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4/21/98
AJ

1

Uranium Sorption on Clinoptilolite Sorption (C20) Experiments

Written By: Alka Jain
Revision No.: 0

Date Written: April 21, 1998
Date Revised: N/A

CONDITIONS:

1. U = 500 ppb
2. 0.1 M NaNO₃ matrix, equilibrium with atmosphere CO₂ (g). pCO₂ = 10^{-3.5}
3. pH range 2-9
4. Initial solutions volume = 40 ml, initial clinoptilolite mass = 0.1 g

OBJECTIVES:

- To investigate the characteristics of U absorption on clinoptilolite as a function of solution pH.
- To investigate the reproducibility and reversibility of U sorption reactions.
- To investigate the effects of using polycarbonate experimental containers in U sorption experiments.

EQUIPMENT:

Gyratory Shaker
Packard liquid scintillation counter
Orion pH/mV/ISE/ C meter
Combination pH electrode
ATC probe
Analytical balances (Mettler 4600 and 240 AE)
survey instruments

SUPPLIES:

- | | |
|---------|--|
| 29 | 2 oz polycarbonate bottles (acid washed and dried) |
| 1 | Repipettor for transfer of scintillation cocktail |
| Several | Eppendorf micropipettors for solution transfer |
| | Eppendorf pipet disposable tips |
| | pH buffer solutions |
| | Ultima-Gold liquid scintillation cocktail |

PROCEDURE:

Special considerations:

- Experimental solutions and sample containers/vials should be weighed at each step. Do not add or subtract contents without weighing before and after each process. Always record

weight of solutions.

- When measuring pH, minimize the amount of time the glass electrode is in contact with the U bearing solution. Make sure to rinse the electrode thoroughly before measuring other solution. Take care to introduce lint particles or other foreign objects into the experimental or sample containers.
 - Liquid scintillation analysis (LSA) should be performed within two days of each sampling interval so that results can be reviewed during the experiment. Sample vials should "rest" in the absence of light for at least 24 hours prior to initial analysis to allow for decay of incident radiation pulses.
 - Following each sampling period, swipes and frisks of the work area should be performed. If contamination is found, follow the radiological procedures for clean-up, and inform the division radiation safety point of contact (RSPOC). Radioactive solutions may not be disposed of without following all radiation safety guidelines. Do not dispose of any solutions without prior approval of the division RSPOC.
1. a) Add 500 ppb U solutions to each experiment container.
b) Label twenty-nine 2 oz. Polycarbonate containers C20- pH_i (where i is the approximate pH of each solution) and pre-weigh. Record weight.
c) Add 40 g of the 500 ppb U solution to each container. Record weight.
d) Label ten 2 oz. Polycarbonate containers C20-C* pH_i (where i is the approximate pH of each solution) and pre-weigh. Record weight.
e) Add 40 g of the 500 ppb U solution to each container. Record weight.
f) Transfer the remaining (~ 40 g) 500 ppb U solution into a pre-weighed 2 oz. Polycarbonate container labeled C20-IU. Add 100 micro liters of 50% HNO₃ solution to the bottle and mix thoroughly. Record weight.
Note: If pH adjustment is to be made at a later time (i.e. few hours later), cap the bottles tightly.
 2. For C20-pH_i C20-C* pH_i solutions, add HNO₃ and NaHCO₃ to adjust pH to desired value.
 - a) Adjust pH by adding HNO₃ and NaHCO₃ using an Eppendorf micropipet. Record the amount and concentration added to each container. Mix well by swirling the solutions.
 - b) Re-weigh each container. Record weight.
 - c) Replace screw caps, but do not tighten! Solutions must be open to atmosphere.
 - d) Place bottles on a gyratory shaker at 120 rpm. Allow at least 10 days for pH equilibration.
 3. Sample C20-IU to determine initial U concentration for experimental solutions.

- a) Using an eppendorf pipette withdraw two 0.5 ml samples from C20-IU and transfer to liquid scintillation vials. Re-weigh vials and record weight.
- b) Add 5 ml of Ultima-Gold cocktail to each vial. Homogenize sample and set aside for LSA.

April 22, 98 Preparation of Clinoptilolite for U-sorption Experiments
AJ

Materials & Equipments

- ① Clinoptilolite [Death Valley Junction, California]
- ② 8-inch diameter stainless steel sieves [35, 100, 200 mesh]
- ③ Ro-Tap Sieve Shaker
- ④ Ultrasonic Cleaner FS-28
- ⑤ Water Bath
- ⑥ 1N NaOAc Buffer at PH = 5.0 [136gms of $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ PH adjusted to 5.0 using (Lot No. 940621) per liter of Glacial acetic acid (Lot # 956018) nano pure water]
- ⑦ 0.3 M Sodium citrate dihydrate solution [88g of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (Lot # 940621) per liter of NH_2O Fischer Scientific]
- ⑧ Sodium Dithionite, $\text{Na}_2\text{S}_2\text{O}_4$ [Lot # FS 916643]
- ⑨ Saturated Solution of NaCl [FS Lot # 912763] in NH_2O .
- ⑩ ~~Acetone~~ 1N NaHCO_3 solution [84g of NaHCO_3 (Lot # FS 936883) in 1L of NH_2O].
- ⑪ Nano pure H_2O .
- ⑫ Several glass graduated beakers
- ⑬ Magnetic Stirrers.
- ⑭ Stir Plate, Corning Stirrer/Hot Plate
- ⑮ Plastic bottles to store solutions.
- ⑯ Several centrifuge tubes (50 ml)
- ⑰ Glass and rubber tip glass stirrer.
- ⑱ Filter Paper (19cm diameter, medium Porosity, glass fiber, FS)
- ⑲ Glass Funnel
- ⑳ Separator funnels for density separation.
- ㉑ Spatulas
- ㉒ Weighing balance Mettler PM 4600 Delta Range
- ㉓ Acetone [FS Lot # 966257]
- ㉔ Microscope
- ㉕ Table top Centrifuge by Fischer Scientific marathon 21K
- ㉖ Bromoform [FS Lot # 916133]
- ㉗ NN-dimethyl formamide [Lot # ~~4921 KE99~~ ^{AJ, 4/22/98}]

AJ, 4/22/98

NN-dimethyl formamide by ~~Matheson~~ ^{Matheson} Analytical Reagent
Fisher Scientific Lot # 893658

- ㉘ Drying oven (Stabil-Therm) C-4850-Q
- ㉙ Eppendorf Micropipettor to transfer solutions.
- ㉚ Eppendorf Pipettor Tips
- ㉛ PH electrode Orion 920A

PROCEDURE:-

- ① Crush Clinoptilolite using mortar & pestle. Sieve thru 8" diameter stainless steel 100-200 mesh sieves 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 5.0g of Clinoptilolite in several 100ml centrifuge tubes.
 - b) Add 50ml of NaOAc buffer (at PH=5) to each tube.
 - c) The suspension is digested in a $\approx 95^\circ\text{C}$ water bath for 30 minutes. The suspension is stirred with a rubber tipped rod before putting in water 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 10.0g of Clinoptilolite in several 100 ml centrifuge tubes.
 - b) Add 40 ml of 0.3 M Na-citrate solution and 5 ml of 1M NaHCO_3 solution (these solutions can be added ahead of time).
 - c) The temperature is brought to 75°C - 80°C (not $>80^\circ\text{C}$) in a water bath. The suspension is transferred to the water bath and 1 gm of solid $\text{Na}_2\text{S}_2\text{O}_4$ is added to each tube. The

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

d) A second 1 gm portion of $\text{Na}_2\text{S}_2\text{O}_4$ (sodium dithionite) is added with stirring and digested for 5 min.

e) A third 1 gm portion of $\text{Na}_2\text{S}_2\text{O}_4$ 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 ($\rho_{\text{BFC}} = 2.8899 \text{ g/cc}$) and diluent, N,N -dimethyl formamide ($\rho_{\text{DMF}} = 0.93445 \text{ g/cc}$) is prepared using the following formula for volume ratio.

$$V_d = V_h \frac{\rho_h - \rho_x}{\rho_x - \rho_d}$$

where ρ_x = density of the mixture desired

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

ρ_d = density of diluent

V_h = volume of heavy liquid

V_d = volume of diluent.

b) Transfer heavy liquid mixture to separatory 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. Separation 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 thru it.

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

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

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

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

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Following procedure ①, clinoptilolite was grounded, sieved into 100-200 mesh size fraction (150-75 μm).

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① Clinoptilolite grounded & sieved to 100-200 mesh size using procedure ① step.

② NaOAc Reagent Prepared for Carbonates removal

Sodium acetate Trihydrate = 136 g mixed with
(Lot # 94062, FS)

950 ml of nanopure water on stir plate.

Adjust pH = 5 with glacial CH_3COOH acid $\approx 29 \text{ ml}$.
Bring to mark of 1000 ml after pH adjust by adding H_2O .

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① Clinoptilolite $\sim 7100 \text{ gms}$, size 100-200 mesh was cleaned with DI H_2O several times (15 times) till supernatant was no more milky. Used ultrasonic cleaner first few times.

② 0.3M Na-citrate Reagent prepared for iron-oxide removal. 0.3 M Na-citrate stock solution.

88 g of Na-citrate dihydrate was mixed with 950 ml of nano pure H₂O on stir plate. Bring it to 1000 ml mark after complete mixing by adding H₂O.

- (3) 1N NaHCO₃ stock solution was prepared by adding 84 g of NaHCO₃ (lot # 936883, FS) to 950 ml of nanopure H₂O. Mix it on stir plate & raise the level to 1000 ml after complete mixing.

- (4) Using procedure step (3), carbonate impurities were removed. Clinoptilolite was transferred upto 5 ml mark in 19 falcon tubes (50 ml).
 (b) 50 ml of NaOAc was added to each tube.
 (c) soaked at ~ boiling water, 95°C water bath for 30 min.
 (d) centrifuged at 6000 rpm for 5 min. The supernatant decanted.
 (e) Repeat centrifuge with nanopure H₂O 2 times.

4/29/98

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Using procedure step (4), iron oxides were removed from clinoptilolite already treated for carbonate removal. Add, upto 20 ml level in Falcon tubes (50 ml), 20 ml Na-citrate solution (0.3 M), 2.5 ml of 1N NaHCO₃ using micropipette. The suspension is mixed with Na dithionite in water bath at 75°C. Add 3 times at the interval of 5 min & quantity of Na dithionite add each time is 0.5 g. Centrifuge at 6000 rpm for 5 min. Finally, saturated NaCl is added & digested in water bath for 5 min. The suspension is then washed with H₂O, centrifuged, filtered & dried in oven.

5/1/98

AJ

Following Procedure step (5) heavy liquid separation was done to remove impurities.

NN-dimethyl formamide (diluent) density = 0.93445 g/cc

Bromoform (heavy liquid) = 150 ml

$\rho_{\text{Bromoform}} = 2.8899 \text{ g/cc}$, $\rho_{\text{desired}} = 2.3 \text{ g/cc}$

$$V_d = 150 \frac{2.8899 \text{ g/cc} - 2.3}{2.3 - 0.93445}$$

$$= 150 \frac{0.5899}{1.36555}$$

$$= 64.8 \text{ ml}$$

$$(\text{volume of diluent})$$

NN-dimethyl formamide

Mix 150 ml of Bromoform with 64.8 ml of NN-dimethyl formamide using graduated cylinders under the exhaust hood.

5/4/98

AJ

- (1) Wash cleaned (pretreated) Clinoptilolite for carbonate, iron oxide & heavy liquids removal with DI H₂O & dry it in oven. Total wt \approx 85 g

- (2) Prepare another batch of Clinoptilolite to 100-200 mesh size fraction by crushing & sieving (step 1 of procedure on page 3).

5/5/98

Prepare Clinoptilolite to 100-200 mesh size fraction
Total wt. \approx 88 g

5/6/98

- (1) Prepare Clinoptilolite to 100-200 mesh size fraction. Total wt \approx 127 g

- (2) Washed Clinoptilolite using procedure step 2.

- (3) 1N NaOAc buffer at PH=5

Na acetate trihydrate = 136 g (937077)

Nano pure water \approx 1 L

acetic acid \approx 29 ml to adjust PH=5

④ Na citrate (0.3M) solution
88 g of Na citrate dihydrate / 1L of nanopure water. Stirred on magnetic stirrer.

⑤ Saturated NaCl solution prepared for iron-oxide removal step.

Nanopure water \approx 550 ml

NaCl crystals = 146 g

⑥ Using procedure step #③, carbonates were removed from clinoptilolite.

⑦ Using procedure step #④, iron oxide impurities were removed from clinoptilolite.

5/2/98
JP

⑧ Using procedure step #⑤ other mineral impurities were removed from clinoptilolite by heavy liquid separation

5/11/98

JP

0800hr

Approximately 155g of purified clinoptilolite was prepared using the above procedures. The solid was placed in a beaker labeled CDV+100/200*UC*W*~~CE~~*HL

5/11/98

JP

0820hr

Preparation of Na-Form clinoptilolite

Obj - generate Na-clinoptilolite

Method - cation exchange sites are filled with Na^+ by reacting solid with 3M NaCl.

Equipment + Materials

- Fisher Versabath Model 236
- 3M NaCl (lot 914193)
- 500ml PP bottles
- Blue M drying oven

Procedure

JP 0800hr

① Approximately 40 g of purified clinoptilolite was placed in a 500ml PP bottle. 3 bottles were prepared.

② 400 ml of 3M NaCl was added to the bottles

③ The bottles were placed in water bath set at 90°C and continuously agitated for about 2 weeks. The 3M NaCl solution was replaced every 2 days.

④ In addition a 20g batch of clinoptilolite was prepared using 200ml of solution.

JP 5/29/98

0830hr

⑤ Bottles were removed from water bath.

⑥ The clinoptilolite was washed several times with deionized water at 90°C and dried in an oven at 65°C .

⑦ After drying the chirophile was transferred to a glass jar CPT and labeled CDV * 100/200 * UC * WA * CPT * HL * NaF. 2/13/98

⑧ The jar was placed in a desiccator over saturated NaCl.

7/10/98 AJ

40 polycarbonate centrifuge tubes (~50 ml capacity) are acid washed in 10% HNO_3 acid bath.

7/13/98 AJ

40 polycarbonate centrifuge tubes were rinsed with nanopure water several times, air dried for few hrs. & then dried in the oven (~65°C) overnight.

Preparation of stock solutions of HNO_3 and NaHCO_3 of various concentrations.

Objective:- Prepare stock solutions

| | |
|-------------------------------|--------------------------|
| 200 ml 1M HNO_3 | 50 ml 50% HNO_3 |
| 200 ml 0.1M HNO_3 | |
| 200 ml 0.02M HNO_3 | |
| 250 ml 1M NaHCO_3 | |
| 250 ml 0.5M NaHCO_3 | |
| 250 ml 0.1M NaHCO_3 | |
| 250 ml 0.05M NaHCO_3 | |
| 250 ml 0.01M NaHCO_3 | |

Equipment & Materials:-

500 ml Glass Beakers
Magnetic Stirrer & stirrer bars.
Conc. HNO_3 (lot 983546 FS)
 NaHCO_3 (lot # 936883)
FS

Nanopure water

Method:- The NaHCO_3 solution should be prepared with degassed ultrapure water (deionized water is degassed by boiling it in flasks & cooling it while the mouth was covered with rubber stopper), and stored in tightly capped glass reagent bottles.

Procedure:-

① Boil 2L of nanopure water in 2 1000 ml capacity flasks & cool it with rubber stopper.

7/14/98 AJ

② 21.02 g of NaHCO_3 & 250 ml of degassed nanopure water. (1M NaHCO_3)

③ 10.51 g of NaHCO_3 dissolved in 250 ml of degassed nanopure water (0.5M NaHCO_3)

④ 2.10 g of NaHCO_3 dissolved in 250 ml of degassed nanopure water (0.1M NaHCO_3)

⑤ 1.05 g of NaHCO_3 dissolved in 250 ml of degassed nanopure water (0.05M NaHCO_3)

⑥ 0.21 g of NaHCO_3 dissolved in 250 ml of degassed nanopure water (0.01M NaHCO_3)

⑦ 12.6 ml of conc. HNO_3 (~16M) & dilute it to 200 ml of nanopure water. [1M HNO_3]

⑧ 20 ml of 1M HNO_3 & dilute it to 200 ml of nanopure H_2O . [0.1M HNO_3]

⑨ 4 ml of 1M HNO_3 & dilute it to 200 ml of nanopure H_2O . [0.02M HNO_3]

7/16/98 AJ

⑩ Dilute 25 ml of conc. HNO_3 with 25 ml of nanopure H_2O . (50% HNO_3).

7/17/98 AJ

C20-U* absorption experiments were started using the following procedure.

Experimental Procedure for experiment C20 - U sorption on clinoptilolite at 25° C.

Objective: To investigate the characteristics of U sorption on clinoptilolite as a function of pH.

Initial conditions:

- $\Sigma \text{U} = 500 \text{ ppb}$ [$2.1 \times 10^{-6} \text{ molal}$]
- equilibrium with atmospheric $\text{CO}_2(\text{g})$; $\text{pCO}_2 = 10^{-3.5}$
- pH range 2.5-9.5
- solution volume = 32.5 ml
- mass of solid = 0.065 g
- M/V = 2

Equipment:

- Orion 920A pH meter and combination pH electrode
- Gyratory shaker
- Fisher Marathon 21K centrifuge
- Mettler PM4600 and AE240 balances
- Packard 2500 TR/AB LSC
- Eppendorf micropipettors and tips
- Oxford pipettes and tips
- Repipettor for transfer of scintillation cocktail

Supplies:

- Na-clinoptilolite (CDV*100/200*UC*WA*CPT*HL*Na)
- $2.1 \times 10^{-6} \text{ m } ^{235}\text{U}$ stock solution (spike 28A)
- polycarbonate centrifuge tubes with caps (50 ml capacity)
- polypropylene centrifuge tubes with caps (50 ml capacity)
- pH buffer solutions
- Ultima-Gold liquid scintillation cocktail
- 7 ml scintillation vials
- reagent grade NaHCO_3 (lot 936883)
- concentrated HNO_3 (lot 983546)
- HNO_3 and NaHCO_3 stock solutions at various concentrations
- weighing paper/boats
- deionized ultrapure water, DH_2O
- teflon beaker

Procedure:

A. Transfer $2.1 \times 10^{-6} \text{ m}$ (500 ppb) ^{235}U stock solution to experimental containers.

- Label 29 polycarbonate centrifuge tubes (~40 ml capacity) C20-U-pHi (where i is the approximate pH of each solution, see Table C20).
- Label 1 polycarbonate centrifuge tube C20-U-IU.
- Weigh and record the weight of each container.
- The concentration of spike 28A is $2.1 \times 10^{-6} \text{ m } ^{235}\text{U}$ and therefore this stock solution does not require dilution. About 1.2L of spike 28A will be required for this experiment.
- Add ~32.5 g of the $2.1 \times 10^{-6} \text{ m } ^{235}\text{U}$ stock solution to each container. *Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.*
- Record weight of each container.

