166	500	10	4.59	20.9
10	1	0.1325	500	10	5.25	22
11	1	0.1874	1000	10	6.19	27.5
12	1	0.1478	1000	10	6.76	28.7
13	1	0.1024	1000	10	7.51	31.8
14	1	0.1591	2000	10	7.94	32.7
15	1	0.1075	2000	10	8.48	34.7
16	1	0.0819	2000	10	8.78	35.7

measured on 11/9/01

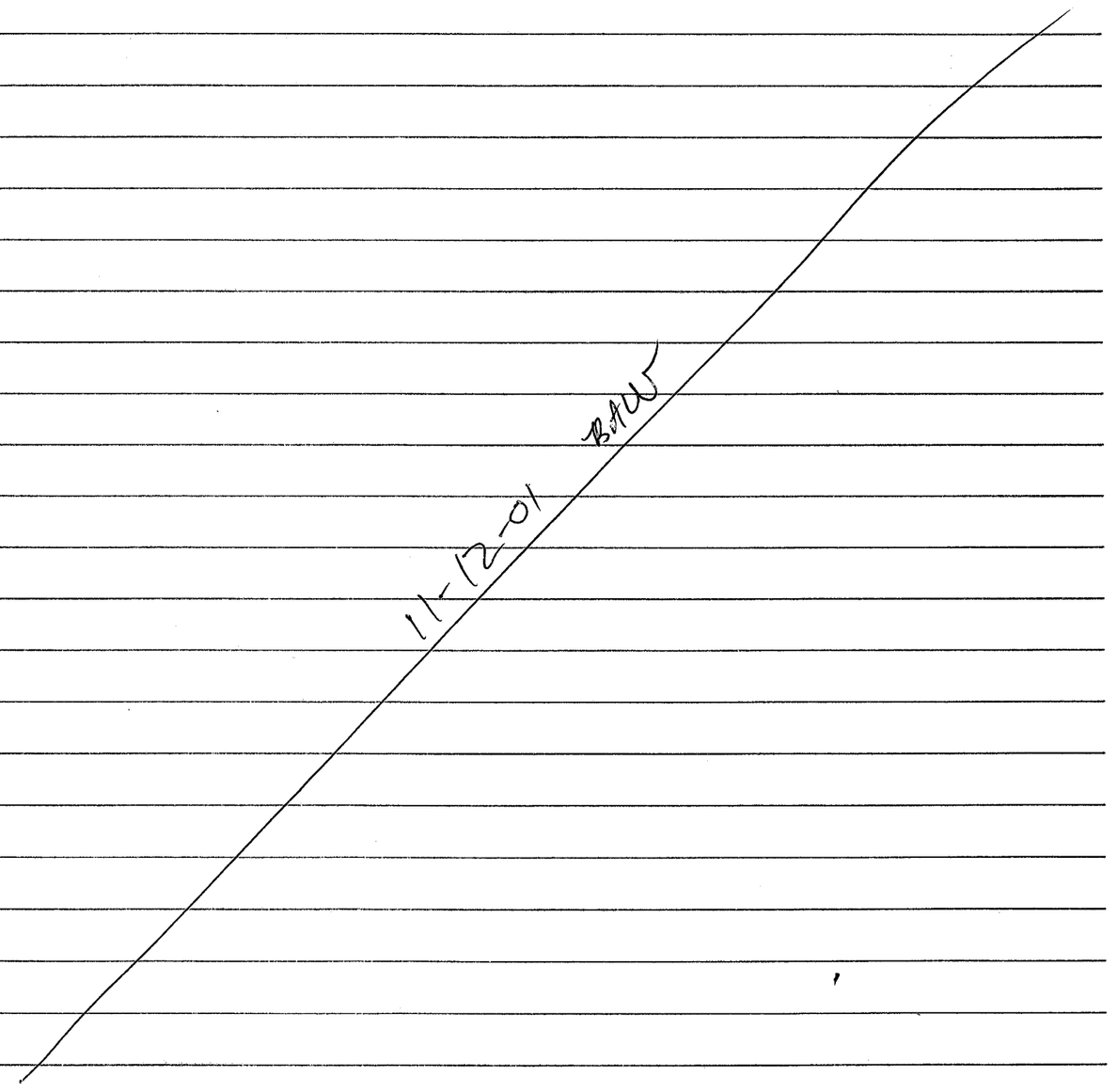
11/9/01 Continue to measure final Ca^{+2} conc. for CaK-0.0005N AT ion exchange experiments. The values are recorded in the ~~above~~ AT table above. 10/9/01

Ca^{+2} std. required: 30 ppm, 25 ppm, 20 ppm, 15 ppm 10 ppm, 5ppm, 3 ppm & 1 ppm.

100 ppm std was prepared from 0.1M Ca^{+2} std. and all other dilutions were made from 100 ppm.

100 ppm or 0.0025 M : $\frac{0.1M \times 2.5ml}{100 ml}$

200 ml of 100 ppm std was prepared.



12 NOV 01 BAW — LSA results for Potassium/Cesium Ion Exchange solns. Second set (partial) of solns 14 to 18 (420/166 to 167)

11/6/01 11:36:34 PM — QuantaSmart (TM) - 1.10 — Page # 1
Protocol# 5 - manual_cpm.lsa — Serial# 405314 — User: Bertetti

Assay Definition-

Assay Description:

Assay Type: CPM
Report Name: Report1
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\manual_cpm
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\manual_cpm
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\manual_cpm\Manual_cpm.010

Count Conditions-

Nuclide: Manual
  Quench Indicator: SIS
  External Std Terminator (sec): n/a
  Pre-Count Delay (min): 0.00
Quench Set: n/a
Count Time (min): 600.00
Count Mode: Normal
Assay Count Cycles: 1 — Repeat Sample Count: 1
#Vials/Sample: 1 — Calculate % Reference: Off

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

Regions	LL	UL	Bkg Subtract	2Sigma % Terminator
A	0.0	2000.0	1st Vial	0.00
B	0.0	600.0	1st Vial	2.00
C	0.0	100.0	1st Vial	0.00

Count Corrections-

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

Half Life-

Half Life Correction: Off

Regions	Half Life	Units	Reference Date	Reference Time
A				
B				
C				

IPA Block Data
Software Version IC: 2.09
Software Version EC: 1.10
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 10.44 Date Processed: 9/24/01 4:37:10 PM
14C Chi Square: 19.61 Date Processed: 9/24/01 4:37:10 PM
3H E^2/B (0-18.6 keV and 1-18.6 keV): 281.02 Date Processed: 9/24/01 4:37:10 PM
14C E^2/B (0-156 keV and 1-156 keV): 522.98 Date Processed: 9/24/01 4:37:10 PM
3H Efficiency (0-18.6 keV): 66.06 Date Processed: 11/6/01 12:08:13 PM
14C Efficiency (0-156 keV): 97.14 Date Processed: 11/6/01 12:08:13 PM
IPA Background Date Processed: 9/24/01 4:37:10 PM
3H Background CPM (0-18.6 keV): 15.37 Date Processed: 9/24/01 4:37:10 PM
14C Background CPM (0-156 keV): 22.55 Date Processed: 9/24/01 4:37:10 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100
========== Errors and Warnings ==========
========== End of Errors and Warnings ==========

12 NOV 01 BAW (CONT)

11/6/01 11:36:34 PM — QuantaSmart (TM) - 1.10 — Page # 2
Protocol# 5 - manual_cpm.lsa — Serial# 405314 — User: Bertetti

Cycle 1 Results

S#	Count Time	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS
MESSAGES 1	*****	26.48	1.59	22.20	1.73	16.63	2.00	863.24
B 14a 2	7.49	1314.24	2.04	1314.92	2.03	799.93	2.61	310.20
14b 3	7.48	1316.83	2.04	1317.37	2.03	798.61	2.61	316.63
15a 4	7.07	1392.05	2.04	1392.65	2.03	832.74	2.63	317.49
15b 5	7.14	1380.10	2.03	1380.46	2.03	840.65	2.61	311.82
16a 6	6.19	1592.42	2.03	1594.12	2.03	973.52	2.60	311.01
16b 7	6.38	1546.24	2.03	1545.67	2.03	925.69	2.63	320.76
17a 8	6.05	1633.68	2.03	1632.18	2.03	982.21	2.62	321.02
17b 9	6.00	1647.35	2.03	1646.30	2.03	992.20	2.61	320.83
18a 10	5.85	1689.93	2.03	1690.45	2.02	1007.30	2.63	316.27
18b 11	5.80	1705.07	2.03	1704.70	2.02	1009.58	2.64	322.68
10a 12	5.43	1823.61	2.02	1823.10	2.02	1103.26	2.60	316.81
10b 13	6.25	1578.96	2.03	1579.40	2.03	946.09	2.62	317.73

11/14/01 AJ — $KCl \cdot CaCl_2$ * 0.0005N * 0.1 X Eq — measured Initial Ca Conc. PPM 29.7

Ca^{+2} std solutions required: ~~50~~ 50 ppm & ~~25~~ 25 ppm
AJ 2/15/02 — AJ 12/15/02
Both solutions were prepared by diluting 100 ppm

100 ppm Ca^{+2} or 0.0025 M : $\frac{0.1M \times 2.5\ ml}{100\ ml}$

5 DEC 01 BAW

5DEC01 BAW

$SrCl_2$-KCl Ion Exchange - Soln Prep

- Addition of 5r-90 radioactive soln to 7L non radioactive soln.

Added 7mL (eppendorf pipette) of spike 51A to 7L soln of $SrCl_2$ (420/161-162)

6DEC01 BAW

$SrCl_2$-KCl Ion Exchange - Soln Prep

17 individual solns were prepared by adding various volumes of $SrCl_2$ soln (12% BW) (from 420/174) to bottles containing various amounts of potassium form zeolite (from 420/165). Target volumes (see Table 1 420/175) were done by weight. The bottle with zeolite was placed on a top load balance and the mass was recorded. Aliquots of the $SrCl_2$ soln were poured into a 1000mL polypropylene beaker. The appropriate amount of $SrCl_2$ soln was added to the bottle w/ zeolite from the 1000mL bottle. Initially the soln was poured from the 1000mL bottle until the appropriate amount was close. Final additions of the soln were done using a disposable plastic pipette. ~~This was d~~ BW 12/6/01 The final mass was recorded. The mass of $SrCl_2$ soln added was calculated.

Mettler PM4600 balance - challenge mass (target) = 400.00g
mass value at start of analysis - 400.00g
mass value at end of analysis - 400.00g

6DEC01
CONT-BAW

Table 1. Mass of $E_{Sr,I}$ solution added to 17 bottles containing potassium form zeolite (420/165)

Mixture # SrK-0.0005	$E_{Sr,I}$ to use	Volume (mL) of $E_{Sr,I}$ to use	Mass (g) of bottle and k-zeo	Mass (g) of Bottle, k-zeo, and $E_{Sr,I}$ solution	Mass (g) of $E_{Sr,I}$ solution*
1	1.0	25	6.13	31.14	25.01
2	1.0	50	9.62	59.64	50.02
3	1.0	50	9.59	59.59	50.00
4	1.0	100	16.76	116.80	100.04
5	1.0	100	16.71	116.74	100.03
6	1.0	100	16.78	116.79	100.01
7	1.0	250	27.94	278.02	250.08
8	1.0	250	27.95	277.97	250.02
9	1.0	250	27.91	277.96	250.05
10	1.0	250	28.13	278.12	249.99
11	1.0	500	51.65	551.67	500.02
12	1.0	500	50.87	550.87	500.00
13	1.0	500	50.87	550.87	500.00
14	1.0	1000	91.59	1091.6	1000.0
15	1.0	1000	90.53	1090.5	1000.0
16	1.0	1000	92.93	1092.9	1000.0
17	1.0	1000	90.53	1090.5	1000.0

*Calculated by subtracting (Mass of Bottle, k-zeo, and $E_{Sr,I}$ solution) from the (Mass of bottle and k-zeo)

Values for solns 12, 13, 15, + 17 are correct.

Solns were agitated by placing in shaker (New Brunswick model G-33) at ~ 150 rpm. Solns were hand swirled once a day (week days)

06DEC01
CONT BAW

Two aliquots (10mL) of the 7 liter $SrCl_2$ was saved for LSA analysis.