B. Add clinoptilolite to sample containers.

- Add ~0.065 g of Na-clinoptilolite to each sample container. **Do not** add clinoptilolite to the C20-U-IU container.
- Record weight of each sample container after addition of clinoptilolite.

Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaHCO_3 to each experimental container as listed in Table C20. Take care to add HNO_3 or NaHCO_3 in the concentrations specified in Table C20.
- For sample C20-U-IU (not listed in Table C20) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

Sample C20-U-IU for initial [U].

- Label two 7 ml LSC vials as C20-U-IUa and C20-U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from C20-U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container C20-U-IU

Wait for equilibrium.

- Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
- Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.

| SAMPLE NAME | WT. OF BOTTLE, g | WT. OF BOTTLE & U SOLN., g | WT. OF U SOLN., g | WT. OF Soln BOTTLE & CLINOPTILO LITE, g | WT. OF CLINOPTILO LITE, g | WT. AFTER pH ADJUST, g |
|-----------------|------------------|----------------------------|-------------------|--|---------------------------|------------------------|
| C20-pH 2.5 | 22.5195 | 55.0516 | 32.5321 | 55.1132 | 0.0616 | 55.2373 |
| C20-pH 2.75 | 22.3431 | 54.8453 | 32.5022 | 54.9092 | 0.0639 | 54.9644 |
| C20-pH 3 | 22.5595 | 55.0681 | 32.5086 | 55.1335 | 0.0654 | 55.2945 |
| C20-pH 3.25 | 22.2296 | 54.7357 | 32.5061 | 54.8006 | 0.0649 | 54.8941 |
| C20-pH 3.5 | 22.2594 | 54.7593 | 32.4999 | 54.8244 | 0.0651 | 54.9868 |
| C20-pH 3.75 | 22.3111 | 54.8262 | 32.5151 | 54.8912 | 0.0650 | 55.1245 |
| C20-pH 4 | 22.2753 | 54.7818 | 32.5065 | 54.8467 | 0.0649 | 55.1156 |
| C20-pH 4.25 | 22.5336 | 55.0265 | 32.4929 | 55.0869 | 0.0604 | 55.3758 |
| C20-pH 4.5 | 22.2878 | 54.7932 | 32.5054 | 54.8579 | 0.0647 | 55.1568 |
| C20-pH 4.75 | 22.1848 | 54.6757 | 32.4909 | 54.7267 | 0.0640 | 55.0252 |
| C20-pH 5 | 22.2032 | 54.7048 | 32.5016 | 54.7625 | 0.0577 | 55.0733 |
| C20-pH 5.25 | 22.4202 | 54.9158 | 32.4956 | 54.9785 | 0.0627 | 55.2724 |
| C20-pH 5.5 | 22.3656 | 54.8626 | 32.4970 | 54.9277 | 0.0651 | 55.2472 |
| C20-pH 5.75 | 22.2489 | 54.7646 | 32.5157 | 54.8281 | 0.0635 | 55.1464 |
| C20-pH 6 | 22.2732 | 54.7803 | 32.5071 | 54.8466 | 0.0663 | 55.1653 |
| C20-pH 6.25 | 22.1768 | 54.6716 | 32.4948 | 54.7298 | 0.0582 | 55.0488 |
| C20-pH 6.5 | 22.5974 | 55.0662 | 32.4688 | 55.1115 | 0.0453 | 55.4298 |
| C20-pH 6.75 | 22.2156 | 54.7078 | 32.4922 | 54.7683 | 0.0605 | 55.1043 |
| C20-pH 7 | 22.3069 | 54.7923 | 32.4854 | 54.8421 | 0.0498 | 55.1866 |
| C20-pH 7.25 | 22.3863 | 54.9186 | 32.5323 | 54.9815 | 0.0629 | 55.3523 |
| C20-pH 7.5 | 22.2582 | 54.7574 | 32.4992 | 54.8013 | 0.0439 | 55.1985 |
| C20-pH 7.75 | 22.5778 | 55.0877 | 32.5099 | 55.1495 | 0.0618 | 55.2541 |
| C20-pH 8 | 22.2590 | 54.7631 | 32.5041 | 54.8268 | 0.0637 | 54.9477 |
| C20-pH 8.25 | 22.2553 | 54.7384 | 32.4831 | 54.8008 | 0.0624 | 54.9737 |
| C20-pH 8.5 | 22.2548 | 54.7545 | 32.4997 | 54.8129 | 0.0584 | 55.0733 |
| C20-pH 8.75 | 22.2338 | 54.7222 | 32.4884 | 54.7876 | 0.0648 | 55.0111 |
| C20-pH 9 | 22.1519 | 54.6393 | 32.4874 | 54.7055 | 0.0662 | 55.1060 |
| C20-pH 9.25 | 22.2052 | 54.7131 | 32.5079 | 54.7789 | 0.0658 | 55.7405 |
| C20-pH 9.5 | 22.3118 | 54.8376 | 32.5258 | 54.9024 | 0.0648 | 56.6113 |
| C20-U-IU | 22.3322 | 54.8251 | 32.4929 | | | 55.9446 |
| | wt. Of vial | wt. Of vial + sample | wt. Of sample | | | |
| C20-U-lua | 7.5781 | 8.0791 | 0.5010 | | | |
| C20-U-lub | 7.6644 | 8.1647 | 0.5003 | | | |
| C20-U-IU (left) | 53.8890 g. | | | | | |

Table 20C. Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 M NaNO₃ solutions with 500 ppb U in contact with clindamycin. ~~40 ml~~ (40 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2.5 | 0.12 | 1 |
| 2.75 | 0.055 | 1 |
| 3 | 0.165 | 0.1 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 3.25 | 0.095 | 0.05 |
| 3.5 | 0.165 | 0.1 |
| 3.75 | 0.235 | 0.1 |
| 4 | 0.270 | 0.1 |
| 4.25 | 0.290 | 0.1 |
| 4.5 | 0.300 | 0.1 |
| 4.75 | 0.310 | 0.1 |
| 5 | 0.315 | 0.1 |
| 5.25 | 0.315 | 0.1 |
| 5.5 | 0.320 | 0.1 |
| 5.75 | 0.320 | 0.1 |
| 6 | 0.320 | 0.1 |
| 6.25 | 0.325 | 0.1 |
| 6.5 | 0.330 | 0.1 |
| 6.75 | 0.335 | 0.1 |
| 7 | 0.350 | 0.1 |
| 7.25 | 0.370 | 0.1 |
| 7.5 | 0.410 | 0.1 |
| 7.75 | 0.100 | 0.5 |
| 8 | 0.120 | 0.5 |
| 8.25 | 0.170 | 0.5 |
| 8.5 | 0.255 | 0.5 |
| 8.75 | 0.210 | 1 |
| 9 | 0.380 | 1 |
| 9.25 | 0.920 | 1 |
| 9.5 | 1.630 | 1 |

C20-U-IU

0.100

50% HNO₃

7/20/98 AJ

Prepare sample solutions to measure initial U-conc. following the Step B "Sample C-20-U-IU for initial [U]" from procedure on page 13.

| | | |
|-----------|--|---|
| | wt. of vial + 0.5 ml of 0.02 M HNO_3 | wt. of vial + 0.02 M HNO_3 + sample |
| C20-U-Iua | 7.5781 | 8.0791 |
| C20-U-Iuh | 7.6644 | 8.1647 |

Add 5 ml of Ultima Gold Cocktail (AB) & set aside for counting. (Data on Page 18-19).
AJ

7/22/98

AJ

Preparation of Heavy liquid of $\rho = 2.75 \text{ g/cc}$ for removal of impurities from quartz.
 $\rho_{\text{quartz}} = 2.65 \text{ g/cc}$

Following Step # 5 on page 4 of this book.
HL of $\rho = 2.75 \text{ g/cc}$ was prepared

150 ml of Bromoform, $\rho_{\text{bromoform}} = 2.8899 \text{ g/cc}$
? of $\text{N-N-dimethyl Formamide}$, $\rho_{\text{formamide}} = 0.93445 \text{ g/cc}$

$$\begin{aligned} \text{N-N-dimethylformamide} &= 150 \frac{2.8899 - 2.75}{2.75 - 0.93445} \\ &= 150 \times 0.07706 \\ &= 11.56 \text{ ml} \end{aligned}$$

Mix 150 ml of bromoform with 11.56 ml of $\text{N-N-dimethyl formamide}$.

on page 4
7/23/98 AJ Using Procedure step # 5 (other mineral impurities were removed from quartz by heavy liquid separation.

Preparation of Reagents for Al determination
② Preparation of 0.02 N H_2SO_4

Add 0.28 ml of Conc. H_2SO_4 (Lot # 913495) to 350 ml of DI H_2O . Dilute it to 500 ml with H_2O & store in plastic bottle.

③ Preparation of Buffer Reagent

136 g of Na acetate trihydrate (Lot # 937077) is added to 800 ml of nanopure H_2O . Dilute 2.9 ml of CH_3COOH (Lot # 971798) in 50 ml of H_2O . Add 40 ml of acetic acid soln. to the sodium acetate soln & dilute to 1 L ^{with} H_2O . Store in plastic bottle.

④ Preparation of Stock Dye Soln.

Eriochrome Cyanine R
Add 0.150 g (150 mg) [Lot # 249482 588] to 50 ml of nanopure H_2O . Dilute 5 ml of glacial CH_3COOH (Lot # 971798) to 10 ml with H_2O . Adjust pH to 2.9 with acetic acid soln. added dropwise. ($\approx 3.5 \text{ ml}$ is required). Dilute the solution with H_2O to 100 ml. Store soln. in plastic bottle.

⑤ Working Dye Soln.

Add 10 ml of Stock Dye Soln. & dilute it to 100 ml with H_2O . Store in plastic bottle.

⑥ Preparation of 0.01 M EDTA (Lot # 905518)

Add 3.7 g of disodium ethylenediamine tetraacetate, EDTA in about 500 ml of H_2O . Dilute it to 1 L with H_2O . Store in plastic bottle.

7/24/98 AJ

Preparation of Stock solutions (Reagents) for Silica determination by spectrophotometry.

① Preparation of 1N H_2SO_4

Dilute 14 ml of conc. H_2SO_4 (Lot # 913495) with H_2O to 500 ml. Store in plastic bottle.

② Preparation of 1+1 HCl soln.

Mix 250 ml of HCl (Lot # 956110) with 250 ml of H_2O . Store in plastic bottle.

③ Preparation of 6M NaOH

Dissolve 240 g of NaOH (Lot # 980698) with H_2O & make it up to 1000 ml in a plastic beaker. Plastic beaker is used to avoid any silica impurities in soln. from glass. Store in plastic bottle.

④ Preparation of Ammonium molybdate Reagent

Place 10 g of ammonium molybdate (Lot # 901533) in water in 100 ml volumetric beaker. Dissolve by stirring & gentle warming. Dilute it to 100 ml. Filter if necessary. ~~Not reqd~~ Adjust solution pH 7 to 8 with 6M NaOH added dropwise. Store soln. in plastic bottle.

⑤ Preparation of Oxalic Acid Solution

Dissolve 7.5 g oxalic acid (Lot # 906135B) in water & dilute it to 100 ml. Store soln in plastic bottle.

⑥ Preparation of Reducing Agent

Dissolve 0.500 g (500 mg) of 1-amino-2-naphthal-4-sulfonic acid (Lot # 1801KEKS) and 1 g of sodium sulfite (Na_2SO_3 , Lot # 904672) in 50 ml of H_2O with gentle warming (if necessary). Dissolve 30 g of sodium bisulfite ($NaHSO_3$, Lot # 915268) in 150 ml of H_2O . Mix the two solutions & filter (fast filter) into a plastic bottle. Cover the bottle with foil & refrigerate the soln. Avoid exposure to light.

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Protocol # 24

7/23/98

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.17 |
| Region B: | 100 - 350 | | 0 | 5.0 | 3.26 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 28.94 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|-----------------|------------|------------|------------|--------------------------|
| 1 | 999.99 | 20.17 | 1.41 | 3.264 3.50 | 28.94 1.18 137.60 B |
| 1 | MISSING TUBE(S) | | | | |
| 3 | 0.30 | 53.17 | 58.82 | 5460.069 | 4.94 5524.39 4.93 613.81 |
| 4 | 0.30 | 16.50 | 134.0 | 5456.736 | 4.94 5487.73 4.94 612.53 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 23-Jul-98 03:50

C14 Eff (0-156 keV) = 96.89 %

C14 CHI SQUARE IPA DATA PROCESSED - 23-Jul-98 04:01

C14 Chi Square = 29.94

H3 IPA DATA PROCESSED - 23-Jul-98 04:02

H3 Eff (0-18.6 keV) = 65.08 %

H3 CHI SQUARE IPA DATA PROCESSED - 23-Jul-98 04:12

H3 Chi Square = 18.60

BKG IPA DATA PROCESSED - 23-Jul-98 05:13

Bkg (0-18.6 keV) = 23.15 cpm

Bkg (0-156 keV) = 33.03 cpm

7/27/98 AJ

① Quartz cleaned with heavy liquid density separation was cleaned with nanopure H_2O & kept in oven for drying.

② Following step "Sample Experimental Solutions for [U] & PH" samples for LSA were prepared ^{AJ 7/27/98} labeled.

Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., C20-U-pH3a and C20-U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

7/28/98 AJ

7/29/98

① Containers were weighed before sampling and pH measurements.

② PH was measured and samples were weighed after sampling. The results are noted in the following table:-

| | 7/28/98 | 7/29/98 | 7/29/98 | 7/29/98 |
|-------------|---|-------------|--|--|
| Sample Name | Container wt. before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements | Container wt. after addition of 0.1 HNO_3 to lower PH. |
| C20-pH 2.5 | 55.0272 | 2.45 | 53.9883 | |
| C20-pH 2.75 | 54.7595 | 2.68 | 53.7366 | |
| C20-pH 3 | 55.1107 | 2.98 | 54.0844 | |
| C20-pH 3.25 | 54.6535 | 3.34 | 53.6248 | |
| C20-pH 3.5 | 54.7475 | 3.80 | 53.7109 | |
| C20-pH 3.75 | 54.9398 | 4.96 | 53.9103 | |
| C20-pH 4 | 54.8912 | 7.0 | 53.8633 | |
| C20-pH 4.25 | 55.1465 | 7.20 | 54.1214 | 54.2207 |
| C20-pH 4.5 | 54.9650 | 7.28 | 53.9551 | 54.0238 |
| C20-pH 4.75 | 54.7908 | 7.36 | 53.7618 | 53.84732 |
| C20-pH 5 | 54.8651 | 7.40 | 53.8357 | 53.9109 |
| C20-pH 5.25 | 55.0966 | 7.24 | 54.0754 | |
| C20-pH 5.5 | 55.0833 | 7.43 | 54.0566 | 54.1333 |
| C20-pH 5.75 | 54.9241 | 7.43 | 53.8497 | 53.9190 |
| C20-pH 6 | 54.9705 | 7.45 | 53.9451 | |
| C20-pH 6.25 | 54.7334 | 7.45 | 53.7060 | 53.7929 |
| C20-pH 6.5 | 55.2456 | 7.68 | 54.2247 | 54.2600 |
| C20-pH 6.75 | 54.9220 | 7.52 | 53.9042 | |
| C20-pH 7 | 54.9124 | 7.68 | 53.8964 | 53.9555 |
| C20-pH 7.25 | 55.1867 | 7.76 | 54.1533 | |
| C20-pH 7.5 | 54.9313 | 7.74 | 53.8739 | 53.9349 |
| C20-pH 7.75 | 54.9647 | 7.96 | 53.9239 | |
| C20-pH 8 | 54.7073 | 8.15 | 53.6828 | |
| C20-pH 8.25 | 54.7334 | 8.31 | 53.7038 | |
| C20-pH 8.5 | 54.8550 | 8.62 | 53.8345 | |
| C20-pH 8.75 | 54.8033 | 8.80 | 53.7592 | |
| C20-pH 9 | 54.9228 | 9.01 | 53.8785 | |
| C20-pH 9.25 | 55.5470 | 9.21 | 53.4645 | |
| C20-pH 9.5 | 56.4335 | 9.17 | 55.4004 | |
| C20-U-U | 53.8586 | | | |

The measured PH in the above table showed error. This error was attributed to the volumes of HNO_3 & $NaHCO_3$ added to adjust PH for 40 ml [U] solution. Actual U solution used in expt. is 32.5 ml. The

corrected table for PH adjustment is as follows :-

Table 20C. Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 500 ppb U in contact with clinoptilolite (32.5 ml)

| Estimated solution pH | Volume of HNO ₃ needed, ml | Molarity of HNO ₃ to use |
|-----------------------|---|---------------------------------------|
| 2.5 | 0.097 | 1 |
| 2.75 | 0.043 | 1 |
| 3 | 0.131 | 0.1 |
| Estimated solution pH | Volume of NaHCO ₃ needed, ml | Molarity of NaHCO ₃ to use |
| 3.25 | 0.075 | 0.05 |
| 3.5 | 0.133 | 0.1 |
| 3.75 | 0.186 | 0.1 |
| 4 | 0.216 | 0.1 |
| 4.25 | 0.233 | 0.1 |
| 4.5 | 0.242 | 0.1 |
| 4.75 | 0.248 | 0.1 |
| 5 | 0.251 | 0.1 |
| 5.25 | 0.254 | 0.1 |
| 5.5 | 0.254 | 0.1 |
| 5.75 | 0.256 | 0.1 |
| 6 | 0.258 | 0.1 |
| 6.25 | 0.260 | 0.1 |
| 6.5 | 0.263 | 0.1 |
| 6.75 | 0.269 | 0.1 |
| 7 | 0.279 | 0.1 |
| 7.25 | 0.297 | 0.1 |
| 7.5 | 0.329 | 0.1 |
| 7.75 | 0.078 | 0.5 |
| 8 | 0.098 | 0.5 |
| 8.25 | 0.136 | 0.5 |
| 8.5 | 0.205 | 0.5 |
| 8.75 | 0.169 | 1 |
| 9 | 0.302 | 1 |
| 9.25 | 0.736 | 1 |
| 9.5 | 1.306 | 1 |

more acid was required to lower the PH of the samples. These samples were reweighed after addition of acid & weight was recorded in table on page 21. These samples were repeated for equilibrium step for 10 days.
The other samples
AJ 7/28/98