Soln: 7L of $SrCl_2$ · 420/174
Container: 20mL plastic LSA vials
Transfered with 10mL oxford pipet
Labeled Esr1 SrKL7a and Esr1 SrKL7b

12DEC01
BAW

LSA results of 7Liter $SrCl_2$ soln

The following pages contain the results of LSA strontium analysis for the stock strontium solution used to prepare the $SrCl_2$-KC ion exchange (420-176 above). Aliquot soln volume was 10mL

~~BAW~~ 12 Dec 01

12DEC01
CONT BAW

12/10/01 6:45:43 PM **QuantaSmart (TM) - 1.10** **Page # 1**
SNC Protocol **Serial# 405314**

Calibration Information
Software Version IC: 2.09
Software Version EC: 1.10
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 13.09 Date Processed: 12/10/01 6:45:42 PM
14C Chi Square: 18.39 Date Processed: 12/10/01 6:45:42 PM
3H E^2/B (0-18.6 keV and 1-18.6 keV): 267.92 Date Processed: 12/10/01 6:45:42 PM
14C E^2/B (0-156 keV and 1-156 keV): 518.10 Date Processed: 12/10/01 6:45:42 PM
3H Efficiency (0-18.6 keV): 65.52 Date Processed: 12/10/01 6:45:42 PM
14C Efficiency (0-156 keV): 96.42 Date Processed: 12/10/01 6:45:42 PM
IPA Background Date Processed: 12/10/01 6:45:42 PM
3H Background CPM (0-18.6 keV): 15.98 Date Processed: 12/10/01 6:45:42 PM
14C Background CPM (0-156 keV): 22.90 Date Processed: 12/10/01 6:45:42 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100
========== Errors and Warnings ==========
========== End of Errors and Warnings ==========

8 vials cycled through for 6 analysis

1= #51A Blank (nanopure water)
2= #51A 1
3= #51A 2
4= #51A 3
5= #51A 4
6= #51A 5

369/40
~~369/40~~
BW 2-15-02

7= skipped
8= Esr1.0 SrKL7a
9= Esr1.0 SrKL7b

420/176
~~420/176~~
BW 2-15-02

12DEC01
CONT BAW

12/11/01 9:49:00 AM QuantaSmart (TM) - 1.10 Page # 1
Protocol# 17 - Sr90Cerenkov.lsa Serial# 405314 User: Bertetti

Assay Definition-

Assay Description:
Cerenkov counting of Sr90/Y90

Assay Type: CPM
Report Name: Sr90 Cerenkov
Output Data Path: C:\Packard\Tricarb\Results\Bertetti\Sr90Cerenkov
Raw Results Path: C:\Packard\Tricarb\Results\Bertetti\Sr90Cerenkov
Comma-Delimited File Name: C:\Packard\Tricarb\Results\Bertetti\Sr90Cerenkov\Sr90Cerenkov.001

Count Conditions-

Nuclide: Sr90 Cerenkov
Quench Indicator: SIS
External Std Terminator (sec): n/a
Pre-Count Delay (min): 0.00
Quench Set: n/a
Count Time (min): 60.00
Count Mode: Normal
Assay Count Cycles: 6 Repeat Sample Count: 1
#Vials/Sample: 1 Calculate % Reference: Off

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

Regions	LL	UL	Bkg Subtract	2Sigma % Terminator
A	0.0	30.0	1st Vial	1.00
B	0.0	100.0	1st Vial	0.00
C	0.0	2000.0	1st Vial	0.00

Count Corrections-

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

Half Life-

Half Life Correction: Off

Regions	Half Life	Units	Reference Date	Reference Time
A				
B				
C				

IPA Block Data
Software Version IC: 2.09
Software Version EC: 1.10
Instrument Model: Tri-Carb 3100TR
Instrument Serial Number: 405314
3H Chi Square: 13.09 Date Processed: 12/10/01 6:45:42 PM
14C Chi Square: 18.39 Date Processed: 12/10/01 6:45:42 PM
3H E^2/B (0-18.6 keV and 1-18.6 keV): 267.92 Date Processed: 12/10/01 6:45:42 PM
14C E^2/B (0-156 keV and 1-156 keV): 518.10 Date Processed: 12/10/01 6:45:42 PM
3H Efficiency (0-18.6 keV): 65.52 Date Processed: 12/10/01 6:45:42 PM
14C Efficiency (0-156 keV): 96.42 Date Processed: 12/10/01 6:45:42 PM
IPA Background Date Processed: 12/10/01 6:45:42 PM
3H Background CPM (0-18.6 keV): 15.98 Date Processed: 12/10/01 6:45:42 PM
14C Background CPM (0-156 keV): 22.90 Date Processed: 12/10/01 6:45:42 PM
3H Calibration DPM: 285000
3H Reference Date: 10/29/99
14C Calibration DPM: 134100
========== Errors and Warnings ==========
========== End of Errors and Warnings ==========

12DEC01
CONT BAW

12/11/01 9:49:01 AM QuantaSmart (TM) - 1.10 Page # 2
Protocol# 17 - Sr90Cerenkov.lsa Serial# 405314 User: Bertetti

Cycle 1 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	14	B	6.9	22	5.5	23	90.6
2	0.51	79609		1.0	79858	1.0	79858	28.8
3	1.02	39464		1.0	39576	1.0	39575	28.7
4	2.50	16008		1.0	16055	1.0	16055	28.8
5	5.40	7399		1.0	7423	1.0	7424	28.5
6	27.39	1447		1.0	1451	1.0	1451	28.2
Missing vial 7.								
8	24.40	1626		1.0	1628	1.0	1628	26.2
9	23.71	1673		1.0	1678	1.0	1678	28.5

Cycle 2 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	15	B	6.7	23	5.4	24	90.1
2	0.51	79105		1.0	79342	1.0	79343	28.7
3	1.03	38991		1.0	39093	1.0	39094	28.6
4	2.53	15815		1.0	15865	1.0	15866	28.9
5	5.53	7227		1.0	7247	1.0	7247	28.7
6	27.44	1443		1.0	1447	1.0	1447	28.4
Missing vial 7.								
8	24.72	1604		1.0	1607	1.0	1606	26.1
9	23.70	1673		1.0	1677	1.0	1677	28.5

Cycle 3 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	15	B	6.7	24	5.3	24	89.8
2	0.51	79846		1.0	80127	1.0	80127	28.9
3	1.02	39528		1.0	39626	1.0	39629	28.4
4	2.52	15880		1.0	15925	1.0	15925	28.8
5	5.51	7256		1.0	7274	1.0	7275	28.6
6	27.46	1442		1.0	1446	1.0	1446	28.3
Missing vial 7.								
8	24.51	1618		1.0	1619	1.0	1619	26.0
9	23.46	1691		1.0	1696	1.0	1695	28.4

Cycle 4 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	15	B	6.8	23	5.4	23	88.6
2	0.51	79405		1.0	79630	1.0	79630	28.8
3	1.03	39127		1.0	39218	1.0	39218	28.6
4	2.55	15743		1.0	15796	1.0	15796	28.8
5	5.49	7283		1.0	7305	1.0	7305	28.7
6	27.37	1447		1.0	1452	1.0	1452	28.4
Missing vial 7.								
8	24.86	1595		1.0	1597	1.0	1596	26.3
9	23.77	1669		1.0	1674	1.0	1674	28.6

Cycle 5 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	14	B	6.8	23	5.4	23	89.1
2	0.51	79262		1.0	79491	1.0	79491	28.7
3	1.02	39348		1.0	39480	1.0	39480	28.4
4	2.53	15860		1.0	15909	1.0	15909	28.8
5	5.52	7236		1.0	7258	1.0	7258	28.5
6	27.41	1445		1.0	1450	1.0	1450	28.3
Missing vial 7.								
8	24.52	1617		1.0	1620	1.0	1620	26.1
9	24.06	1649		1.0	1654	1.0	1654	28.6

Cycle 6 Results

S#	Count Time	CPMA	MESSAGES	A:2S%	CPMB	B:2S%	CPMC	SIS
1	60.00	14	B	6.9	23	5.4	23	91.3

12DEC01

CONT BAW

12/11/01 9:49:01 AM QuantaSmart (TM) - 1.10 Page # 3
Protocol# 17 - Sr90Cerenkov.lsa Serial# 405314 User: Bertetti

2	0.51	79886	1.0	80142	1.0	80144	28.7
3	1.03	39229	1.0	39328	1.0	39327	28.6
4	2.52	15935	1.0	15995	1.0	15995	28.7
5	5.46	7320	1.0	7346	1.0	7346	28.7
6	27.08	1463	1.0	1468	1.0	1468	28.3
Missing vial 7.							
8	24.50	1619	1.0	1619	1.0	1619	26.0
9	23.77	1669	1.0	1674	1.0	1674	28.7

Solns 8 and 9 were 10mL aliquots of the same soln, Vial 9 results were consistantly higher than Vial 8. The solns were examined after the analysis and the volume of soln in vial 8 was noticably larger than the volume of soln in vial 9. This could explain the results.

14 12DEC01 BAW 12/14/01

Preparation of Ternary CsCl-NaCl-KCl Stock Solutions (0.05N)

Prepared six 1000mL solutions with a total normality of 0.05 N. See table 1 for the target masses of CsCl, NaCl, and KCl required for each solution. Table 2 contains the actual experimental values. The mass of the weighing boat or weighing paper was recorded. The appropriate amount of the compound was added and the mass was recorded. The compound was transferred to a 400mL beaker. Nanopure water from a squirt bottle was used to rinse the weighing boat or paper several times. The washings were carefully transferred to the beaker. This step was repeated until all of the appropriate compounds were added to the beaker. The solution was left standing for about a minute and then decanted into a 1000mL volumetric flask. The beaker was washed with nanopure water several times, carefully transferring all washings into the volumetric flask. The volumetric flask was filled to about 2-3 inches below the mark with nanopure water and swirled for mixing. Then nanopure water was added dropwise up to the mark and remixed. Each solution was transferred to a 1000 mL polypropylene bottle and labeled appropriately.

Cesium Chloride: Fisher BP210-500, lot# 010406, rec. 5/14/01
Sodium Chloride: Fisher S271-3, lot# 986412
Potassium Chloride: Fisher P217-500, lot# 006242
Nanopure water

14 12DEC01 BAW CONT BAW 14/Dec/01

Mettler electronic balance AE240
challange mass target = 20.0001g
measurement at start of analysis = 19.9999
measurement at end of analysis = 19.9999

Table 1. Target values for stock solutions for ternary ion exchange experiment

Solution ID	ECs	ENa	EK	Mass (g) of CsCl for 1 L	Mass (g) of NaCl for 1 L	Mass (g) of KCl for 1 L	Calc. initial Cs ppm	Calc. initial Na ppm	Calc. initial K ppm
CsCl-NaCl-KCl*0.05N*1.0ECs	1	0	0	8.4180	0.0000	0.0000	6645	0	0
CsCl-NaCl-KCl*0.05N*0.7ECs	0.7	0.3	0	5.8926	0.8766	0.0000	4652	345	0
CsCl-NaCl-KCl*0.05N*0.3ECs	0.3	0.7	0	2.5254	2.0454	0.0000	1994	805	0
CsCl-NaCl-KCl*0.05N*0.1ECs	0.1	0.9	0	0.8418	2.6298	0.0000	665	1035	0
CsCl-NaCl-KCl*0.05N*0.05ECs	0.05	0.95	0	0.4209	2.7759	0.0000	332	1092	0
CsCl-NaCl-KCl*0.05N*0.2ECs	0.2	0.4	0.4	1.6836	1.1688	1.4910	1329	460	782