③ The following table shows the weights of samples for LSA

| Sample Name | wt. Of vial | wt. Of vial + sample |
|---------------|-------------|----------------------|
| C20-pH 2.5at | 7.6402 | 8.1425 |
| C20-pH 2.5bt | 7.6463 | 8.1466 |
| C20-pH 2.75at | 7.5871 | 8.0849 |
| C20-pH 2.75bt | 7.5827 | 8.0830 |
| C20-pH 3at | 7.6228 | 8.1198 |
| C20-pH 3bt | 7.5644 | 8.0638 |
| C20-pH 3.25at | 7.6048 | 8.1036 |
| C20-pH 3.25bt | 7.6658 | 8.1653 |
| C20-pH 3.5at | 7.5986 | 8.0983 |
| C20-pH 3.5bt | 7.6680 | 8.1674 |
| C20-pH 3.75at | 7.6194 | 8.1170 |
| C20-pH 3.75bt | 7.5444 | 8.0441 |
| C20-pH 4at | 7.6569 | 8.1565 |
| C20-pH 4bt | 7.6485 | 8.1476 |
| C20-pH 4.25at | 7.5999 | 8.0969 |
| C20-pH 4.25bt | 7.6446 | 8.1429 |
| C20-pH 4.5at | 7.5688 | 8.0660 |
| C20-pH 4.5bt | 7.6286 | 8.1284 |
| C20-pH 4.75at | 7.6324 | 8.1356 |
| C20-pH 4.75bt | 7.6556 | 8.1547 |
| C20-pH 5at | 7.6193 | 8.1202 |
| C20-pH 5bt | 7.5929 | 8.0944 |
| C20-pH 5.25at | 7.6525 | 8.1516 |
| C20-pH 5.25bt | 7.5878 | 8.0868 |
| C20-pH 5.5at | 7.6069 | 8.1076 |
| C20-pH 5.5bt | 7.6433 | 8.1419 |
| C20-pH 5.75at | 7.5761 | 8.0759 |
| C20-pH 5.75bt | 7.6290 | 8.1285 |
| C20-pH 6at | 7.6597 | 8.1574 |
| C20-pH 6bt | 7.5879 | 8.0869 |
| C20-pH 6.25at | 7.6078 | 8.1070 |
| C20-pH 6.25bt | 7.6090 | 8.1073 |
| C20-pH 6.5at | 7.6559 | 8.1540 |
| C20-pH 6.5bt | 7.5580 | 8.0560 |
| C20-pH 6.75at | 7.6157 | 8.1136 |
| C20-pH 6.75bt | 7.6097 | 8.1072 |
| C20-pH 7at | 7.6552 | 8.1483 |
| C20-pH 7bt | 7.7028 | 8.1997 |
| C20-pH 7.25at | 7.6577 | 8.1556 |
| C20-pH 7.25bt | 7.5581 | 8.0565 |
| C20-pH 7.5at | 7.6105 | 8.1047 |
| C20-pH 7.5bt | 7.6711 | 8.1659 |
| C20-pH 7.75at | 7.6374 | 8.1354 |
| C20-pH 7.75bt | 7.582 | 8.0746 |
| C20-pH 8at | 7.6600 | 8.1587 |
| C20-pH 8bt | 7.6323 | 8.1297 |

| | | |
|---------------|--------|--------|
| C20-pH 8.25at | 7.6404 | 8.1387 |
| C20-pH 8.25bt | 7.6943 | 8.1915 |
| C20-pH 8.5at | 7.6385 | 8.1336 |
| C20-pH 8.5bt | 7.6603 | 8.1550 |
| C20-pH 8.75at | 7.5757 | 8.0724 |
| C20-pH 8.75bt | 7.6216 | 8.1181 |
| C20-pH 9at | 7.5950 | 8.0884 |
| C20-pH 9bt | 7.5731 | 8.0678 |
| C20-pH 9.25at | 7.5965 | 8.0949 |
| C20-pH 9.25bt | 7.6326 | 8.1298 |
| C20-pH 9.5at | 7.6257 | 8.1234 |
| C20-pH 9.5bt | 7.5642 | 8.0634 |

These samples were left under the glass cover (away from light) for 24 hrs before LSA counting.

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AJ

7/30/98 ① LSA results are as follows: The
samples were removed & kept aside.

② Using the procedure on page 26, these
samples were desorbed.

30 Jul 98 05:12 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

User

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.41 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.72 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.98 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLA |
|----|--------|------------|------------|------------|--------------------------|
| 1 | 999.99 | 20.41 | 1.40 | 3.720 | 3.28 29.98 1.16 148.22 |
| 2 | 0.82 | 10.07 | 121.1 | 5452.377 | 2.99 5470.02 2.99 629.72 |
| 3 | 0.84 | 21.25 | 66.29 | 5314.137 | 2.99 5334.30 3.00 622.99 |
| 4 | 0.84 | 18.87 | 72.49 | 5318.899 | 2.99 5340.26 2.99 624.08 |
| 5 | 0.81 | 10.45 | 118.2 | 5550.601 | 2.98 5567.55 2.99 623.01 |
| 6 | 0.85 | 14.88 | 86.63 | 5242.162 | 3.00 5261.78 3.00 626.52 |
| 7 | 0.83 | 28.98 | 53.24 | 5377.003 | 2.99 5408.57 2.99 623.29 |
| 8 | 0.86 | 17.96 | 74.41 | 5218.373 | 2.99 5244.44 2.99 627.03 |
| 9 | 0.84 | 21.25 | 66.29 | 5310.565 | 3.00 5331.92 3.00 626.51 |
| 10 | 0.85 | 20.76 | 67.06 | 5268.044 | 2.99 5294.72 2.99 626.61 |
| 11 | 0.84 | 28.40 | 53.70 | 5299.852 | 3.00 5333.11 3.00 626.72 |
| 12 | 1.03 | 19.39 | 64.13 | 4317.639 | 3.00 4338.95 3.00 624.49 |
| 13 | 1.03 | 7.74 | 135.1 | 4317.639 | 3.00 4324.39 3.01 628.89 |
| 14 | 1.57 | 7.61 | 111.1 | 2828.764 | 3.00 2833.71 3.01 625.80 |
| 15 | 1.59 | 15.44 | 61.55 | 2808.859 | 2.99 2822.22 3.00 624.54 |
| 16 | 1.39 | 21.31 | 51.43 | 3205.633 | 3.00 3225.41 3.00 626.04 |
| 17 | 1.35 | 9.22 | 101.7 | 3290.354 | 3.00 3295.94 3.01 629.45 |
| 18 | 1.35 | 15.14 | 67.81 | 3308.873 | 2.99 3326.31 3.00 627.34 |
| 19 | 1.34 | 20.63 | 53.67 | 3322.399 | 3.00 3344.65 3.00 629.20 |
| 20 | 1.25 | 14.79 | 71.80 | 3564.280 | 3.00 3578.82 3.00 627.67 |

25

| | | | | | |
|----|------|-------|-------|----------|--------------------------|
| 21 | 1.27 | 10.81 | 67.59 | 3498.642 | 3.00 3513.33 3.01 627.55 |
| 22 | 1.26 | 10.54 | 94.09 | 3526.439 | 3.00 3542.24 3.01 628.63 |
| 23 | 1.25 | 23.59 | 50.32 | 3563.480 | 3.00 3594.02 3.00 626.61 |
| 24 | 1.34 | 12.42 | 79.73 | 3313.444 | 3.00 3328.23 3.01 626.95 |
| 25 | 1.38 | 7.12 | 125.5 | 3222.367 | 3.00 3231.61 3.01 627.75 |
| 26 | 1.26 | 14.51 | 72.60 | 3546.280 | 2.99 3562.08 3.00 626.84 |
| 27 | 1.28 | 13.18 | 77.77 | 3479.093 | 3.00 3489.55 3.01 628.86 |
| 28 | 1.27 | 3.21 | 269.0 | 3515.965 | 2.99 3525.92 3.00 625.59 |
| 29 | 1.27 | 13.45 | 76.84 | 3521.477 | 2.99 3536.16 3.00 625.90 |
| 30 | 1.25 | 20.39 | 56.07 | 3574.680 | 2.99 3594.82 3.00 627.29 |
| 31 | 1.24 | 20.72 | 55.62 | 3591.441 | 3.00 3618.41 3.00 627.38 |
| 32 | 1.22 | 5.82 | 159.5 | 3647.100 | 3.00 3655.26 3.01 624.98 |
| 33 | 1.20 | 17.92 | 63.10 | 3718.780 | 3.00 3737.52 3.00 624.20 |
| 34 | 1.27 | 22.11 | 52.36 | 3503.367 | 3.00 3525.14 3.00 626.85 |
| 35 | 1.24 | 36.04 | 37.45 | 3586.603 | 3.00 3626.47 2.99 621.02 |
| 36 | 1.19 | 6.48 | 146.8 | 3742.498 | 3.00 3748.17 3.01 629.92 |
| 37 | 1.21 | 15.95 | 68.76 | 3670.660 | 3.00 3687.37 3.01 626.19 |
| 38 | 1.18 | 12.64 | 83.79 | 3797.128 | 2.99 3813.24 2.99 630.11 |
| 39 | 1.17 | 22.32 | 54.17 | 3800.554 | 3.00 3823.86 3.00 627.24 |
| 40 | 1.07 | 20.71 | 59.89 | 4157.027 | 3.00 4176.56 3.00 626.14 |
| 41 | 1.09 | 15.37 | 74.59 | 4096.280 | 2.99 4111.30 3.00 625.63 |

30 Jul 98 06:12 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

User

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLA |
|----|-----------------|------------|------------|------------|--------------------------|
| 42 | 1.01 | 19.19 | 65.28 | 4403.210 | 3.00 4426.45 3.00 627.87 |
| 43 | 1.04 | 17.09 | 70.31 | 4293.396 | 2.99 4311.36 3.00 625.75 |
| 44 | 0.94 | 18.95 | 68.32 | 4734.578 | 3.00 4756.19 3.00 623.32 |
| 45 | 0.91 | 27.94 | 52.19 | 4896.280 | 3.00 4928.26 3.00 624.17 |
| 46 | 0.88 | 6.86 | 162.4 | 5083.780 | 2.99 5092.75 3.00 630.53 |
| 47 | 0.87 | 32.46 | 48.04 | 5115.820 | 3.00 5144.73 3.00 625.84 |
| 48 | 0.87 | 27.86 | 53.48 | 5161.797 | 2.99 5190.71 2.98 627.76 |
| 49 | 0.83 | 19.35 | 71.57 | 5401.099 | 2.99 5425.44 2.99 626.58 |
| 50 | 0.83 | 21.76 | 65.54 | 5379.413 | 2.99 5407.37 2.99 627.60 |
| 51 | 0.84 | 35.54 | 45.94 | 5353.423 | 2.98 5386.69 2.98 626.25 |
| 52 | 0.81 | 31.44 | 50.91 | 5508.625 | 3.00 5545.33 2.99 630.78 |
| 53 | 0.82 | 14.95 | 87.86 | 5415.792 | 3.00 5434.65 3.00 631.54 |
| 54 | 0.84 | 11.73 | 105.5 | 5346.280 | 2.99 5361.69 2.99 634.31 |
| 55 | 0.85 | 20.76 | 67.06 | 5265.691 | 2.99 5292.37 2.99 632.16 |
| 56 | 0.85 | 7.82 | 147.4 | 5262.162 | 2.99 5271.19 3.00 637.88 |
| 57 | 0.85 | 17.23 | 77.25 | 5226.868 | 3.00 5241.78 3.00 636.34 |
| 58 | 0.85 | 30.18 | 51.14 | 5237.456 | 3.00 5267.67 3.00 639.34 |
| 59 | 0.90 | 28.48 | 51.78 | 4982.947 | 2.99 5011.13 2.99 641.17 |
| 1 | MISSING TUBE(S) | | | | |
| 61 | 0.82 | 44.22 | 40.16 | 5441.402 | 3.00 5494.41 2.99 616.79 |
| 62 | 0.81 | 20.33 | 69.79 | 5512.329 | 2.99 5544.09 2.99 621.98 |
| 1 | MISSING TUBE(S) | | | | |
| 64 | 0.83 | 26.57 | 56.64 | 5363.750 | 3.00 5395.32 3.00 621.44 |
| 65 | 0.85 | 31.35 | 49.79 | 5233.927 | 3.00 5273.55 3.00 618.95 |

Transfer clinoptilolite for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., C20d-U-pH3 for sample container C20-U-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw clinoptilolite from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO₃ to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

Desorption

| Sample Name | wt. Of PP tubes | wt. Of PP tubes after trans clinop | wt. Of PP tubes after acid add | Container wt. after trans clinop | Container wt. After acid add |
|--------------|-----------------|------------------------------------|--------------------------------|----------------------------------|------------------------------|
| C20d-pH 2.5 | 13.3937 | 17.6665 | 20.6793 | 49.6952 | 52.7176 |
| C20d-pH 2.75 | 14.0943 | 20.2947 | 23.3421 | 47.4918 | 50.5159 |
| C20d-pH 3 | 13.7952 | 17.5577 | 20.5892 | 50.2928 | 53.3098 |
| C20d-pH 3.25 | 13.3364 | 17.0436 | 20.0856 | 49.8889 | 52.9205 |
| C20d-pH 3.5 | 13.2573 | 17.8181 | 20.8562 | 49.1200 | 52.1377 |
| C20d-pH 3.75 | 13.4046 | 18.1220 | 21.1696 | 49.1489 | 52.1775 |
| C20d-pH 4 | 13.8878 | 18.0771 | 21.1250 | 49.6406 | 52.6649 |
| C20d-pH 4.25 | 13.3635 | | | | |
| C20d-pH 4.5 | 13.3306 | | | | |
| C20d-pH 4.75 | 13.3341 | | | | |
| C20d-pH 5 | 13.4177 | | | | |
| C20d-pH 5.25 | 13.3634 | 18.0681 | 21.0969 | 49.3445 | 52.3776 |
| C20d-pH 5.5 | 13.3830 | | | | |
| C20d-pH 5.75 | 13.4074 | | | | |
| C20d-pH 6 | 13.8372 | 17.9078 | 20.9474 | 48.8495 | 52.8736 |
| C20d-pH 6.25 | 13.3690 | | | | |
| C20d-pH 6.5 | 13.4433 | | | | |
| C20d-pH 6.75 | 13.8150 | 17.5241 | 20.4861 | 50.1771 | 53.1982 |
| C20d-pH 7 | 13.8525 | | | | |
| C20d-pH 7.25 | 13.3341 | 16.3240 | 19.3594 | 51.1363 | 54.1618 |
| C20d-pH 7.5 | 13.3010 | | | | |
| C20d-pH 7.75 | 13.7052 | 16.3613 | 19.4126 | 51.2558 | 54.2770 |
| C20d-pH 8 | 13.8881 | 16.9394 | 19.4824 | 50.5512 | 53.5756 |
| C20d-pH 8.25 | 13.8926 | 17.8423 | 20.9159 | 49.7234 | 52.7523 |
| C20d-pH 8.5 | 13.7589 | 16.5960 | 19.6555 | 50.9623 | 53.9836 |
| C20d-pH 8.75 | 13.2605 | 16.7361 | 19.7689 | 50.2061 | 53.2208 |
| C20d-pH 9 | 13.3703 | 17.7788 | 20.9279 | 49.4130 | 52.4272 |
| C20d-pH 9.25 | 13.3234 | 17.1455 | 20.2016 | 50.3998 | 53.6230 |
| C20d-pH 9.5 | 13.3687 | 17.6889 | 20.7286 | 51.0341 | 54.0515 |

These samples were placed on gyratory shaker at 125 rpm for 10 days.

8/5/98 AJ

① BET surface ^{area} analysis was performed on cleaned quartz, using Coulter 3100. Quartz used was 60/100 fraction.

wt. of tube assembly = 33.5688 g.

wt. of quartz added = 8.0014 g

wt. of tube assembly + outgassed sample = 41.5407 g

wt. of outgassed quartz = 41.5407
- 33.5688
7.9731 g.

BET surface area results are compiled in binder marked as "Coulter 3100 Surface Analysis"

8/6/98 AJ

objective:- BET surface area analysis on standard NIST 8005.

Equipments needed:- ① Coulter 3100
② NIST 8005 powder
③ He & N gas tanks
④ liquid nitrogen
⑤ tube & assembly.

Procedure 1-

wt. of tube assembly = 33.4238 g

wt. of NIST 8005 added = 1.7080 g
to tube assembly

wt. of outgassed sample = 35.1160 g
+ assembly

Actual wt. of outgassed sample = 35.1160
- 33.4238
1.6922 g

Results:-

The results of BET surface area are
compiled in binder marked as "Coulter
3100 Surface Analysis".

8/11/98

8/12/98

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8/19/98

AJ

8/14/98

Following procedure was used for desorption
samples. The weights were recorded in
a table before LSA counting.

C. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., C20-U-pH3a and C20-U-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Polycarbonate containers (Desorption)

| Sample Name | wt. Of vial | wt. Of vial + sample |
|---------------|-------------|----------------------|
| C20-pH 2.5aC | 7.6318 | 8.1340 |
| C20-pH 2.5bC | 7.6516 | 8.1515 |
| C20-pH 2.75aC | 7.6136 | 8.1146 |
| C20-pH 2.75bC | 7.6954 | 8.1958 |
| C20-pH 3aC | 7.7015 | 8.2010 |
| C20-pH 3bC | 7.5857 | 8.0863 |
| C20-pH 3.25aC | 7.6895 | 8.1905 |
| C20-pH 3.25bC | 7.6006 | 8.1039 |
| C20-pH 3.5aC | 7.6248 | 8.1260 |
| C20-pH 3.5bC | 7.59341 | 8.0976 |
| C20-pH 3.75aC | 7.5948 | 8.0968 |
| C20-pH 3.75bC | 7.7134 | 8.2152 |
| C20-pH 4aC | 7.6463 | 8.1461 |
| C20-pH 4bC | 7.6161 | 8.1167 |
| C20-pH 4.25aC | 7. | |
| C20-pH 4.25bC | | |
| C20-pH 4.5aC | | |
| C20-pH 4.5bC | | |
| C20-pH 4.75aC | | |
| C20-pH 4.75bC | | |
| C20-pH 5aC | | |
| C20-pH 5bC | | |
| C20-pH 5.25aC | 7.6178 | 8.1208 |
| C20-pH 5.25bC | 7.6621 | 8.1635 |
| C20-pH 5.5aC | | |
| C20-pH 5.5bC | | |
| C20-pH 5.75aC | | |
| C20-pH 5.75bC | | |
| C20-pH 6aC | 7.5618 | 8.0617 |
| C20-pH 6bC | 7.6323 | 8.1341 |
| C20-pH 6.25aC | | |
| C20-pH 6.25bC | | |
| C20-pH 6.5aC | | |
| C20-pH 6.5bC | | |
| C20-pH 6.75aC | 7.6292 | 8.1308 |
| C20-pH 6.75bC | 7.6503 | 8.1514 |
| C20-pH 7aC | | |
| C20-pH 7bC | | |
| C20-pH 7.25aC | 7.6207 | 8.1234 |
| C20-pH 7.25bC | 7.6229 | 8.1255 |
| C20-pH 7.5aC | | |
| C20-pH 7.5bC | | |
| C20-pH 7.75aC | 7.6080 | 8.1092 |
| C20-pH 7.75bC | 7.6134 | 8.1142 |

| | | |
|---------------|--------|--------|
| C20-pH 8aC | 7.6326 | 8.1345 |
| C20-pH 8bC | 7.6755 | 8.1776 |
| C20-pH 8.25aC | 7.5850 | 8.0869 |
| C20-pH 8.25bC | 7.5934 | 8.0967 |
| C20-pH 8.5aC | 7.5904 | 8.0935 |
| C20-pH 8.5bC | 7.6187 | 8.1215 |
| C20-pH 8.75aC | 7.6396 | 8.1415 |
| C20-pH 8.75bC | 7.6368 | 8.1396 |
| C20-pH 9aC | 7.6741 | 8.1749 |
| C20-pH 9bC | 7.6232 | 8.1256 |
| C20-pH 9.25aC | 7.6377 | 8.1379 |
| C20-pH 9.25bC | 7.5486 | 8.0515 |
| C20-pH 9.5aC | 7.6134 | 8.1163 |
| C20-pH 9.5bC | 7.6156 | 8.1195 |

8/11/98

Sorption for samples

| Sample Name | Container wt. before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements |
|-------------|---|-------------|--|
| C20-pH 2.5 | | | |
| C20-pH 2.75 | | | |
| C20-pH 3 | | | |
| C20-pH 3.25 | | | |
| C20-pH 3.5 | | | |
| C20-pH 3.75 | | | |
| C20-pH 4 | | | |
| C20-pH 4.25 | 53.9150 | 4.47 | 52.3013 |
| C20-pH 4.5 | 53.4987 | 4.56 | 51.9716 |
| C20-pH 4.75 | 53.6112 | 4.54 | 52.5912 |
| C20-pH 5 | 53.6443 | 5.73 | 52.6177 |
| C20-pH 5.25 | 0.0000 | | 0.0000 |
| C20-pH 5.5 | 53.8103 | 5.17 | 52.7739 |
| C20-pH 5.75 | 53.6238 | 6.3 | 52.5906 |
| C20-pH 6 | 0.0000 | | 0.0000 |
| C20-pH 6.25 | 53.5048 | 6.55 | 52.4807 |
| C20-pH 6.5 | 53.9512 | 6.88 | 52.9234 |
| C20-pH 6.75 | 0.0000 | | 0.0000 |
| C20-pH 7 | 53.6693 | 7.1 | 52.6516 |
| C20-pH 7.25 | 0.0000 | | 0.0000 |
| C20-pH 7.5 | 53.6446 | 7.48 | 52.6225 |

PP Test tubes [Description]

| Sample Name | wt. Of vial | wt. Of vial + sample |
|---------------|-------------|----------------------|
| C20-pH 2.5at | 7.6287 | 8.1294 |
| C20-pH 2.5bt | 7.6282 | 8.1260 |
| C20-pH 2.75at | 7.6257 | 8.1251 |
| C20-pH 2.75bt | 7.6440 | 8.1409 |
| C20-pH 3at | 7.6264 | 8.1210 |
| C20-pH 3bt | 7.6506 | 8.1454 |
| C20-pH 3.25at | 7.6271 | 8.1250 |
| C20-pH 3.25bt | 7.6598 | 8.1565 |
| C20-pH 3.5at | 7.7362 | 8.2317 |
| C20-pH 3.5bt | 7.6348 | 8.1316 |
| C20-pH 3.75at | 7.6901 | 8.1865 |
| C20-pH 3.75bt | 7.6495 | 8.1442 |
| C20-pH 4at | 7.6567 | 8.1556 |
| C20-pH 4bt | 7.6438 | 8.1410 |
| C20-pH 4.25at | | |
| C20-pH 4.25bt | | |
| C20-pH 4.5at | | |
| C20-pH 4.5bt | | |
| C20-pH 4.75at | | |
| C20-pH 4.75bt | | |
| C20-pH 5at | | |
| C20-pH 5bt | | |
| C20-pH 5.25at | 7.5929 | 8.0867 |
| C20-pH 5.25bt | 7.6278 | 8.1264 |
| C20-pH 5.5at | | |
| C20-pH 5.5bt | | |
| C20-pH 5.75at | | |
| C20-pH 5.75bt | | |
| C20-pH 6at | 7.6433 | 8.1408 |
| C20-pH 6bt | 7.6186 | 8.1164 |
| C20-pH 6.25at | | |
| C20-pH 6.25bt | | |
| C20-pH 6.5at | | |
| C20-pH 6.5bt | | |
| C20-pH 6.75at | 7.6372 | 8.1364 |
| C20-pH 6.75bt | 7.6153 | 8.1174 |
| C20-pH 7at | | |
| C20-pH 7bt | | |
| C20-pH 7.25at | 7.6345 | 8.1344 |
| C20-pH 7.25bt | 7.5776 | 8.0789 |
| C20-pH 7.5at | | |
| C20-pH 7.5bt | | |
| C20-pH 7.75at | 7.6467 | 8.1569 |
| C20-pH 7.75bt | 7.6119 | 8.1139 |

| | | |
|---------------|--------|--------|
| C20-pH 8at | 7.6342 | 8.1303 |
| C20-pH 8bt | 7.5945 | 8.0923 |
| C20-pH 8.25at | 7.6363 | 8.1335 |
| C20-pH 8.25bt | 7.6486 | 8.1471 |
| C20-pH 8.5at | 7.5842 | 8.0795 |
| C20-pH 8.5bt | 7.6601 | 8.1594 |
| C20-pH 8.75at | 7.6412 | 8.1473 |
| C20-pH 8.75bt | 7.6612 | 8.1582 |
| C20-pH 9at | 7.6133 | 8.1111 |
| C20-pH 9bt | 7.6120 | 8.1115 |
| C20-pH 9.25at | 7.6659 | 8.1837 |
| C20-pH 9.25bt | 7.6631 | 8.1613 |
| C20-pH 9.5at | 7.6455 | 8.1442 |
| C20-pH 9.5bt | 7.6652 | 8.1647 |

Sorption samples

| Sample Name | wt. Of vial | wt. Of vial + sample |
|--------------|-------------|----------------------|
| C20-pH 4.25a | 7.5460 | 8.0753 |
| C20-pH 4.25b | 7.6452 | 8.1503 |
| C20-pH 4.5a | 7.6118 | 8.1119 |
| C20-pH 4.5b | 7.5922 | 8.0926 |
| C20-pH 4.75a | 7.6712 | 8.1707 |
| C20-pH 4.75b | 7.5805 | 8.0786 |
| C20-pH 5a | 7.6656 | 8.1659 |
| C20-pH 5b | 7.5172 | 8.0173 |
| C20-pH 5.25a | 0.0000 | 0.0000 |
| C20-pH 5.25b | 0.0000 | 0.0000 |
| C20-pH 5.5a | 7.6624 | 8.1630 |
| C20-pH 5.5b | 7.6423 | 8.1430 |
| C20-pH 5.75a | 7.6882 | 8.1875 |
| C20-pH 5.75b | 7.6221 | 8.1243 |
| C20-pH 6a | 0.0000 | 0.0000 |
| C20-pH 6b | 0.0000 | 0.0000 |
| C20-pH 6.25a | 7.6310 | 8.1323 |
| C20-pH 6.25b | 7.6888 | 8.1897 |
| C20-pH 6.5a | 7.5834 | 8.0858 |
| C20-pH 6.5b | 7.6521 | 8.1536 |
| C20-pH 6.75a | 0.0000 | 0.0000 |
| C20-pH 6.75b | 0.0000 | 0.0000 |
| C20-pH 7a | 7.5889 | 8.0894 |
| C20-pH 7b | 7.6313 | 8.1314 |
| C20-pH 7.25a | 0.0000 | 0.0000 |
| C20-pH 7.25b | 0.0000 | 0.0000 |
| C20-pH 7.5a | 7.6134 | 8.1137 |
| C20-pH 7.5b | 7.5638 | 8.0650 |

| Sample Name | Container wt. before sampling | wt. Of PP tubes before sampling | Container wt. after sampling | wt. Of PP tubes after sampling |
|--------------|-------------------------------|---------------------------------|------------------------------|--------------------------------|
| C20d-pH 2.5 | 52.6282 | 20.6676 | 51.6179 | 19.6623 |
| C20d-pH 2.75 | 50.4222 | 23.3043 | 49.4148 | 22.3028 |
| C20d-pH 3 | 53.2363 | 20.5342 | 52.2300 | 19.5332 |
| C20d-pH 3.25 | 52.8689 | 20.0664 | 51.7831 | 19.0651 |
| C20d-pH 3.5 | 52.0348 | 20.8321 | 51.0243 | 19.8304 |
| C20d-pH 3.75 | 52.0518 | 21.1280 | 51.0433 | 20.1276 |
| C20d-pH 4 | 52.5956 | 21.0889 | 51.5908 | 20.0902 |
| C20d-pH 4.25 | | | | |
| C20d-pH 4.5 | | | | |
| C20d-pH 4.75 | | | | |
| C20d-pH 5 | | | | |
| C20d-pH 5.25 | 52.3222 | 21.0698 | 51.3124 | 20.0695 |
| C20d-pH 5.5 | 49.8450 AJ | | | |
| C20d-pH 5.75 | | | | |
| C20d-pH 6 | 52.8024 | 20.8991 | 51.7944 | 19.8895 |
| C20d-pH 6.25 | | | | |
| C20d-pH 6.5 | | | | |
| C20d-pH 6.75 | 53.1398 | 20.4318 | 52.1336 | 19.4274 |
| C20d-pH 7 | | | | |
| C20d-pH 7.25 | 54.0721 | 19.3719 | 53.0620 | 18.3173 |
| C20d-pH 7.5 | | | | |
| C20d-pH 7.75 | 54.1754 | 19.3550 | 53.1684 | 18.3545 |
| C20d-pH 8 | 53.4990 | 19.9702 | 52.4896 | 18.9692 |
| C20d-pH 8.25 | 52.6016 | 20.7850 | 51.5926 | 19.7862 |
| C20d-pH 8.5 | 53.8669 | 19.6029 | 52.8574 | 18.6023 |
| C20d-pH 8.75 | 53.1460 | 19.6676 | 52.1362 | 18.6656 |
| C20d-pH 9 | 52.3440 | 20.8067 | 51.3349 | 19.8061 |
| C20d-pH 9.25 | 53.5005 | 20.1074 | 52.4921 | 19.1060 |
| C20d-pH 9.5 | 53.9872 | 20.7079 | 52.9765 | 19.7075 |

The samples were kept under glass cover away from light for 24 hrs before counting.

8/17/98 AJ

8/18/98

① LSA counting started. The results are posted on page 32.

② Cleaned agate mortar & pestle, 325 mesh for quartz crushing to 325 mesh.

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

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.20 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.71 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.61 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS F |
|--------------------|--------|-------------|---------------|--------------|--------|
| 1 | 999.99 | 20.20 1.41 | 3.705 3.29 | 29.61 1.16 | 149.56 |
| 2 | 0.89 | 15.76 80.70 | 5036.745 2.99 | 5051.29 2.99 | 628.05 |
| 3 | 0.91 | 50.13 35.08 | 4921.569 2.99 | 4978.08 2.98 | 620.07 |
| 4 | 0.94 | 26.61 53.05 | 4748.422 2.99 | 4781.03 2.99 | 625.53 |
| 5 | 0.95 | 22.96 58.73 | 4691.032 3.00 | 4717.76 3.00 | 624.42 |
| 6 | 0.97 | 44.75 36.58 | 4799.388 2.93 | 4844.62 2.93 | 623.35 |
| 7 | 0.93 | 18.51 69.72 | 4793.069 3.00 | 4813.40 3.00 | 626.02 |
| 8 | 1.68 | 11.94 73.28 | 2654.033 3.00 | 2668.01 3.00 | 625.45 |
| 9 | 1.70 | 16.86 55.41 | 2621.589 3.00 | 2638.63 3.00 | 623.14 |
| 10 | 1.21 | 13.69 77.37 | 3693.815 2.99 | 3710.06 3.00 | 626.11 |
| 11 | 1.21 | 7.07 134.3 | 3671.501 3.00 | 3677.00 3.01 | 626.69 |
| 12 | 2.08 | 5.76 122.7 | 2137.160 3.00 | 2144.43 3.02 | 623.27 |
| 13 | 2.03 | 1.48 443.3 | 2191.862 3.00 | 2191.58 3.02 | 625.47 |
| 14 | 2.06 | 8.44 88.41 | 2154.062 3.00 | 2161.17 3.02 | 624.71 |
| 15 | 2.01 | 14.63 56.95 | 2215.698 3.00 | 2231.59 3.01 | 622.15 |
| 16 | 1.87 | 10.28 78.58 | 2375.439 3.00 | 2389.64 3.01 | 624.28 |
| 17 | 1.88 | 15.97 54.96 | 2360.657 3.00 | 2379.44 3.01 | 625.49 |
| 18 | 1.64 | 6.02 132.9 | 2724.953 2.99 | 2734.42 3.00 | 626.20 |
| 19 | 1.60 | 27.30 39.93 | 2775.670 3.00 | 2803.52 3.00 | 619.79 |
| 20 | 1.26 | 28.21 43.95 | 3547.089 2.99 | 3577.54 2.99 | 623.03 |
| 21 | 1.27 | 8.93 107.3 | 3504.956 3.00 | 3516.85 3.01 | 624.13 |
| 15 MISSING TUBE(S) | | | | | |
| 37 | 1.31 | 21.02 53.38 | 3394.768 3.00 | 3422.30 3.00 | 611.66 |
| 38 | 1.33 | 9.88 96.34 | 3339.904 3.00 | 3356.11 3.01 | 612.77 |
| 39 | 1.14 | 25.41 49.79 | 3903.313 3.00 | 3932.67 3.00 | 610.35 |
| 40 | 1.13 | 18.74 62.67 | 3951.162 2.99 | 3971.28 3.00 | 615.25 |
| 41 | 1.28 | 12.61 80.31 | 3491.607 2.99 | 3511.02 3.00 | 612.04 |
| 42 | 1.32 | 8.59 108.8 | 3375.840 3.00 | 3389.33 3.00 | 616.05 |
| 43 | 1.27 | 14.45 72.34 | 3501.807 3.00 | 3519.21 3.00 | 611.00 |
| 44 | 1.28 | 19.64 56.82 | 3491.607 2.99 | 3515.71 2.99 | 610.48 |
| 45 | 1.13 | 16.08 70.48 | 3935.233 3.00 | 3953.58 3.00 | 619.08 |
| 46 | 1.09 | 14.66 77.16 | 4078.864 3.00 | 4094.25 3.00 | 614.90 |
| 47 | 0.61 | 25.70 67.51 | 7338.917 2.99 | 7368.75 2.99 | 613.31 |
| 48 | 0.63 | 38.53 50.12 | 7159.787 2.98 | 7205.31 2.97 | 612.05 |
| 49 | 0.33 | 64.65 49.61 | 13875.08 2.96 | 13949.2 2.95 | 612.03 |
| 50 | 0.33 | 31.32 79.80 | 13726.60 2.97 | 13770.4 2.97 | 614.46 |
| 51 | 0.41 | 11.51 152.9 | 10993.86 2.98 | 11009.4 2.98 | 616.13 |
| 52 | 0.39 | 13.13 140.8 | 11611.68 2.97 | 11642.2 2.97 | 615.50 |
| 53 | 0.42 | 51.23 50.92 | 10634.39 2.99 | 10689.4 2.99 | 612.60 |
| 54 | 0.42 | 15.52 118.9 | 10848.68 2.96 | 10868.0 2.96 | 614.02 |
| 55 | 0.44 | 22.98 86.22 | 10334.93 2.97 | 10372.7 2.96 | 616.31 |