14 12DEC01 BAW 14DEC01

14DEC01
CONT BLW

Table 2. Preparation of stock solutions for ternary ion exchange experiment

Solution ID	Compound	Mass (g) of boat	Mass (g) of boat + compound	Mass (g) of compound
CsCl-NaCl-KCl*0.05N*1.0ECs	CsCl	1.5613	9.9793	8.4180
	NaCl	na	na	na
	KCl	na	na	na
CsCl-NaCl-KCl*0.05N*0.7ECs	CsCl	1.5806	7.4730	5.8924
	NaCl	0.5083	1.3845	0.8762
	KCl	na	na	na
CsCl-NaCl-KCl*0.05N*0.3ECs	CsCl	0.5077	3.0333	2.5256
	NaCl	0.5426	2.5881	2.0455
	KCl	na	na	na
CsCl-NaCl-KCl*0.05N*0.10ECs	CsCl	0.5129	1.3551	0.8422
	NaCl	1.6139	4.2436	2.6297
	KCl	na	na	na
CsCl-NaCl-KCl*0.05N*0.05ECs	CsCl	0.5075	0.9286	0.4211
	NaCl	1.5829	4.3589	2.7760
	KCl	na	na	na
CsCl-NaCl-KCl*0.05N*0.2ECs	CsCl	0.4932	2.1772	1.6840
	NaCl	0.5059	1.6745	1.1686
	KCl	0.4913	1.9821	1.4908

14DEC01 BLW

17DEC01
BAW

Preparation of CsCl-NaCl-KCl Solutions (0.05N) – Addition of zeolite

Table 3 provides a breakdown of the 18 solutions to be prepared. It contains the target amounts of zeolite and identity of the reference solution used to make the mixture. It also contains initial target concentrations of Cs, Na, and K. The potassium form of the zeolite (k-zeo) will be used. A piece of weighing paper was folded into quarter sections and the mass was recorded (see Table 4). The appropriate amount of k-zeo was placed on the weighing paper and the mass was recorded. The k-zeo is then carefully poured into an appropriately sized polypropylene bottle. A policeman (glass rod with rubber tip) was used to tap the outside of the paper or scrub the inside of the paper to completely transfer the solid from the paper to the bottle.

The appropriate amount (volumetric pipet) of the appropriate reference solution (see Table 3) was added to the bottle containing the k-zeo. Pipets were not directly dipped into the 1000mL reference solution. Aliquots of the reference solution were decanted into 100mL beakers. Excess reference solution from the 100mL beakers was discarded.

2/15/02 BLW Potassium ~~Potassium~~ form of zeolite:
CDV*200/325*UC*WA*RC*HL*RFe*KF
420/54-67 AJ 5/2/01

Table 3. Target values for stock solutions for ternary ion exchange experiment solutions

Mixture #	Reference Solution	K zeol mass (g) zeol.	exp soln vol (mL)	Calc. Cs ppm i	Calc. Na ppm i	Calc. K ppm i
1	CsCl-NaCl-KCl*0.05N*1.0ECs	0.1457	10	6645	0	0
2	"	0.2014	10	6645	0	0
3	"	0.1500	25	6645	0	0
4	"	0.2000	25	6645	0	0
5	CsCl-NaCl-KCl*0.05N*0.7ECs	0.1617	10	4652	345	0
6	"	0.2218	10	4652	345	0
7	"	0.3158	10	4652	345	0
8	CsCl-NaCl-KCl*0.05N*0.3ECs	0.1538	10	1994	805	0
9	"	0.3105	10	1994	805	0
10	CsCl-NaCl-KCl*0.05N*0.1ECs	0.1000	10	665	1035	0
11	"	0.2000	10	665	1035	0
12	"	0.3000	10	665	1035	0
13	CsCl-NaCl-KCl*0.05N*0.05ECs	0.1000	10	332	1092	0
14	"	0.2000	10	332	1092	0
15	"	0.3000	10	332	1092	0
16	CsCl-NaCl-KCl*0.05N*0.2ECs	0.1000	10	1329	460	782
17	"	0.2000	10	1329	460	782
18	"	0.3000	10	1329	460	782

Reference solutions: See '420/182

17 DEC01 CONT

BAW Mettler AE240 balance

Challange mass = 20.0001 g

Mass at start of analysis = 19.9999 g

Mass at end of analysis = 19.9999 g

Table 4. Preparation of ternary ion exchange experiment solutions

Mixture #	Mass (g) of paper	Mass (g) of paper + zeolite	Mass (g) of zeolite
1	0.2409	0.3865	0.1456
2	0.2355	0.4369	0.2014
3	0.2455	0.3954	0.1499
4	0.2556	0.4557	0.2001
5	0.2545	0.4161	0.1616
6	0.2453	0.4670	0.2217
7	0.2488	0.5646	0.3158
8	0.2464	0.4001	0.1537
9	0.2395	0.5499	0.3104
10	~~0.2406~~ BAW 12-15-01	0.3406	0.1000
11	0.2405	0.4403	0.1998
12	0.2441	0.5442	0.3001
13	0.2518	0.3517	0.0999
14	0.2552	0.4551	0.1999
15	0.2524	0.5523	0.2999
16	0.2530	0.3531	0.1001
17	0.2491	0.4490	0.1999
18	0.2464	0.5463	0.2999

17DEC01

CONT BAW Placed 18 solutions in gyratory shaker at ~ 200rpm.

12/18/01 AJ Reanalyze final Ca^{+2} ion conc. for 0.0005N KCl/$CaCl_2$ ion exchange experiments to test the reproducibility of measured values.

Ca^{+2} std. solutions required :-

100 ppm or 0.0025 M : $\frac{0.1M \times 2.5ml}{100ml}$

200 ml of 100 ppm Ca^{+2} std. was prepared and all dilutions were made from 100 ppm std. as follows:

40 ppm : $\frac{100\ ppm \times 40ml}{100\ ml}$

30 ppm : $\frac{100\ ppm \times 30\ ml}{100\ ml}$

20 ppm : $\frac{100\ ppm \times 20\ ml}{100\ ml}$

25 ppm : $\frac{100\ ppm \times 25\ ml}{100\ ml}$

15 ppm : $\frac{100\ ppm \times 15\ ml}{100\ ml}$

10 ppm : $\frac{100\ ppm \times 10\ ml}{100\ ml}$

Electrode was calibrated using 100 ml solutions.

Mixture # CaK-0005*	ECa,i	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc.Ca ppm,i	Calc. Ca ppm,f	12/18/01 Calc. Ca ppm,f
1	1	0.1507	25	10	0.16	
2	1	0.1473	50	10	0.39	
3	1	0.1162	50	10	0.52	
4	1	0.1602	100	10	0.85	9.7
5	1	0.10428	100	10	1.49	12.9
6	1	0.2039	250	10	2.02	14.3
7	1	0.1447	250	10	2.92	17.2
8	1	0.1158	250	10	3.58	19.6
9	1	0.166	500	10	4.59	21.0
10	1	0.1325	500	10	5.25	22.8
11	1	0.1874	1000	10	6.19	24.2
12	1	0.1478	1000	10	6.76	25.6
13	1	0.1024	1000	10	7.51	26.8
14	1	0.1591	2000	10	7.94	26.5
15	1	0.1075	2000	10	8.48	26.8
16	1	0.0819	2000	10	8.78	25.8

12/19/01
AJ Reanalyze K^+ ion conc. (final) for 0.0005N CaCl/KCl ion exchange experiments to test the reproducibility of the measured values.

0.01 M K^+ std. solution :- from 0.1 M K^+ std. 200ml of 0.01 M K^+ std. was prepared. All other dilutions will be made

using 0.01 M K^+ solution.

K^+ std. solutions required :

0.00005M or 1.96 ppm : $\frac{0.01M \times 0.5ml}{100\ ml}$

0.0001 M or 3.91 ppm : $\frac{0.01M \times 1ml}{100\ ml}$

0.0002M or 7.82 ppm : $\frac{0.01 \times 2ml}{100\ ml}$

0.0004M or 15.64 ppm : $\frac{0.01M \times 4ml}{100\ ml}$

0.0006M or 23.46 ppm : $\frac{0.01M \times 6ml}{100\ ml}$

0.001 M or 39.1 ppm : $\frac{0.01M \times 10ml}{100\ ml}$

ISA : 2ml / 100 ml

Note: Ion selective electrode for K^+ inactive due to damage from fall.

12/19/01
AJ

12/19/01 AJ

Preparation of $CaCl_2$-KCl (0.01N) Solutions for ion-exchange experiments

1. Prepare $CaCl_2$-KCl aqueous mixtures with a total normality of 0.01 N and a fixed Ca/K ratio by taring reagent grade KCl and $CaCl_2 \bullet 2H_2O$ in the amounts given in Table 1. In preparing the solutions, make sure that 10-mL samples will be available for analysis of initial Ca2+and K+ concentration.

Transfer the solution into clean polypropylene bottles of the appropriate size. Label the bottle (e.g., $CaCl_2$/KCl *0.01N*0.1E_{Ca}, plus Date and Initial, and lab notebook volume & page#).

$E_{i,Ca}$ (0.01N) $CaCl_2$/KCl soln.	wt. of $CaCl_2 \cdot 2H_2O$ needed for 500 ml	wt. % KCl needed for 500 ml
0.3	0.1103	0.2610
0.6	0.2205	0.1491

Wt. $CaCl_2.2H_2O$ used	Wt. KCl used
0.1106	0.2613
0.2206	0.1495

KCl – FS P217-500, lot # 006242

$CaCl_2 \cdot 2H_2O$ – FS C79-500, lot # 016231, Rcvd 10/15/01

12/21/01 AJ	$E_{i,Ca}$ (0.01N) $CaCl_2$/KCl soln.	wt. of $CaCl_2 \cdot 2H_2O$ needed for ~~2000~~ AJ 2/15/02 2000 ml	wt. of KCl needed	wt. of $CaCl_2 \cdot 2H_2O$ used
	1.0	01.4702	0	~~1.470~~ AJ 1.4708 12/4/01
	1.0	1.4702	0	1.4707

Total of 4L of $E_{i,Ca}$ (0.01N) $CaCl_2$/KCl soln. was prepared * 1.0E_{Ca}

EXPERIMENT CAKCL-.01N

Mixture # CaK-01*	$E_{Ca,i}$ to use	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc. Ca ppm,i	Calc. Ca ppm,f	Calc. K ppm,i	Calc. K ppm,f
1	0.3	0.2498	10	60.1	19.27	273.7	353.38
2	0.3	0.1586	25	60.1	39.38	273.7	314.16
3	0.6	0.1248	10	120.2	59.01	156.4	275.85
4	0.6	0.1637	25	120.2	77.42	156.4	239.94
5	1	0.1299	10	200.4	94.22	0.0	207.15
6	1	0.1981	25	200.4	116.19	0.0	164.29
7	1	0.1261	25	200.4	134.44	0.0	128.69
8	1	0.1643	50	200.4.	149.36	0.0	99.57
9	1	0.0943	50	200.4	164.93	0.0	69.19
10	1	0.1082	100	200.4	176.52	0.0	46.59
11	1	0.1519	250	200.4	185.00	0.0	30.05
12	1	0.1630	500	200.4	191.07	0.0	18.20
13	1	0.1604	1000	200.4	195.28	0.0	9.98
14	1	0.1001	1500	200.4	198.05	0.0	4.58

0.2500
0.1590
0.125
0.1640
0.~~1304~~ 1304 AJ 2/15/02
0.~~1985~~ 1985 AJ 2/15/02
0.1264
0.1646
0.0947
0.1082
0.1522
0.1633
0.1608
0.1005

zeolite used :- COV * 200/325 * UC * WA * RC * HL * RFe * Kf prepared by AJ on 5/2/01 (420/54-67)

Place these solns. on New Brunswick (model G-33) shaker at ~ 100 rpm. Swirl the solutions by hand everyday.