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS F |
|--------------------|------|-------------|---------------|--------------|--------|
| 56 | 0.44 | 25.26 80.50 | 10107.66 3.00 | 10129.5 3.00 | 612.62 |
| 57 | 0.48 | 27.72 72.10 | 9312.962 2.99 | 9349.56 2.99 | 613.74 |
| 58 | 0.48 | 58.97 43.56 | 9385.879 2.98 | 9451.64 2.97 | 612.40 |
| 59 | 0.84 | 40.52 41.97 | 5322.485 2.99 | 5373.96 2.98 | 608.69 |
| 60 | 0.89 | 14.63 85.53 | 5018.767 2.99 | 5047.92 2.99 | 612.06 |
| 61 | 1.05 | 14.09 81.16 | 4242.962 3.00 | 4265.63 3.00 | 612.67 |
| 62 | 1.05 | 6.47 155.9 | 4257.248 2.99 | 4268.49 3.00 | 612.36 |
| 63 | 1.26 | 4.40 200.8 | 3542.327 3.00 | 3548.96 3.00 | 616.44 |
| 64 | 1.23 | 12.32 83.50 | 3632.067 2.99 | 3641.94 3.00 | 614.23 |
| 65 | 1.53 | 9.21 95.23 | 2911.328 3.00 | 2927.26 3.00 | 613.42 |
| 66 | 1.51 | 24.83 44.00 | 2951.262 3.00 | 2984.96 2.99 | 611.47 |
| 67 | 1.42 | 13.60 71.76 | 3132.210 3.00 | 3149.27 3.00 | 614.04 |
| 68 | 1.52 | 15.33 63.11 | 2941.032 2.99 | 2959.87 3.00 | 613.75 |
| 69 | 1.38 | 13.13 74.87 | 3231.802 3.00 | 3247.20 3.00 | 617.51 |
| 70 | 1.36 | 19.51 55.42 | 3278.648 3.00 | 3304.95 3.00 | 616.82 |
| 71 | 1.43 | 17.56 58.54 | 3118.673 3.00 | 3137.53 3.00 | 618.02 |
| 72 | 1.46 | 7.88 111.3 | 3050.405 3.00 | 3060.12 3.01 | 621.99 |
| 73 | 1.40 | 22.66 48.85 | 3170.581 3.00 | 3192.54 3.01 | 624.07 |
| 74 | 1.42 | 19.94 53.34 | 3131.506 3.00 | 3158.42 3.00 | 617.59 |
| 16 MISSING TUBE(S) | | | | | |
| 91 | 0.88 | 25.26 56.93 | 5058.795 3.00 | 5085.17 3.00 | 615.89 |
| 92 | 0.92 | 22.19 61.19 | 4864.773 2.99 | 4891.04 2.99 | 616.70 |
| 93 | 0.93 | 20.66 64.18 | 4797.370 3.00 | 4820.93 3.00 | 616.43 |
| 94 | 0.94 | 25.55 54.63 | 4745.231 3.00 | 4770.39 3.00 | 618.82 |
| 95 | 0.91 | 21.56 62.86 | 4902.888 3.00 | 4927.54 3.00 | 618.15 |
| 96 | 0.94 | 22.35 60.21 | 4759.061 2.99 | 4783.16 2.99 | 617.58 |
| 97 | 0.93 | 18.51 69.72 | 4804.897 2.99 | 4823.08 3.00 | 619.47 |
| 98 | 0.95 | 24.01 56.83 | 4688.927 3.00 | 4713.55 3.00 | 617.65 |
| 99 | 0.93 | 13.13 91.19 | 4812.424 2.99 | 4831.68 2.99 | 619.38 |
| 100 | 0.96 | 15.22 79.85 | 4662.962 2.99 | 4679.77 2.99 | 619.30 |
| 101 | 1.11 | 5.93 163.8 | 4000.799 3.00 | 4008.23 3.01 | 621.07 |
| 102 | 1.12 | 3.91 237.5 | 3973.973 3.00 | 3982.89 3.01 | 625.86 |
| 103 | 1.83 | 19.69 47.44 | 2426.350 3.00 | 2447.44 3.01 | 618.37 |
| 104 | 1.78 | 9.01 89.94 | 2500.789 3.00 | 2514.77 3.01 | 622.16 |
| 105 | 1.51 | 3.64 218.4 | 2948.613 3.00 | 2959.13 3.01 | 621.52 |
| 106 | 1.58 | 15.24 62.17 | 2996.928 2.91 | 3015.33 2.91 | 623.90 |
| 107 | 1.35 | 12.39 79.33 | 3296.295 3.00 | 3313.36 3.00 | 621.51 |
| 108 | 1.37 | 9.00 102.7 | 3257.609 3.00 | 3266.74 3.00 | 622.32 |
| 109 | 1.30 | 8.26 113.3 | 3430.910 3.00 | 3441.93 3.00 | 619.34 |
| 110 | 1.29 | 13.13 77.44 | 3462.187 2.99 | 3477.37 3.00 | 621.75 |
| 111 | 1.20 | 4.80 190.2 | 3707.128 3.00 | 3713.73 3.01 | 619.85 |
| 112 | 1.19 | 7.53 128.2 | 3761.001 2.99 | 3772.07 3.00 | 621.71 |
| 113 | 0.98 | 20.62 62.62 | 4559.560 2.99 | 4582.64 2.99 | 619.10 |
| 114 | 1.00 | 19.80 63.90 | 4441.295 3.00 | 4464.39 3.00 | 618.09 |
| 115 | 0.94 | 10.65 107.6 | 4751.614 2.99 | 4760.82 3.00 | 620.59 |
| 116 | 0.98 | 15.52 77.84 | 4571.805 2.99 | 4587.74 2.99 | 620.06 |
| 117 | 0.93 | 12.06 97.71 | 4790.918 3.00 | 4800.50 3.00 | 623.64 |
| 118 | 0.93 | 32.49 46.34 | 4791.994 3.00 | 4830.61 2.99 | 620.97 |
| 119 | 0.93 | 24.96 55.84 | 4804.897 2.99 | 4828.46 2.99 | 622.55 |
| 120 | 0.92 | 24.37 57.14 | 4849.556 3.00 | 4875.83 3.00 | 625.27 |
| 121 | 0.91 | 36.94 42.91 | 4919.372 2.99 | 4960.50 2.99 | 611.70 |
| 122 | 0.91 | 23.76 58.52 | 4908.383 2.99 | 4940.72 2.99 | 622.20 |
| 123 | 0.94 | 7.46 145.5 | 4777.146 2.99 | 4785.29 2.99 | 632.37 |
| 124 | 0.94 | 11.72 99.50 | 4747.359 3.00 | 4760.82 3.00 | 629.13 |
| 125 | 0.93 | 29.26 49.85 | 4822.102 2.99 | 4851.04 2.99 | 629.20 |
| 126 | 0.93 | 31.41 47.44 | 4796.295 3.00 | 4827.38 2.99 | 629.02 |

18 Aug 98 06:32

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLA |
|-----|------|-------------|------------|--------------|-------------|
| 127 | 1.00 | 12.80 89.78 | 4797.295 | 2.89 4810.39 | 2.89 643.92 |
| 128 | 0.94 | 39.38 40.44 | 4753.742 | 2.99 4792.73 | 2.99 633.26 |

8/18/98

AJ

① Following procedure on page (4). other mineral impurities were removed from quartz by heavy liquid separation

② Prepare containers (50 ml) & teflon 2lt. bottles for mixed mineral sorption experiments.

8/21/98

AJ

① Crush quartz (60/100 fraction) to 325 mesh.

② Continue heavy liquid separation to remove impurities from quartz.

③ Preparation of 0.1M NaNO_3 matrix

16.9968 gms of NaNO_3 (lot # 930601) in 2000 gms of nanopure H_2O .

④ Preparation of 50 PPb U-233 solution

Dilute 200.0 g of 500 PPb soln. (Spike # 28A) to 2000.0 g of with 0.1M NaNO_3 matrix.

8/24/98

AJ

① Crush quartz (60/100 fraction) to 325 mesh.

② BET surface analysis started on Clinoptilolite using Coulter 3100 SA.

wt. of tube assembly = 33.6212 g

wt. of clinoptilolite added = 1.9905 g

The sample was outgassed for minutes.

8/25/98 AJ

① wt. of outgassed clinoptilolite assembly = 35.3158

wt. of outgassed clinoptilolite = 35.3158
- 33.6212

1.6946 g

The results are stored in binder marked as "Coulter 3100 SA".

② Sampling for desorption step on samples which were adjusted for PH. The samples were kept under glass cover for 24 hrs before counting.

8/26/98 AJ

① BET surface analysis started on Clinoptilolite using Coulter 3100 SA.

wt. of tube assembly = 33.6328 g

wt. of Clinop. added = 2.5052 g.

The sample was outgassed for minutes.

② LSA run started.

8/27/98 AJ

① wt. of outgassed Clinop. + assembly = 35.7470 g

wt. of outgassed Clinop. = 35.7470

- 33.6328

2.1142 g

The run was completed & results are stored in binder marked as "Coulter 3100 SA surface analysis results".

② LSA results are recorded in following table.

27 Aug 98 05:41 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.18 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.80 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.79 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

PP Tubes

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS F |
|----|--------|------------|------------|------------|--------------------------|
| 1 | 999.99 | 20.18 | 1.41 | 3.798 | 3.25 29.79 1.16 150.93 |
| 2 | 0.86 | 32.15 | 48.54 | 5192.714 | 2.99 5228.35 2.99 610.77 |
| 3 | 0.86 | 20.52 | 67.07 | 5204.342 | 2.99 5224.86 2.99 611.70 |
| 4 | 0.73 | 19.55 | 75.49 | 6153.736 | 2.98 6175.69 2.99 608.42 |
| 5 | 0.73 | 12.70 | 105.7 | 6153.736 | 2.98 6170.21 2.99 610.57 |
| 6 | 0.73 | 27.77 | 58.38 | 6085.243 | 3.00 6120.89 3.00 607.24 |
| 7 | 0.73 | 15.44 | 90.51 | 6126.339 | 2.99 6141.44 2.99 608.45 |
| 8 | 0.33 | 46.49 | 61.15 | 13756.81 | 2.97 13815.7 2.97 608.59 |
| 9 | 0.34 | 50.41 | 57.17 | 13313.85 | 2.97 13379.0 2.97 613.16 |
| 10 | 0.54 | 16.86 | 98.27 | 8266.572 | 2.99 8283.17 3.00 611.59 |
| 11 | 0.54 | 53.89 | 43.47 | 8294.350 | 2.99 8347.99 2.98 610.32 |
| 12 | 0.31 | 24.98 | 96.63 | 14425.23 | 2.99 14463.8 2.99 611.33 |
| 13 | 0.32 | 54.82 | 55.85 | 14064.95 | 2.98 14132.7 2.98 611.57 |
| 14 | 0.35 | 36.96 | 69.14 | 12827.63 | 2.99 12867.4 2.98 609.85 |
| 15 | 0.36 | 35.38 | 70.24 | 12515.65 | 2.98 12548.0 2.98 611.97 |
| 16 | 0.28 | 54.82 | 59.71 | 16039.06 | 2.98 16098.8 2.98 606.19 |
| 17 | 0.29 | 38.44 | 73.97 | 15765.17 | 2.96 15821.9 2.96 609.21 |
| 18 | 0.42 | 36.96 | 63.12 | 10786.68 | 2.97 10825.0 2.97 606.82 |
| 19 | 0.42 | 22.68 | 89.10 | 10593.82 | 3.00 10622.6 3.00 610.08 |
| 20 | 0.47 | 28.76 | 70.97 | 9570.670 | 2.98 9595.74 2.98 609.40 |
| 21 | 0.47 | 30.88 | 67.50 | 9466.415 | 3.00 9497.87 3.00 610.49 |

| 15 MISSING TUBE(S) | containers |
|--------------------|--|
| 37 | 1.03 15.74 75.05 4321.445 3.00 4345.94 3.00 620.46 |
| 38 | 1.03 10.89 100.9 4321.445 3.00 4333.31 3.00 617.95 |
| 39 | 1.06 11.90 92.51 4195.259 3.00 4206.06 3.01 618.33 |
| 40 | 1.02 10.21 106.9 4365.810 3.00 4377.07 3.00 616.72 |
| 41 | 1.04 14.44 79.95 4277.933 3.00 4295.21 3.00 621.05 |
| 42 | 1.04 12.51 89.64 4274.087 3.00 4285.59 3.01 614.86 |
| 43 | 1.80 10.93 76.10 2473.980 3.00 2489.10 3.01 619.73 |
| 44 | 1.77 13.72 63.83 2517.106 3.00 2532.35 3.00 614.95 |
| 45 | 1.28 5.60 160.3 3489.952 2.99 3498.33 3.00 617.30 |
| 46 | 1.30 9.05 104.8 3441.587 2.99 3456.36 3.00 616.70 |
| 47 | 2.40 190.65 9.83 1853.702 3.00 2047.29 2.87 546.08 |
| 48 | 2.06 14.77 55.80 2153.483 3.01 2172.64 3.01 615.65 |
| 49 | 2.09 0.87 727.5 2122.518 3.01 2124.27 3.02 617.25 |
| 50 | 2.16 10.38 72.55 2062.406 3.00 2071.60 3.01 614.66 |
| 51 | 1.86 5.63 132.5 2387.062 3.00 2394.94 3.02 615.85 |
| 52 | 1.89 5.22 140.6 2354.932 3.00 2359.10 3.01 616.95 |
| 53 | 1.74 9.71 85.45 2563.443 3.00 2575.95 3.00 617.18 |
| 54 | 1.69 12.37 71.02 2626.971 3.00 2640.62 3.01 617.11 |
| 55 | 1.33 13.66 73.90 3363.119 2.99 3378.48 3.00 615.91 |

27 Aug 98 06:34 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FL |
|----|------|------------|------------|------------|--------------------------|
| 56 | 1.39 | 9.32 | 98.93 | 3304.116 | 2.95 3316.97 2.96 617.37 |

8/31/98 AJ

- ① Crush quartz 60/100 fraction to <325 mesh.
- ② Containers dried in oven at 75°C.

9/1/98 AJ

- ① Preparation of heavy liquid for removing impurities from quartz.

$$\rho_{\text{tetrabromoethane}} = 2.9672 \text{ g/c.c.}$$

$$\rho_{\text{quartz}} = 2.65 \text{ g/c.c.}$$

$$\rho_{\text{N-N dimethyl formamide}} = 0.93445 \text{ g/c.c.}$$

liquid to be prepared with $\rho_{\text{liquid}} = 2.75 \text{ g/c.c.}$
Using procedure for heavy liquid separation on page (4), for 150 ml of tetrabromoethane, ?? of N-N dimethyl formamide is required

$$V_1 = \frac{(2.9672 - 2.75)}{2.75 - 0.93445} 150 \text{ ml}$$

$$= \frac{0.217200}{1.81555} \times 150 \text{ ml}$$

$$= 0.11963317 \times 150 \text{ ml}$$

$$= 17.945 \text{ ml of N-N dimethyl.}$$

Mix 150 ml of tetrabromoethane to 17.95 ml of N-N dimethyl formamide & store in dark color bottle.

A batch of 200 ml was prepared by mixing 200 ml of tetrabromoethane to 23.4 ml of N-N dimethyl formamide.

- ② Crush quartz 60/100 fraction to ≤ 325 mesh.

9/2/98 AJ

- ① Crush quartz 60/100 fraction to ≤ 325 mesh

- ② with agate mortar & pestle & Ro-Tap sieve shaker.

- ② Treat quartz with heavy liquid for removal of another mineral impurities. Use procedure on page (5).

9/3/98 AJ

- ① Crush 60/100 fraction of quartz to ≤ 325 mesh using mortar & pestle & Ro-Tap sieve shaker.

- ② Treat quartz with heavy liquid for removal of mineral impurities. Followed procedure on page (5).

- ③ Clean ≤ 325 mesh quartz ultrasonically with nanopure water. Centrifuge at 1500 rpm for 1 min to remove particles $< 0.044 \text{ mm}$.

9/4/98 AJ

- ① Continue heavy liquid treatment of quartz. Refer to page (5).

- ② Crush quartz to ≤ 325 mesh with agate mortar & pestle.

- ③ 45 g of quartz ≤ 325 mesh dried in oven at 80°C .

9/8/98 AJ

- ① Crush quartz to 325 mesh with agate mortar & pestle & Ro-Tap sieve shaker.

- ② Rinse 60/100 quartz after HL treatment with nanopure H_2O several times & also in ultrasonic cleaner. Kept in oven for drying at $\sim 80^\circ\text{C}$.

9/9/98 AJ

- ① Crush quartz to 325 mesh using agate mortar & pestle & Ro-Tap sieve shaker.
- ② Detection of Al & SiO₂ in solution of C20-U experiments. The procedure is mentioned in TOP-013 & TOP-014 & followed to obtain the following results:-

SiO₂ and Al solution concentrations
Measurement of solutions from desorption containers.

| exp soln | equilib. pH | SiO ₂ ppm | Al ppb |
|----------|----------------|-------------------------|-----------|
| pH-2.5 | 2.45 | 5.83 | 2739 |
| pH-2.75 | 2.68 | | |
| pH-3 | 2.98 | 2.3 | 1028 |
| pH-3.25 | 3.34 | | |
| pH-3.5 | 3.80 | 1.09 | 259 |
| pH-3.75 | 4.96 | | |
| pH-4 | 7.00 | 1.17 | 171 |
| pH-4.25 | 4.47 | | |
| pH-4.5 | 4.56 | 1.61 | 117 |
| pH-4.75 | 4.54 | | |
| pH-5 | 5.73 | 1.4 | 67 |
| pH-5.25 | 7.24 | | |
| pH-5.5 | 5.17 | 1.48 | 72 |
| pH-5.75 | 6.30 | | |
| pH-6 | 7.45 | 1.16 | 126 |
| pH-6.25 | 6.55 | | |
| pH-6.5 | 6.88 | 1.32 | 58 |
| pH-6.75 | 7.52 | | |
| pH-7 | 7.10 | 1.44 | 80 |
| pH-7.25 | 7.76 | | |
| pH-7.5 | 7.48 | 1.56 | 60 |
| pH-7.75 | 7.96 | | |
| pH-8 | 8.15 | 1.93 | 154 |
| pH-8.25 | 8.31 | | |
| pH-8.5 | 8.62 | 2.37 | 102 |
| pH-8.75 | 8.80 | | |
| pH-9 | 9.01 | 2.74 | 41 |
| pH-9.25 | 9.21 | | |
| pH-9.5 | 9.17 | 3.24 | 0 |

Al std. Soln.
Lot # 981033-18

SiO₂ std. Soln.
Lot # 8037-5

9/10/98 AJ

- ① Crush quartz to ≤325 mesh using agate mortar & pestle & Ro-Tap sieve shaker.

9/11/98 AJ

- ① Crush 60/100 fraction of quartz to ≤325 mesh using agate mortar & pestle & Ro-Tap sieve shaker.

9/14/98 AJ

- ① Crush quartz to ≤325 mesh size using agate mortar & pestle & Ro-Tap sieve shaker.

9/16/98 AJ

- ① Crush quartz to ≤325 mesh size using agate mortar & pestle & Ro-Tap sieve shaker.
- ② Rinse 60/100 quartz with acetone & then water. Kept in oven to dry at ~80°C.