1/8/02 BAW — Preparation of ternary (Cs-Na-K) ion exchange experimental and stock solutions for ICP analysis by Division 01

The 18 experimental solutions (420/183-184) will be diluted with nanopure water before transfer to Div 01.
The 6 stock solutions (420/180-182) will not be diluted before transfer to Div 01.
No acid will be used for perserving the samples. The estimated pH of the solutions is ~ 5 to 6. Two duplicates (two aliquots of same original solution) will be done. One duplicate will be from an experimented solution and the other duplicate will be from a stock solution. The samples will be labeled as followed:

Reference Solution	Label for Div 01 Sample
CsCl-NaCl-KCl * 0.05N * 1.0 ECs	R1.0
" 0.7 ECs	R0.7
" 0.3 ECs	R0.3
" 0.1 ECs	R0.1
" 0.05 ECs	R0.05
" 0.2 ECs	R0.2A
" 0.2 ECs duplicate	R0.2B

Mixture #1-18 will be labeled M1 to M18. A duplicate of Mixture 18 BAW 1/8/02 will be taken. They will be labeled ~~M18A and M18B~~ BAW 1/8/02 M4A and M4B

1/8/02
CONT BAW Added 5 mL (volumetric pipet) of experimental solution to a 100 mL volumetric ~~BAW~~ 1/8/02 flask and diluted to mark with nanopure water. A 1:20 dilution of the 18 experimental solutions (420/183-184) and one duplicate (~~M18~~ BAW 1/8/02 M4 BAW 1/8/02 DUP) were made this way. About 50 mL of each diluted solution was decanted into a 60 polypropylene bottle. The remaining 50 mL was also decanted into another 60 polypropylene bottle. One bottle was transfered to Div 01 for ICP analysis. The other bottle was saved at Div 20.

The six stock solutions (420/180-182) were not diluted. Aliquots were decanted into appropriately labeled 60 mL polypropylene bottles and transfered to Div 01.

1/9/02 BAW — pH measurement of one ternary reference sample

Soln = CsCl-NaCl-KCl * 0.05N * 0.3 ECs (420/182)
Orion model 920A pH meter, serial ~~#~~ 039522
Orion model 8103 Ross combination electrode (3C) w/ ATC probe
Calibrated with
pH 4 buffer soln (1-8-02) Fisher SB98-500 lot# 003509-24
pH 7 buffer soln (1-8-02) Fisher SB108-500 lot# 012717-24
Cal temp @ 21.7°C, cal set pts @ 4.00 and 7.02
Slope of cal curve = 99.7%
Measurement of soln = 6.18 pH

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Client Name/Address: Bradley Werling
CNWRA - DIV 20
Bld 57

SAMPLE LIST/CHAIN OF CUSTODY

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

Requested Turnaround:
☐ 1 Week
☐ 2 Weeks (Normal)
☐ 3 Weeks
☑ Other: 4 weeks

Client Purchase Order/Other ID | Site/Zone ID

Analysis

Analyses Requested

SwRI Contact: Mike Dammann

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP Analysis for 3 cations (Cs, Na, K)
M4A	1-8-02		W	DM	1	X
M4B	"		"	"	"	X
M5	"		"	"	"	X
M6	"		"	"	"	X
M7	"		"	"	"	X
M8	"		"	"	"	X
M9	"		"	"	"	X
M10	"		"	"	"	X
M11	"		"	"	"	X
M12	"		"	"	"	X

REMARKS
No preservation
Preservation pH ~ 5-6.5
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

Project is nuclear safety related - 10 CFR Part 21 - Append B

POC for questions is Bradley Werling phone 6565 fax 5184

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by (Signature):

Received by (Signature)

SwRI Project No. 20.R9211.01.001

Received by SwRI Lab (Signature): Marie Pulsinelli

Relinquished by Sampler (Signature): Bradley Werling

Relinquished by (Signature)

Samples Disposed by:

Received by (Signature):

Comments: 420/183+190

Date/Time: 1-9-02 / 1452

Div 01 COC Form 01-01-001, Rev 1/97

Page 1 of 3

1-9-02 CONT BMW

1-9-02 CONT BMW Transfer of Custody Paperwork for Samples Delivered to DIV 01 for Analysis (ternary from 420/190+191)

Client Name/Address: Bradley Werling
CNWRA - DIV 20
Bld 57

SAMPLE LIST/CHAIN OF CUSTODY

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

Requested Turnaround:
☐ 1 Week
☐ 2 Weeks (Normal)
☐ 3 Weeks
☑ Other: 4 weeks

Client Purchase Order/Other ID | Site/Zone ID

Analysis

Analyses Requested

SwRI Contact: Mike Dammann

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP Analysis for 3 cations (Cs, Na, K)
R1.0	1-8-02		W	DM	1	X
R0.7	"		"	"	"	X
R0.3	"		"	"	"	X
R0.1	"		"	"	"	X
R0.05	"		"	"	"	X
R0.2A	"		"	"	"	X
R0.2B	"		"	"	"	X
M1	"		"	"	"	X
M2	"		"	"	"	X
M3	"		"	"	"	X

REMARKS
No preservation
Preservation pH ~ 5-6.5
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

Project is nuclear safety related - 10 CFR Part 21 - Append B

POC for questions is Bradley Werling phone 6565 fax 5184

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by (Signature):

Received by (Signature)

SwRI Project No. 20.R9211.01.001

Received by SwRI Lab (Signature): Marie Pulsinelli

Relinquished by Sampler (Signature): Bradley Werling

Relinquished by (Signature)

Samples Disposed by:

Received by (Signature):

Comments: 420/183+190

Date/Time: 1-9-02 / 1452

Div 01 COC Form 01-01-001, Rev 1/97

Page 1 of 3

1-9-02 CONT BMW

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1-09-02

CONT BWS

Client Name/Address: Bradley Wentling c/o WRA - DIV 10 Bld 57

Client Purchase Order/Other ID:

Site/Zone ID:

SAMPLE LIST/CHAIN OF CUSTODY

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

Analyses Requested

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP Analysis/ICP MS Analysis of 3 cations Cs, Na, K
M13	1-8-02		W	DM	1	X
M14	1		1	1	1	X
M15	1		1	1	1	X
M16	1		1	1	1	X
M17	1		1	1	1	X
M18	1		1	1	1	X

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): Bradley Wentling

Received by (Signature):

Relinquished by (Signature):

Received by (Signature)

Relinquished by (Signature)

Received by (Signature)

Comments: 420/183+190

Requested Turnaround:
- ☐ 1 Week
- ☐ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☑ Other: 4 weeks

SwRI Contact: Mike Dapkewicz

REMARKS
No preservation
Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

Project is nuclear safety related - 10 CFR Part 21 App B

POC for questions is Bradley Wentling phone 6565 fax 5184

SwRI Project No. 20.R9211.01.001

Received by SwRI Lab (Signature):

Samples Disposed by:

Date/Time: 1-9-02 4:45 pm

Div 01 COC Form 01-01-001, Rev 1/97

1-10-02

BAW

Correction on Sr ppm calculation in Table 1 (420/164) for $SrCl_2/KCl$ ~ 0.0005 N

The initial Sr ppm should be 43.8 ppm. The final Sr and K ppm need to be recalculated.