9/17/98

- ① Crush quartz to ≤325 mesh size using agate mortar & pestle & Ro-Tap sieve shaker.

9/18/98

Crush 60/100 fraction pretreated quartz to ≤325 mesh size using agate mortar & pestle and Ro-Tap sieve shaker.

9/21/98

- ① Crush 60/100 fraction pretreated quartz to 325 mesh size using agate mortar & pestle and Ro-Tap sieve shaker. Total quartz is ~ 170 g.

- ② Divide 170 g of cleaned 325 mesh quartz into two beakers & clean ultrasonically with DI water 2 times.

9/22/98 AJ

Objective:- To remove particles $< 0.004 \text{ mm}$ from 325 mesh fraction of quartz.

Procedure:- Aliquot $\sim 2 \text{ g}$ of quartz in 50 ml PP tubes. Add 30-40 ml of H_2O & centrifuge at 1500 rpm for 1 min. Decant the supernatant liquid & discard till it is clear.

Equipments needed:- PP tubes
Table top centrifuge
(Marathon 211C)
Nanopure water
glasswares.
Oven

Using the above procedure quartz was treated for removal of particles $< 0.004 \text{ mm}$ centrifuged 2 times. Quartz was kept in oven at $\sim 82^\circ\text{C}$ for overnight to dry.

9/23/98 AJ

9/24/98

- ① Weigh quartz which was dried overnight $\approx 140 \text{ g}$.

Total = 45 g (previous batch) + 140 g
= 185 g

- ② Objective:- To analyse surface area of 325 mesh fraction of quartz prepared.

Equipments needed:- ① Coulter SA3100
② Quartz
③ Liquid N_2
④ N_2 & He gas tanks
⑤ Weighing balance
⑥ Weighing boat

Procedure:- Followed per Coulter SA3100 product manual.

Results:- The results are filed in a ring binder marked as "Results of Surface Analysis" in the lab.

- ① wt. of tube + assembly = 33.5656 g
wt. of qz added $\approx 4.2540 \text{ g}$
outgas time = 120 min, outgas temp. = 350°C
wt. of outgassed qz + tube assembly = 37.7861 g
wt. of outgassed qz = 37.7861
- 33.5656
4.2205 g

AJ ② Sample didn't outgas for 120 min & so test was repeated.

- ② Preparation of 0.005 M NaHCO_3 soln.

Dilute 0.084 g of NaHCO_3 (Lot # 936883) with nanopure H_2O up to 200 ml mark.

- ③ wt. of tube + assembly = 33.2573 g.
wt. of sample added $\approx 2.0125 \text{ g}$
outgas time = 720 min

max^m temp = 350°C

wt. of outgassed qz + tube assembly = 35.2541g

wt. of outgassed qz = $\begin{array}{r} 35.2541 \\ - 33.2573 \\ \hline 1.9968 \end{array}$ g.

Results filed in binder marked as "Surface Area Analysis" in Geochemistry Lab.

④ wt. of tube assembly = 33.5656
outgas time = 120 min, temp. = 350°C

wt. of tube assembly + outgassed qz = 37.7864g

wt. of outgassed qz = $\begin{array}{r} 37.7864 \\ - 33.5656 \\ \hline 4.2208 \end{array}$ g

Results filed in binder marked as "Surface Area Analysis" in Geochem. Lab.

AS
8/9/25/98

AJ

BET Surface Area Analysis

① wt. of tube assembly = 33.6672 g
outgas time = 720 min, temp. = 350°C
wt. of outgassed 325 mesh qz + tube assembly = 35.6149 g

8

wt. of outgassed 325 mesh qz = $\begin{array}{r} 35.6149 \\ - 33.6672 \\ \hline 1.9477 \end{array}$ g

Results filed in ring binder

② wt. of tube assembly = 33.5713 g

outgas time = 720 min, max. temp. = 350°C

wt. of outgassed qz < 325 mesh + tube assembly = 35.5612 g

wt. of outgassed qz < 325 mesh = $\begin{array}{r} 35.5612 \\ - 33.5713 \\ \hline 1.9899 \end{array}$ g

Results filed in binder named as "Surface Area Analysis" in Geochemistry Lab.

③ wt. of tube assembly = 33.3789 g

outgas time = 720 min, max^m temp. = 350°C

wt. of outgassed qz (old batch) + tube assembly = 35.3897 g

wt. of outgassed qz (old batch) = $\begin{array}{r} 35.3897 \\ - 33.3789 \\ \hline 2.0108 \end{array}$ g

Results filed in binder named as "Results of Surface Area Analysis" in Geochem. Lab.

9/28/98 AJ

Crush 60/100 fraction quartz to <325 mesh size using mortar & pestle & Ro-Tap sieve shaker.
≈ 26 g.

9/29/98 AJ

Crush 60/100 fraction quartz to <325 mesh size using igate mortar & pestle & Ro-Tap sieve shaker.

$\approx 55g$.
clean with nanopure water in ultrasonic cleaner.

9/30/98 AJ

① Centrifuge $\sim 2g$ of ≤ 325 mesh quartz with 35-40 ml of nanopure H_2O in PP tubes at 1500 rpm for 1 min. This process is necessary to remove any fine particles $< 0.004mm$.

② Prepare sample holder for surface area measurements. Wash the sample holder with nanopure water using disposable plastic pipette & dry in oven overnight.

10/1/98 AJ

① Wt of total quartz (crushed to 325 mesh & particles $< 0.004mm$ removed)
= 225.09 g.

② BET surface area analysis of quartz 325 mesh size. The procedure is in reference to Coulter SA3100 product manual in the Geochemistry lab.

wt. of tube assembly = 33.6892 g

outgas temperature = $350^\circ C$
outgas time = 120 min.

wt. of tube assembly + outgassed quartz
= 37.7102 g

wt. of outgassed quartz = 37.7102
- 33.6892
= 4.0210 g.

The results are filed in binder marked as "Results of Surface Area Analysis".

BET surface area = 0.145 m^2/g

③ Objective:- BET surface area analysis on prepared Quartz (325 mesh) and on mixture of Clinoptilolite and quartz in the following ratio:

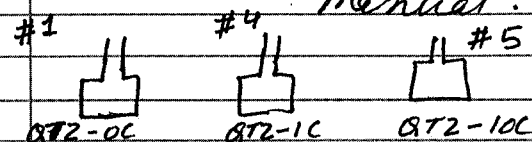
① No Clinoptilolite i.e. 0.0 mg

② Qz = 1000 mg & Clino = 3.5 mg

③ Qz = 1000 mg & Clino = 35 mg

Equipments:- ① Sample holders & tube assembly
② N_2 liquid
③ He gas tank & N_2 gas tank
④ Weighing scale & weighing boats.
⑤ Coulter SA 3100 surface area analyzer

Procedure:- As per Coulter SA3100 product manual.



① wt. of #1 tube assembly = 33.6664 g
amount of qz added ≈ 2.0191 g
amount of Clino added = 0.0 g

② wt. of #4 tube assembly = 33.6328 g

wt. of qz added = $1.00g \times 3 \approx 3.0016$ g
wt. of Clino added = $0.0035 (3.5mg) \times 3$
= 0.0105 g

$$\begin{array}{r} \text{total wt. of mixture sample} = 3.0016 \text{ g} \\ + 0.0105 \\ \hline 3.0121 \text{ g} \end{array}$$

(C) wt. of #5 tube assembly = 33.6677 g

$$\begin{array}{l} \text{wt. of g2 added} = 1.0 \text{ g} \times 2 = 2.000 \text{ g} \\ \text{wt. of clino added} = 0.0359 \text{ g} \times 2 = 0.0718 \text{ g} \end{array}$$

Outgas time for #1, #4 & #5 tubes
= 720 min \approx 12 hrs.

outgas temp. for #1, #4 & #5 tubes
= 350°C

10/2/98 AJ

(1) #1 & #4 samples had leak & therefore could not be outgassed. This is to be repeated.

(2) wt. of tube assembly (#5) & outgassed sample
= 35.4056 g.

$$\begin{array}{r} \text{wt. of outgassed sample} = 35.4056 \\ - 33.6677 \\ \hline 1.7379 \text{ g} \end{array}$$

The results of #5 tube sample showed surface area = 0.794 sq.m/g on screen but couldn't complete the run as other samples were kept to outgas. Both the runs were stopped & has to be repeated.

10/5/98 AJ

10/6/98

(1)

Objective:- To redo the surface area analysis of the samples on page 47.

#1

#4

#5

(a) wt. of tube assembly #1 = 33.6664 g.

(b) wt. of tube assembly #4 = 33.6328 g

(c) wt. of tube assembly #5 = 3.6677 g.

Procedure:- As per Coulter SA3100 product manual.

Equipments:- As on page 47.

The samples were outgassed for 720 min at 350°C.

(a) wt. of outgassed sample & tube assembly #1
= 35.6904 g.
wt. of outgassed g2 = 35.6904
- 33.6664
= 2.0240 g

BET surface area = 0.304 sq.m/g

The complete results are stored in binder marked "Results of Surface Area Analysis".

(b) wt. of outgassed sample & tube assembly #4
= 36.6155 g.

wt. of outgassed g2 & clino mixture = 36.6155
(0.1 g/L) - 33.6328

$$= 2.9827 \text{ g.}$$

BET surface area = 0.314 sq.m/g

The complete results are stored in binder marked as "Surface Area Analysis Results".

(C) wt. of outgassed sample & tube assembly #5
= 35.4041 g

$$\begin{array}{r} \text{wt. of outgassed mixture of quartz \& clino.} \\ = 35.4041 \\ - 33.6677 \\ \hline 1.7364 \text{ g.} \end{array}$$

BET surface area analysis = 0.698 sq.m/g

The complete results are stored in binder "Results of Surface Area Analysis".

(d) Objective: Surface Area Analysis for the mixture of quartz & Clinophillolite with ratio 10g/L.

$$\begin{array}{l} \text{(i) } Q_2 = 1 \text{ g} \quad , \quad \text{Clino} = 350 \text{ mg} \\ \quad \quad \quad \quad \quad \quad \quad \quad = 0.35 \text{ g} \end{array}$$

Procedure & Equipments are same as on page 47.

$$\text{wt. of tube assembly \#9} = 33.1748 \text{ g}$$

$$\text{wt. of } Q_2 \text{ added } \approx 2.008 \text{ g} \quad [1 \text{ g} \times 2]$$

$$\text{wt. of clino added } \approx 0.703 \text{ g} \quad [0.35 \times 2]$$

$$\begin{array}{l} \text{wt. of total mixture sample} = 2.008 + 0.703 \\ \quad \quad \quad \quad \quad \quad \quad \quad = 2.711 \text{ g.} \end{array}$$

(e) BET surface area analysis for quartz (325 mesh) repeated.

$$\begin{array}{l} \text{wt. of tube assembly \#7} = 33.6892 \text{ g} \\ \text{wt of } Q_2 \text{ added } \approx 2 \text{ g} \end{array}$$

(f) BET surface area analysis repeated for 0.1g/L mixture of quartz & clino.

$$\text{wt. of tube assembly \#1} = 33.6328 \text{ g.}$$

10/7/98 AJ

(1) wt. of tube assembly #9 + mixture sample outgassed
= 35.7579 g

$$\begin{array}{r} \text{wt. of outgassed mixture sample (10g/L)} \\ = 35.7579 \\ - 33.1748 \\ \hline 2.5831 \text{ g.} \end{array}$$

BET surface area = 2.943 sq.m/g

The results are filed in a binder marked as "Results of Surface Analysis".

$$\begin{array}{l} \text{(2) wt. of tube assembly \#7 and quartz outgassed} = 35.6912 \text{ g} \\ \text{wt. of outgassed quartz} = 35.6912 \\ \quad \quad \quad \quad \quad \quad \quad \quad 33.6892 \\ \quad \quad \quad \quad \quad \quad \quad \quad \hline \quad \quad \quad \quad \quad \quad \quad \quad 2.0020 \text{ g.} \end{array}$$

BET surface area = 0.306 sq.m/g

The results are filed in binder marked as "Results of Surface Area Analysis".

③ wt. of tube assembly # + mixture of outgassed 92/Clino (0.12/1) = g.

$$\begin{array}{r} \text{wt. of outgassed mixture} = 36.6142 \\ - 33.6328 \\ \hline 2.9814 \text{ g} \end{array}$$

$$\text{BET surface area} = 0.311 \text{ sq.m/g}$$

The results are filed in binder marked as "Results of surface area analysis".

10/8/98

AJ

Objective:-

① BET surface area analysis for quartz (325 mesh size) and mixtures of quartz and Clinoptilolite.

Procedure and equipments required are same as on page 47.

① wt. of tube assembly # 1 = 33.6665 g
wt. of quartz added = 2.0132 g

outgas temp. = 350°C & outgas time = 720 min.

② wt. of tube assembly # 4 = 33.5702 g
wt. of quartz added = 3.0030 g
wt. of Clinoptilolite added = 0.07 AT
0.0117 g

outgas temp. = 350°C
outgas time = 720 min

③ wt. of tube assembly # 5 = 33.3636 g
wt. of quartz added = 2.0016 g
wt. of Clinoptilolite added = 0.0709 g

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

All three samples were loaded for outgassing.

10/9/98 AJ

① wt. of tube assembly # 1 + outgassed quartz = 35.6628 g

$$\begin{array}{r} \text{wt. of outgassed quartz} = 35.6628 \\ - 33.6665 \\ \hline 1.9963 \text{ g} \end{array}$$

The samples were analyzed using procedure as per BET Coulter SA 3100 product manual.
BET surface area = 0.308 m²/g

② wt. of tube assembly # 4 + outgassed mixture of quartz & Clinoptilolite (0.12/1) = 36.5651 g

$$\begin{array}{r} \text{wt. of outgassed mixture of 92/Clino} = 36.5651 \\ - 33.5702 \\ \hline 2.9949 \text{ g} \end{array}$$

$$\text{BET surface area} = 0.329 \text{ m}^2/\text{g}$$

③ wt. of tube assembly # 5 + outgassed mixture of quartz and Clinoptilolite (0.12/1) = 35.4110 g

$$\begin{array}{r} \text{wt. of outgassed mixture of 92/Clino} \\ = 35.4110 \\ - 33.3636 \\ \hline 2.0474 \text{ g} \end{array}$$

$$\text{BET surface area} = 0.597 \text{ m}^2/\text{g}$$

10/12/98 AJ

Objective:- BET surface area analysis of Clinoptilolite and quartz & Clinoptilolite mixtures.

Procedure and equipments needed are same as on page 47.

- ① wt. of tube assembly #4 = 33.5719 g
wt. of Clinoptilolite added = 1.9510 g

outgas time = 720 min. outgas temp. = 350°C

- ② wt. of tube assembly #5 = 33.2866 g
wt. of quartz added = 2.0000 g
wt. of Clinoptilolite added = 0.0718 g

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

- ③ wt. of tube assembly #9 = 33.2354 g.
wt. of quartz added = 2.0036 g
wt. of Clinop added = 0.7086 g

outgas time = 720 min. outgas temp. = 350°C

10/13/98 AJ

- ① wt. of tube assembly #4 + outgassed Clinoptilolite = 35.2374 g

wt. of outgassed Clinoptilolite = 35.2374
33.5719
1.6655 g

BET surface area = 11.818 m²/g

- ② wt. of tube assembly #5 + outgassed O₂/Clinop mixture = 35.3287 g (18/L)

wt. of outgassed mixture O₂/Clinop = 35.3287
- 33.2866
2.0421 g

BET surface area = 0.588 m²/g

- ③ wt. of tube assembly #9 + outgassed Clinop & O₂ mixture = 35.8269 g (10 g/L)

wt. of outgassed O₂/Clinop mixture = 35.8269
- 33.2354
2.5915 g

BET surface area = 2.970 m²/g

- ④ Mixed mineral sorption experiments started (Refer Pg. 57) 57
10/14/98 AJ AJ

Objective:- BET surface area analysis on Clinoptilolite (PCDV), surfactant added Clinoptilolite (HDTMA-PCDV) and mixture of quartz & Clinoptilolite in ratio, O₂ = 1 g & Clinoptilolite = 350 mg (0.350 g)

procedure & equipments needed are same as on page 47.

- ① wt. of tube assembly #5 = 33.3624 g.
wt. of PCDV added = 2.0045 g.
outgas temp. = 350°C & outgas time = 720 min.

- ② wt. of tube assembly #7 = 33.6844 g
wt. of HDTMA-PCDV added = 1.6974 g
outgas temp. = 350°C & outgas time = 720 min.

- (3) wt. of tube assembly #9 = 33.1803 g
 wt. of Clinoptilolite added = ~~2.0003~~^{AT} 0.7020 g
 wt. of quartz added = 2.0003 g

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

10/15/98 AJ

- (1) wt. of tube assembly #5 + outgassed PCDV
 = 35.0995 g
 wt. of outgassed PCDV = 35.0995
 - 33.3624
 1.7371 g.

BET SA = 18.827 m²/g

- (2) wt. of tube assembly #7 + outgassed HDTMA-PCDV
 = 35.1506 g
 wt. of outgassed PCDV (HDTMA) = 35.1506
 - 33.6844
 1.4662 g

BET SA = 76.692 m²/g

- (3) wt. of tube assembly #9 + outgassed mixture of
 quartz and clinoptilolite = 35.7646 g
 wt. of outgassed qz/Clinop = 35.7646
 - 33.1803
 2.5843 g

BET SA = 2. m²/g

- (4) wt. of tube assembly #1 = 33.5880 g
 wt. of PCDV added from different container
 = 1.7506 g
 outgas temp. = 350°C & outgas time = 720 min.