Based on info from 420/161,

$SrCl_2 \cdot 6H_2O$ = 266.618 g/mol

0.93317 g of $SrCl_2 \cdot 6H_2O$ into 7000 mL

Sr = 87.62 g/mol ppm = mg/mL

$$0.93321\,g\ SrCl_2 \cdot 6H_2O \left(\frac{87.62\,g\ Sr}{266.618\,g\ SrCl_2 \cdot 6H_2O}\right)\left(\frac{10^6\,\mu g}{J}\right)$$

$$= 43.8\ \frac{\mu g}{mL}$$

7000 mL

or 43.8 ppm Sr

The Normality w/r/t Sr is correct at 0.0005 N

For $SrCl_2 \cdot 6H_2O$ $XN_{Sr} = XM_{Sr}$ M = moles/Liter

$$0.93321\,g\ SrCl_2 \cdot 6H_2O \left(\frac{87.62\,g\ Sr}{266.618\,g\ SrCl_2 \cdot 6H_2O}\right)\left(\frac{1\,mol\ Sr}{87.62\,g\ Sr}\right)$$

$$= 5\text{+}10^{-4}\ M$$

7L

or 0.0005 M Sr = 0.0005 N Sr

1/9/02 Continue 0.01N $CaCl_2$/KCl ion-exchange expts
AJ

Required Ca^{+2} solutions:

100 ppm or 0.0025 M : $\frac{0.1\,M \times 2.5\,ml}{100\,ml}$

All other dilutions were made from 100 ppm Ca^{+2}
10 ppm, 20 ppm, 15 ppm, 2 ppm & 5 ppm.

200 ppm or 0.005 M : $\frac{0.1\,M \times 5\,ml}{100\,ml}$

150 ppm was prepared using 200 ppm Ca^{+2} soln.

150 ppm : $\frac{200\,ppm \times 75\,ml}{100\,ml}$

20 ppm : $\frac{100\,ppm \times 20\,ml}{100\,ml}$

15 ppm : $\frac{100\,ppm \times 15\,ml}{100\,ml}$

10 ppm : $\frac{100\,ppm \times 10\,ml}{100\,ml}$

5 ppm : $\frac{100\,ppm \times 5\,ml}{100\,ml}$

2 ppm : $\frac{100\,ppm \times 2\,ml}{100\,ml}$

Mixture # CaK-01*	ECa,I to use	Weight K-zeol. to use (gm)	Volume soln. to use (ml)	Calc. Ca ppm,f	1st trial 1/9/02 Measured Ca ppm, f	2nd trial 1/9/02 Measured Ca ppm, f
1	0.3	0.2498	10	19.27	23.4	23.1
2	0.3	0.1586	25	39.38	46.5	42.4
3	0.6	0.1248	10	59.01	75.5	71.4
4	0.6	0.1637	25	77.42	88.1	84.1
5	1	0.1299	10	94.22	108	117
6	1	0.1981	25	116.19	130	134
7	1	0.1261	25	134.44	144	144
8	1	0.1643	50	149.36	151	156
9	1	0.0943	50	164.93	165	165
10	1	0.1082	100	176.52	165	177
11	1	0.1519	250	185	173	184
12	1	0.163	500	191.07	181	186
13	1	0.1604	1000	195.28	184	190
14	1	0.1001	1500	198.05	193	

were

dilute to 1/10

1 ml was diluted to 10 ml

1-11-02 Preparation of 10% $LaCl_3$ soln for Aa stock solution.
BAW

Reagents: nanopure water
$LaCl_3 \cdot 7H_2O$ - Fisher L4-250, Lot # 985153A, fw of 371.38 g/mol (La at 138.91 g/mole)

1-11-02
CONT BAW Tared a 250 pp bottle on the Mettler PM 4600 balance. Added 100.00 g of nanopure water & tared again. Added 36.50 g of $LaCl_3 \cdot 7H_2O$ Mixed thoroughly.

Preparation of 1% LaCl Soln

1:10 Dilution of 10% LaCl

Added 50 mL (vol. pipette) of 10% LaCl (420/197) to a 500 mL vol. flask and diluted to mark with nanopure water.

Preparation of Strontium Stock soln for AA Analysis

Labeled Sr Stock
Target 25 ppm Sr
Reagents: nanopure water
Sr 1000 ppm SpexCertiprep PLSR2-2Y, lot 8-1345R, rec 1/4/02, open 1/11/02

Added 5 mL of 1000 ppm Sr (Spex Std) via volumetric pipette to a 200 mL Vol flask and diluted to mark with nanopure water.

BW 4/12/02

1-11-02
CONT BAW

Preparation of Strontium Cal Curve for AA

Volumetric pipettes used for transfers into volumetric flasks. Diluted to mark with nanopure water. All final Vols 50 mL

25 ppm Sr was Sr Stock (420/198)

10% LaCL was 10% LaCl (420/197+8)

Strontium calibration curve

Soln ID	Target Conc of Sr (ppm)	Target Conc of LaCl (%)	Vol (mL) of 25 ppm Sr	Vol (mL) of 10% LaCl
Sr1*	5	1	10	5
Sr2	2.5	1	5	5
Sr3	1.5	1	3	5
Sr4	1	1	2	5
Sr5	0.5	1	1	5
Sr6	0.25	1	0.5	5

* AA sensitivity check

1-14-02
BAW

AA Analysis of Strontium in Stock solution For $SrCl_2$-LaCl (0.0005N):

7L Strontium solution (420/161+162) before spiked with Sr^{90} radioactive spike. Target conc = 43.8 ppm Sr

Dilution Factor 20 (DF20) for 2.19 ppm target conc. Added 5 mL of stock solution (420/161) and 5 mL (corked pipette)* to a 100 mL volumetric flask and diluted to mark with nanopure water. * of 10% LaCl (420/197)

Page Extention 1/14/02

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- End of Ntbk -

Continued in 494

BW

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

E.C. Seney 3/12/2002