- (5) wt. of tube assembly #4 = 33.5722 g
 wt. of qz added = 2.1553 g
 wt. of Clinop added = 0.0756 g

Quartz only, unapped

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25°C

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma U = 50$ ppb (2.14×10^{-7})
 - equilibrium with atmospheric CO₂(g); pCO₂ = 10^{-3.5}
 - pH range 4.0-9.5
 - solution volume = 35 ml
 - mass of solid 1, quartz = 1.000 g
 - mass of solid 2, clinoptilolite = 0.000 g
 - M/V = 28.57 g/L

Equipment: Orion 920A pH meter and combination pH electrode
 Gyrotory shaker
 Fisher Marathon 21K centrifuge
 Mettler PM4600 and AE240 balances
 Packard 2500 TR/AB LSC
 Eppendorf micropipettes and tips
 Oxford pipettes and tips
 Repipettor for transfer of scintillation cocktail

Supplies: ≤ 325 mesh qtz AT
 50 ppb ²³³U stock solution
 polycarbonate centrifuge tubes with caps (50 ml capacity)
 polypropylene centrifuge tubes with caps (50 ml capacity)
 pH buffer solutions
 Ultima-Gold liquid scintillation cocktail
 7 ml scintillation vials
 reagent grade NaOH (lot 936883) AT
 concentrated HNO₃ (lot 983546)
 HNO₃ and NaHCO₃ or NaOH stock solutions at various concentrations
 weighing paper/boats
 deionized ultrapure water, DH₂O
 teflon beaker

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M NaNO₃ stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ²³³U stock solution to experimental containers.
- Label 26 polycarbonate centrifuge tubes (50 ml capacity) CQU-pHi (where i is the approximate pH of each solution, see Table CQU).
 - Label 1 polycarbonate centrifuge tube CQ0U-IU.
 - Weigh and record the weight of each container.
 - About 1L of ²³³U solution will be required for this experiment.
 - Add ~35 g of the 50 ppb ²³³U stock solution to each container. Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.

- Record weight of each container.

B. Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the CQ20-U-IU container.
- Record weight of each sample container after addition of quartz.

- Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaOH to each experimental container as listed in Table CQU. Take care to add HNO_3 or NaOH in the concentrations specified in Table CQU.
- For sample CQ0U-IU (not listed in Table CQU) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

- Sample CQ0U-IU for initial [U].

- Label two 7 ml LSC vials as CQ0U-IUa and CQ0U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from CQ20-U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container CQ0U-IU

- Wait for equilibrium.

- Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
- Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.

- Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ0U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

- Transfer quartz for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., CQ0dU-pH3 for sample container CQ0U-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw quartz from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO_3 to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

C. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ02U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table CQU: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with q/clinoz (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |

Table CQU: Estimated solution pH and volumes of HNO₃ or NaOH solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with qz/clino (35 ml)
ONLY FOR CAPPED VIALS

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--------------------------------------|-------------------------------------|
| 2.5 | 0.143 | 1 |
| 2.75 | 0.080 | 1 |
| 3 | 0.045 | 1 |
| 3.25 | 0.025 | 1 |
| 3.5 | 0.143 | 0.1 |
| 3.75 | 0.080 | 0.1 |
| 4 | 0.452 | 0.01 |
| 4.25 | 0.254 | 0.01 |
| 4.5 | 0.143 | 0.01 |
| 4.75 | 0.081 | 0.01 |
| 5 | 0.045 | 0.01 |
| 5.25 | 0.025 | 0.01 |
| 5.5 | 0.014 | 0.01 |
| 5.75 | 0.073 | 0.001 |
| 6 | 0.035 | 0.001 |
| 6.25 | 0.014 | 0.001 |
| 6.5 | 0.001 | 0.001 |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use |
| 6.75 | 0.007 | 0.001 |
| 7 | 0.012 | 0.001 |
| 7.25 | 0.018 | 0.001 |
| 7.5 | 0.026 | 0.001 |
| 7.75 | 0.038 | 0.001 |
| 8 | 0.059 | 0.001 |
| 8.25 | 0.095 | 0.001 |
| 8.5 | 0.016 | 0.01 |
| 8.75 | 0.027 | 0.01 |
| 9 | 0.047 | 0.01 |
| 9.25 | 0.082 | 0.01 |
| 9.5 | 0.145 | 0.01 |
| 9.75 | 0.256 | 0.01 |
| 10 | 0.454 | 0.01 |

UNCAPPED (Sorption)

| SAMPLE NAME | Wt. Of PC container, g | Wt. Of PC container+ quartz, g | Wt. Of PC container + quartz + U soln., g | Wt. After additions of HNO ₃ and NaHCO ₃ , g |
|--------------|------------------------|--------------------------------|---|--|
| QC0U-pH 3.0 | 22.3469 | 23.3499 | 58.3571 | 58.7462 |
| QC0U-pH 3.5 | 22.416 | 23.4155 | 58.4135 | 58.5186 |
| QC0U-pH 4.0 | 22.2385 | 23.2388 | 58.2338 | 58.3053 |
| QC0U-pH 4.5 | 22.3547 | 23.3532 | 58.3683 | 58.6576 |
| QC0U-pH 4.75 | 22.3396 | 23.3365 | 58.3354 | 58.5395 |
| QC0U-pH 5.0 | 22.2663 | 23.2671 | 58.2635 | 58.5022 |
| QC0U-pH 5.25 | 22.6252 | 23.6239 | 58.6204 | 58.8786 |
| QC0U-pH 5.5 | 22.2349 | 23.2324 | 58.2501 | 58.5219 |
| QC0U-pH 5.75 | 22.4509 | 23.4505 | 58.4722 | 58.7543 |
| QC0U-pH 6.0 | 22.3244 | 23.3236 | 58.3101 | 58.6068 |
| QC0U-pH 6.25 | 22.3204 | 23.3194 | 58.3872 | 58.7054 |
| QC0U-pH 6.5 | 22.3304 | 23.3310 | 58.3419 | 58.4160 |
| QC0U-pH 6.75 | 22.3333 | 23.3316 | 58.3524 | 58.4454 |
| QC0U-pH 7.0 | 22.4125 | 23.4109 | 58.4124 | 58.5223 |
| QC0U-pH 7.25 | 22.2155 | 23.2110 | 58.2007 | 58.3426 |
| QC0U-pH 7.5 | 22.2492 | 23.2485 | 58.2375 | 58.4487 |
| QC0U-pH 7.75 | 22.5698 | 23.5697 | 58.5892 | 58.7660 |
| QC0U-pH 8.0 | 22.3028 | 23.3009 AS | 58.2892 | 58.5690 |
| QC0U-pH 8.25 | 22.3435 | 23.3385 AS | 58.3543 | 58.4692 |
| QC0U-pH 8.5 | 22.3688 | 23.3651 | 58.3678 | 58.5478 |
| QC0U-pH 8.75 | 22.2773 AS | 23.2757 | 58.2786 | 58.4603 |
| QC0U-pH 9.0 | 22.2210 | 23.2190 | 58.2303 | 58.5797 |
| QC0U-pH 9.25 | 22.5929 | 23.5918 | 58.5891 | 58.5023 |
| QC0U-pH 9.5 | 22.3097 | 23.3096 | 58.3125 | 58.6169 |
| QC0U-IU | 22.3186 | | 57.3182 | 57.4426 |

| | | |
|----------|---------------------------|------------------------------------|
| QC0U-IVa | Wt. of vial (g) 7.5825 | Wt. of vial + sample (g) 8.0873 |
| QC0U-IVb | 7.6374 | 8.1734 |

10/13/98

Table CQ20: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with q/clinoz (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2 | 0.421 0.250 + 0.150 + 0.020 | 1 |
| 2.25 | 0.235 0.150 + 0.075 + 0.010 | 1 |
| 2.5 | 0.131 0.100 + 0.020 + 0.010 | 1 |
| 2.75 | 0.072 0.050 + 0.020 | 1 |
| 3 | 0.394 0.250 + 0.020 + 0.075 + 0.050 | 0.1 |
| 3.25 | 0.209 0.100 + 0.100 + 0.010 | 0.1 |
| 3.5 | 0.106 0.075 + 0.020 + 0.010 | 0.1 |
| 3.75 | 0.047 0.025 + 0.020 | 0.1 |
| 4 | 0.071 0.050 + 0.020 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 0.075 + 0.010 | 0.005 |
| 4.5 | 0.292 0.250 + 0.020 + 0.020 | 0.005 |
| 4.75 | 0.205 0.100 + 0.075 + 0.020 + 0.010 | 0.01 |
| 5 | 0.239 0.100 + 0.100 + 0.020 + 0.020 | 0.01 |
| 5.25 | 0.260 0.250 + 0.010 | 0.01 |
| 5.5 | 0.274 0.250 + 0.025 | 0.01 |
| 5.75 | 0.286 0.250 + 0.025 + 0.010 | 0.01 |
| 6 | 0.300 0.250 + 0.050 | 0.01 |
| 6.25 | 0.322 0.250 + 0.050 + 0.020 | 0.01 |
| 6.5 | 0.071 0.050 + 0.020 | 0.05 |
| 6.75 | 0.084 0.050 + 0.025 + 0.010 | 0.05 |
| 7 | 0.106 0.075 + 0.020 + 0.010 | 0.05 |
| 7.25 | 0.145 0.100 + 0.025 + 0.020 | 0.05 |
| 7.5 | 0.214 0.100 + 0.050 + 0.025 + 0.040 | 0.05 |
| 7.75 | 0.169 0.150 + 0.020 | 0.1 |
| 8 | 0.282 0.250 + 0.020 + 0.010 | 0.1 |
| 8.25 | 0.097 0.075 + 0.020 | 0.5 |
| 8.5 | 0.174 0.100 + 0.075 | 0.5 |
| 8.75 | 0.160 0.150 + 0.010 | 1 |
| 9 | 0.305 0.200 + 0.075 + 0.020 + 0.010 | 1 |

QC0U-IV
9.25
9.5

0.825
1.200

1
1

Quartz only, Clinop = 0 g

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C.

CAPPED EXPTS.

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions:

- $\Sigma U = 50 \text{ ppb}$ (2.14×10^{-7})
- Capped containers
- pH range 3.0-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 0.000 g
- M/V = 28.57 g/L

Equipment:

- Orion 920A pH meter and combination pH electrode
- Gyratory shaker
- Fisher Marathon 21K centrifuge
- Mettler PM4600 and AE240 balances
- Packard 2500 TR/AB LSC
- Eppendorf micropipetters and tips
- Oxford pipettes and tips
- Repipettor for transfer of scintillation cocktail

Supplies:

50 ppb ^{233}U stock solution

polycarbonate centrifuge tubes with caps (50 ml capacity)

polypropylene centrifuge tubes with caps (50 ml capacity)

pH buffer solutions

Ultima-Gold liquid scintillation cocktail

7 ml scintillation vials

reagent grade NaOH (lot 976631)

concentrated HNO_3 (lot 983546)

HNO_3 and NaHCO_3 or NaOH stock solutions at various concentrations

weighing paper/boats

deionized ultrapure water, DH_2O

teflon beaker

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M NaNO_3 stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.
- Label 26 polycarbonate centrifuge tubes (50 ml capacity) CQU-pHi (where i is the approximate pH of each solution, see Table CQ0U).
 - Label 1 polycarbonate centrifuge tube CQ0U-IU.
 - Weigh and record the weight of each container.
 - About 1L of ^{233}U solution will be required for this experiment.
 - Add ~35 g of the 50 ppb ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.*

- Record weight of each container.
- B. Add quartz to sample containers.
- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the CQ20-U-IU container.
 - Record weight of each sample container after addition of quartz.
- Adjust pH of each solution by adding HNO_3 or NaHCO_3 .
 - Add the volume of HNO_3 or NaOH to each experimental container as listed in Table CQU. Take care to add HNO_3 or NaOH in the concentrations specified in Table CQU.
 - For sample CQ0U-IU (not listed in Table CQU) add 100 μl of 50/50 V/V HNO_3 to the sample container.
 - Weigh and record the weight of each sample container.
- Sample CQ0U-IU for initial [U].
 - Label two 7 ml LSC vials as CQ0U-IUa and CQ0U-IUb.
 - Add 0.5 ml of 0.02 M HNO_3 to each vial.
 - Record weight of each vial.
 - Transfer 0.5 ml of solution from CQ20-U-IU to each sample vial.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of container CQ0U-IU
- Wait for equilibrium.
 - Place caps on sample containers but tighten the caps.
 - Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate for at least 10 days.
- Sample experimental solutions for [U] and pH.
 - For each sample solution label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ0U-pH3b).
 - Add 0.5 ml of 0.02 M HNO_3 to each vial.
 - Record weight of each vial.
 - Record weight of each sample container.
 - From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of each sample container.
 - Measure pH of each experimental solution.
 - Record weight of each sample container after pH measurement.
- Transfer quartz for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., CQ0dU-pH3 for sample container CQ0U-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw quartz from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO₃ to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

G. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ02U-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table CQ0U: Estimated solution pH and volumes of HNO₃ or NaOH solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with qz/clino (35 ml and capped)

| Estimated solution pH | Volume of HNO3 added, ml | Molarity of HNO3 to use |
|-----------------------|--------------------------|-------------------------|
| 3 | 0.045 | 1 |
| 3.25 | 0.254 | 1 |
| 3.5 | 0.143 | 0.1 |
| 3.75 | 0.080 | 0.1 |
| 4 | 0.452 | 0.01 |
| 4.25 | 0.254 | 0.01 |
| 4.5 | 0.143 | 0.01 |
| 4.75 | 0.081 | 0.01 |
| 5 | 0.045 | 0.01 |
| 5.25 | 0.025 | 0.01 |
| 5.5 | 0.140 | 0.001 |
| 5.75 | 0.073 | 0.001 |
| 6 | 0.035 | 0.001 |
| 6.25 | 0.140 | 0.001 |
| 6.5 | 0.010 | 0.001 |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use |
| 6.75 | 0.007 | 0.001 |
| 7 | 0.012 | 0.001 |
| 7.25 | 0.018 | 0.001 |
| 7.5 | 0.026 | 0.001 |
| 7.75 | 0.038 | 0.001 |
| 8 | 0.059 | 0.001 |
| 8.25 | 0.095 | 0.001 |
| 8.5 | 0.160 | 0.001 |
| 8.75 | 0.027 | 0.01 |
| 9 | 0.047 | 0.01 |
| 9.25 | 0.082 | 0.01 |
| 9.5 | 0.145 | 0.01 |

| CAPPED | |
|--------------|---------------------------|
| 10/15/98 | |
| Sample Name | Original Container wt., g |
| QC0U-pH 3.0 | 22.2814 |
| QC0U-pH 3.5 | 22.3164 |
| QC0U-pH 4.0 | 22.2015 |
| QC0U-pH 4.25 | 22.4189 |
| QC0U-pH 4.5 | 22.2534 |
| QC0U-pH 4.75 | 22.3343 |
| QC0U-pH 5.0 | 22.2908 |
| QC0U-pH 5.25 | 22.3581 |
| QC0U-pH 5.5 | 22.2616 |
| QC0U-pH 5.75 | 22.2747 |
| QC0U-pH 6.0 | 22.3086 |
| QC0U-pH 6.25 | 22.5789 |
| QC0U-pH 6.5 | 22.2996 |
| QC0U-pH 6.75 | 22.5477 |
| QC0U-pH 7.0 | 22.2264 |
| QC0U-pH 7.25 | 22.4240 |
| QC0U-pH 7.5 | 22.5538 |
| QC0U-pH 7.75 | 22.3680 |
| QC0U-pH 8.0 | 22.2458 |
| QC0U-pH 8.25 | 22.5762 |
| QC0U-pH 8.5 | 22.2989 |
| QC0U-pH 8.75 | 22.2344 |
| QC0U-pH 9.0 | 22.3123 |
| QC0U-pH 9.25 | 22.3063 |
| QC0U-pH 9.5 | 22.2314 |

10/16/98

AJ

$$\begin{array}{r}
 \textcircled{1} \text{ wt. of tube assembly + outgassed PCOV} = 35.0195 \text{ g.} \\
 \text{wt. of outgassed PCOV} = 35.0195 \\
 \hline
 - 33.5880 \\
 \hline
 1.4314 \text{ g.}
 \end{array}$$

$$\text{BET SA} = 27.673 \text{ m}^2/\text{g.}$$

Data stored in binder marked as "Results of Surface Area Analysis".

$$\begin{array}{r}
 \textcircled{2} \text{ wt. of tube assembly + outgassed Clinop + 92} \\
 \text{mixture} = 35.7772 \text{ g.}
 \end{array}$$

$$\begin{array}{r}
 \text{wt. of outgassed 92/Clinop mixture} = 35.7772 \\
 \hline
 - 33.5722 \\
 \hline
 2.2050 \text{ g}
 \end{array}$$

$$\text{BET SA} = 0.643 \text{ m}^2/\text{g}$$

Data stored in binder marked as "Results of Surface Area Analysis".

$\textcircled{3}$ 1g of quartz was added to each container & weighed. Data on next page (Capped Containers expt.).

CAPPED

10/16/98

| Sample Name | Wt. Of container + quartz, g |
|--------------|------------------------------|
| QC0U-pH 3.0 | 23.298 |
| QC0U-pH 3.5 | 23.3365 |
| QC0U-pH 4.0 | 23.2202 |
| QC0U-pH 4.25 | 23.4397 |
| QC0U-pH 4.5 | 23.2727 |
| QC0U-pH 4.75 | 23.3554 |
| QC0U-pH 5.0 | 23.3106 |
| QC0U-pH 5.25 | 23.3782 |
| QC0U-pH 5.5 | 23.2786 |
| QC0U-pH 5.75 | 23.2969 |
| QC0U-pH 6.0 | 23.3256 |
| QC0U-pH 6.25 | 23.5966 |
| QC0U-pH 6.5 | 23.3003 |
| QC0U-pH 6.75 | 23.5496 |
| QC0U-pH 7.0 | 23.2273 |
| QC0U-pH 7.25 | 23.4238 |
| QC0U-pH 7.5 | 23.5566 |
| QC0U-pH 7.75 | 23.3674 |
| QC0U-pH 8.0 | 23.2473 |
| QC0U-pH 8.25 | 23.5771 |
| QC0U-pH 8.5 | 23.3002 |
| QC0U-pH 8.75 | 23.2324 |
| QC0U-pH 9.0 | 23.3139 |
| QC0U-pH 9.25 | 23.3056 |
| QC0U-pH 9.5 | 23.2314 |

10/20/98 AT

- ① Add U-soln (50 PPb stock soln.) to each container
 - ② Weigh ~ 35g of solution & add to each container
 - ③ Record weight of each container.
- Data on next page & this page

CAPPED

10/20/98

| Sample Name | Wt. Of Container + quartz + U soln., g |
|--------------|--|
| QC0U-pH 3.0 | 58.3299 |
| QC0U-pH 3.5 | 58.4445 |
| QC0U-pH 4.0 | 58.2426 |
| QC0U-pH 4.25 | 58.4646 |
| QC0U-pH 4.5 | 58.2996 |
| QC0U-pH 4.75 | 58.3833 |
| QC0U-pH 5.0 | 58.3403 |
| QC0U-pH 5.25 | 58.4168 |
| QC0U-pH 5.5 | 58.2984 |
| QC0U-pH 5.75 | 58.3111 |
| QC0U-pH 6.0 | 58.3649 |
| QC0U-pH 6.25 | 58.6248 |
| QC0U-pH 6.5 | 58.3332 |
| QC0U-pH 6.75 | 58.5738 |
| QC0U-pH 7.0 | 58.2730 |
| QC0U-pH 7.25 | 58.4423 |
| QC0U-pH 7.5 | 58.5764 |
| QC0U-pH 7.75 | 58.3712 |
| QC0U-pH 8.0 | 58.2573 |

| | |
|--------------|---------|
| QC0U-pH 8.25 | 58.6098 |
| QC0U-pH 8.5 | 58.3618 |
| QC0U-pH 8.75 | 58.2537 |
| QC0U-pH 9.0 | 58.3124 |
| QC0U-pH 9.25 | 58.3212 |
| QC0U-pH 9.5 | 58.2468 |

10/20/98 AJ

① Preparation of solutions of different conc. of NaOH & HNO₃.

② Starting Stock soln. 6M NaOH.

③ Starting Stock soln. 1M HNO₃.

④ 100 ml of 1M NaOH

$$6M \times \text{Volume} = 1M \times 100 \text{ ml}$$

$$\text{Volume of NaOH} = \frac{100}{6} = 16.7 \text{ ml}$$

Add 16.7 ml of 6M NaOH solution to DI H₂O & fill it up to 100 ml mark in 100 ml volumetric flask.

⑤ 0.1M NaOH (100 ml) from 1M NaOH.

$$1M \times V = 0.1 \times 100$$

$$V = \frac{0.1 \times 100}{1} \text{ ml} = 10 \text{ ml}$$

Add 10 ml of 1M NaOH to H₂O & dilute it up to 100 ml mark on volumetric flask.

⑥ 100 ml of 0.01M NaOH from 1M NaOH

$$1M \times V = 0.01M \times 100 \text{ ml}$$

$$V = 1 \text{ ml}$$

Add 1 ml of 1M NaOH to H₂O & dilute it to 100 ml

⑦ 100 ml of 0.01M NaOH from 1M NaOH soln
Dilute 0.1 ml of 1M NaOH with H₂O to 100 ml mark.

10/21/98 AJ

CAPPED

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| Sample Name | Container Wt. After additions of HNO ₃ and NaOH., g |
|--------------|--|
| QC0U-pH 3.0 | 58.3719 |
| QC0U-pH 3.5 | 58.5872 |
| QC0U-pH 4.0 | 58.6910 |
| QC0U-pH 4.25 | 58.7128 |
| QC0U-pH 4.5 | 58.4385 |
| QC0U-pH 4.75 | 58.4590 |
| QC0U-pH 5.0 | 58.43814 |
| QC0U-pH 5.25 | 58.4371 |
| QC0U-pH 5.5 | 58.4334 |
| QC0U-pH 5.75 | 58.3788 |
| QC0U-pH 6.0 | 58.3938 |
| QC0U-pH 6.25 | 58.7599 |
| QC0U-pH 6.5 | 58.3382 |
| QC0U-pH 6.75 | 58.5789 |
| QC0U-pH 7.0 | 58.2826 |
| QC0U-pH 7.25 | 58.4563 |
| QC0U-pH 7.5 | 58.6001 |
| QC0U-pH 7.75 | 58.4047 |
| QC0U-pH 8.0 | 58.2994 |
| QC0U-pH 8.25 | 58.7000 |
| QC0U-pH 8.5 | 58.4715 |
| QC0U-pH 8.75 | 58.2768 |
| QC0U-pH 9.0 | 58.3530 |
| QC0U-pH 9.25 | 58.3978 |
| QC0U-pH 9.5 | 58.3850 |

The amounts of HNO₃ & NaOH additions are on next page.

10/21/98 CAPPED

Table CQU: Estimated solution pH and volumes of HNO₃ or NaOH solutions needed for adjustment of pH in 0.1 M NaNO₃ solutions with 50 ppb U in contact with qz/clino (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--------------------------------------|-------------------------------------|
| 2.5 | 0.143 | 1 |
| 2.75 | 0.080 | 1 |
| 3 | 0.045 | 1 |
| 3.25 | 0.025 | 1 |
| 3.5 | 0.143 | 0.1 |
| 3.75 | 0.080 | 0.1 |
| 4 | 0.452 | 0.01 |
| 4.25 | 0.254 | 0.01 |
| 4.5 | 0.143 | 0.01 |
| 4.75 | 0.081 | 0.01 |
| 5 | 0.045 | 0.01 |
| 5.25 | 0.025 | 0.01 |
| 5.5 | 0.014 0.140 | 0.01 0.001 |
| 5.75 | 0.073 | 0.001 |
| 6 | 0.035 | 0.001 |
| 6.25 | 0.014 0.140 | 0.001 0.0001 |
| 6.5 | 0.001 0.010 | 0.001 0.0001 |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use |
| 6.75 | 0.007 | 0.001 |
| 7 | 0.012 | 0.001 |
| 7.25 | 0.018 | 0.001 |
| 7.5 | 0.026 | 0.001 |
| 7.75 | 0.038 | 0.001 |
| 8 | 0.059 | 0.001 |
| 8.25 | 0.095 | 0.001 |
| 8.5 | 0.016 0.160 | 0.01 0.001 |
| 8.75 | 0.027 | 0.01 |
| 9 | 0.047 | 0.01 |
| 9.25 | 0.082 | 0.01 |
| 9.5 | 0.145 | 0.01 |
| 9.75 | 0.256 | 0.01 |
| 10 | 0.454 | 0.01 |

Keep the containers on gyratory shaker at 125 rpm tightly capped for 10 days.

10/22/98 AJ

① Preparation of 0.1M NaNO₃ matrix.
Refer to page 34, #3 on 8/21/98.

② Preparation of 50 ppb U-233 solution.
Refer to step #4 on page 34 dated 8/21/98.

③ Label and weigh LSA vials for uncapped sorption.

10/23/98 Data on page 107-117 →

10/26/98
10/27/98
AJ

UNCAPPED

| Sample Name | 10/26/98 Container wt. before sampling & pH measurements, g | 10/26/98 Measured pH | 10/27/98 Container wt. after sampling & pH measurements, g |
|--------------|--|-------------------------|---|
| QC0U-pH 3.0 | 58.4620 | 2.89 | 57.4363 |
| QC0U-pH 3.5 | 58.3103 | 3.42 | 57.2708 |
| QC0U-pH 4.0 | 58.0361 | 4.02 | 57.0022 |
| QC0U-pH 4.5 | 58.4261 | 4.60 | 57.3723 |
| QC0U-pH 4.75 | 58.2380 | 5.40 | 57.1786 |
| QC0U-pH 5.0 | 58.1550 | 5.90 | 57.0944 |
| QC0U-pH 5.25 | 58.6790 | 6.16 | 57.6144 |
| QC0U-pH 5.5 | 58.2458 | 6.28 | 57.1855 |
| QC0U-pH 5.75 | 58.5302 | 6.36 | 57.4821 |
| QC0U-pH 6.0 | 58.3222 | 6.47 | 57.2920 |
| QC0U-pH 6.25 | 58.4866 | 6.56 | 57.4593 |
| QC0U-pH 6.5 | 58.1347 | 6.67 | 57.1036 |
| QC0U-pH 6.75 | 58.1654 | 6.86 | 57.1318 |
| QC0U-pH 7.0 | 58.3242 | 7.02 | 57.2782 |
| QC0U-pH 7.25 | 58.0054 | 7.25 | 56.9681 |
| QC0U-pH 7.5 | 58.1339 | 7.43 | 57.0996 |
| QC0U-pH 7.75 | 58.5229 | 7.69 | 57.4212 |
| QC0U-pH 8.0 | 58.3107 | 7.93 | 57.2810 |
| QC0U-pH 8.25 | 58.2595 | 8.22 | 57.2184 |
| QC0U-pH 8.5 | 58.3156 | 8.4 | 57.2743 |
| QC0U-pH 8.75 | 58.1642 | 8.7 | 57.1146 |
| QC0U-pH 9.0 | 58.3253 | 8.94 | 57.2979 |
| QC0U-pH 9.25 | 58.9514 | 9.17 | 57.8931 |
| QC0U-pH 9.5 | 59.3251 | 9.23 | AJ 58.2753 |
| QC0U-IU | 57.3725 | | 56.331 |

Sorption 10/26/98 UNCAPPED 10/27/98

| Sample Name | wt. Of vial, g | wt. Of vial + sample, g |
|---------------|----------------|-------------------------|
| QC0U-pH 2a | 7.5995 | 8.0965 |
| QC0U-pH 2b | 7.6098 | 8.1107 |
| QC0U-pH 2.5a | 7.5969 | 8.1056 |
| QC0U-pH 2.5b | 7.6040 | 8.1141 |
| QC0U-pH 3a | 7.6250 | 8.1328 |
| QC0U-pH 3b | 7.6885 | 8.1973 |
| QC0U-pH 3.5a | 7.6420 | 8.1486 |
| QC0U-pH 3.5b | 7.5713 | 8.0808 |
| QC0U-pH 4a | 7.5936 | 8.1006 |
| QC0U-pH 4b | 7.5756 | 8.0847 |
| QC0U-pH 4.5a | 7.6521 | 8.1594 |
| QC0U-pH 4.5b | 7.6181 | 8.1281 |
| QC0U-pH 4.75a | 7.6215 | 8.1274 |
| QC0U-pH 4.75b | 7.6168 | 8.1238 |
| QC0U-pH 5a | 7.6462 | 8.1508 |
| QC0U-pH 5b | 7.5074 | 8.0146 |
| QC0U-pH 5.25a | 7.6550 | 8.1570 |
| QC0U-pH 5.25b | 7.6484 | 8.1536 |
| QC0U-pH 5.5a | 7.5945 | 8.1015 |
| QC0U-pH 5.5b | 7.6517 | 8.1608 |
| QC0U-pH 5.75a | 7.7248 | 8.2296 |
| QC0U-pH 5.75b | 7.6767 | 8.1829 |
| QC0U-pH 6a | 7.6155 | 8.1229 |
| QC0U-pH 6b | 7.6447 | 8.1512 |
| QC0U-pH 6.25a | 7.6753 | 8.1803 |
| QC0U-pH 6.25b | 7.6098 | 8.1157 |
| QC0U-pH 6.5a | 7.5903 | 8.0909 |
| QC0U-pH 6.5b | 7.6146 | 8.1176 |
| QC0U-pH 6.75a | 7.6478 | 8.1506 |
| QC0U-pH 6.75b | 7.6146 | 8.1177 |
| QC0U-pH 7a | 7.6030 | 8.1080 |
| QC0U-pH 7b | 7.6006 | 8.1073 |
| QC0U-pH 7.25a | 7.6286 | 8.1331 |
| QC0U-pH 7.25b | 7.6595 | 8.1631 |
| QC0U-pH 7.5a | 7.5742 | 8.0766 |
| QC0U-pH 7.5b | 7.6378 | 8.1418 |
| QC0U-pH 7.75a | 7.6207 | 8.1248 |
| QC0U-pH 7.75b | 7.6675 | 8.1718 |
| QC0U-pH 8a | 7.6814 | 8.1859 |
| QC0U-pH 8b | 7.6313 | 8.1361 |

| | | |
|---------------|--------|--------|
| QC0U-pH 8.25a | 7.6963 | 8.1998 |
| QC0U-pH 8.25b | 7.6333 | 8.1396 |
| QC0U-pH 8.5a | 7.7209 | 8.2267 |
| QC0U-pH 8.5b | 7.6821 | 8.1878 |
| QC0U-pH 8.75a | 7.6408 | 8.1508 |
| QC0U-pH 8.75b | 7.5509 | 8.0570 |
| QC0U-pH 9a | 7.6371 | 8.1398 |
| QC0U-pH 9b | 7.6053 | 8.1075 |
| QC0U-pH 9.25a | 7.5855 | 8.0874 |
| QC0U-pH 9.25b | 7.5239 | 8.0281 |
| QC0U-pH 9.5a | 7.6024 | 8.1060 |
| QC0U-pH 9.5b | 7.6570 | 8.1629 |

| | | | |
|------|----------|-------------|----------------------|
| | | wt. of vial | wt. of vial + sample |
| From | QC0U-IVa | 7.5825 g | 8.0873 g |
| Page | QC0U-IVb | 7.6374 g | 8.1734 g |
| 63 | | | |

UNCAPPED (Description)

| | 10/27/98 | 11/2/98 | 11/2/98 | 11/2/98 | 11/2/98 |
|---------------|--------------------|---------------------------------------|-----------------------------------|--|--|
| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after trans quartz, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. After acid add & supernatant retransfer, g |
| QC0dU-pH 3.0 | 13.9370 | 19.7021 | 22.6931 | 28.1662 | 54.5584 |
| QC0dU-pH 3.5 | 13.3977 | 18.4884 | 21.4646 | 27.5504 | 55.0728 |
| QC0dU-pH 4 | 13.6386 | 19.6744 | 22.6495 | 28.3190 | 53.8556 |
| QC0dU-pH 4.5 | 13.3731 | 19.5322 | 22.5070 | 28.5747 | 54.0888 |
| QC0dU-pH 4.75 | 13.4440 | 18.9486 | 21.9173 | 28.9500 | 54.5690 |
| QC0dU-pH 5 | 13.3082 | 18.9112 | 21.8803 | 28.8525 | 54.3923 |
| QC0dU-pH 5.25 | 13.3002 | 19.4492 | 22.4172 | 28.3850 | 54.3492 |
| QC0dU-pH 5.5 | 13.3362 | 19.4266 | 22.3925 | 28.8661 | 53.9627 |
| QC0dU-pH 5.75 | 13.4615 | 19.8219 | 22.8020 | AJ 28.7298 | 53.9961 |
| QC0dU-pH 6 | 13.3058 | 18.6368 | 21.6075 | 28.9765 | 54.8188 |
| QC0dU-pH 6.25 | 13.8832 | 20.3925 | 23.3546 | 28.4339 | 53.8392 |
| QC0dU-pH 6.5 | 13.3545 | 19.3664 | 22.3296 | 28.4738 | 53.9934 |
| QC0dU-pH 6.75 | 13.3763 | 19.4420 | 22.4016 | 29.3177 | 53.9672 |
| QC0dU-pH 7 | 13.3160 | 20.1758 | 23.1398 | 27.3177 | 53.2533 |
| QC0dU-pH 7.25 | 13.9328 | 19.2558 | 22.2121 | 28.6095 | 54.5424 |
| QC0dU-pH 7.5 | 13.8498 | 20.0710 | 23.0286 | 28.5504 | 53.7594 |
| QC0dU-pH 7.75 | 13.8157 | 20.1472 | 23.1164 | 28.9607 | 53.9406 |
| QC0dU-pH 8 | 13.8956 | 20.9892 | 23.9604 | 28.4487 | 53.0075 |
| QC0dU-pH 8.25 | 13.9114 | 20.1958 | 23.1594 | 28.7827 | 53.6849 |
| QC0dU-pH 8.5 | 13.3848 | 19.8462 | 22.8040 | 28.8835 | 53.6768 |
| QC0dU-pH 8.75 | 13.2552 | 19.3537 | 22.3091 | 28.4279 | 53.8206 |
| QC0dU-pH 9 | 13.3564 | 19.2908 | 22.2547 | 28.2387 | 54.2202 |
| QC0dU-pH 9.25 | 13.8177 | 19.9246 | 22.8843 | 28.7752 | 54.6056 |
| QC0dU-pH 9.5 | 13.8417 | 21.1124 | 24.071 | AJ 28.6548 | 53.7028 |

UNCAPPED

| Sample Name | 11/12/98 Container wt. before sampling | 11/12/98 Container wt. after sampling | 11/13/98 wt. Of PP tubes before sampling | 11/16/98 wt. Of PP tubes after sampling |
|--------------|--|---|--|---|
| QC0U-pH 3.0 | 54.3694 | 53.3672 | 22.6079 | 21.6196 |
| QC0U-pH 3.5 | 54.9548 | 53.9503 | 21.4538 | 20.4400 |
| QC0U-pH 4.0 | 53.7894 | 52.7797 | 22.6406 | 21.6576 |
| QC0U-pH 4.5 | 54.0169 | 52.9890 | 22.4985 | 21.4221 |
| QC0U-pH 4.75 | 54.4816 | 53.4762 | 21.8850 | 20.7662 |
| QC0U-pH 5.0 | 54.3155 | 52.2754 | 21.8184 | 20.7876 |
| QC0U-pH 5.25 | 54.2364 | 53.2243 | 22.3602 | 21.3089 |
| QC0U-pH 5.5 | 53.8962 | 52.8860 | 22.3789 | 21.3067 |
| QC0U-pH 5.75 | 53.9273 | 52.9224 | 22.7762 | 21.7630 |
| QC0U-pH 6.0 | 54.6987 | 53.6962 | 21.5463 | 20.5227 |
| QC0U-pH 6.25 | 53.7313 | 52.7239 | 23.3190 | 22.2796 |
| QC0U-pH 6.5 | 53.9292 | 52.9048 | 23.3019 | 21.2711 |
| QC0U-pH 6.75 | 53.8602 | 52.8569 | 22.2793 | 21.2264 |
| QC0U-pH 7.0 | 53.1704 | 52.1560 | AJ 22.0535 | 22.0162 |
| QC0U-pH 7.25 | 54.4493 | 53.4394 | 22.1217 | 21.0784 |
| QC0U-pH 7.5 | 53.6893 | 52.6831 | 22.8737 | 21.8218 |
| QC0U-pH 7.75 | 53.8378 | 52.8331 | 23.1049 | 22.0889 |
| QC0U-pH 8.0 | 52.9416 | 51.9409 | 23.9330 | 22.8944 |
| QC0U-pH 8.25 | 53.5846 | 52.5743 | 23.1445 | 22.1288 |
| QC0U-pH 8.5 | 53.5601 | 52.5517 | 22.7589 | 21.7285 |
| QC0U-pH 8.75 | 53.7539 | 52.7544 | 22.2900 | 21.2573 |
| QC0U-pH 9.0 | 54.1434 | 53.1365 | 22.2288 | 21.2246 |
| QC0U-pH 9.25 | 54.5034 | 53.4947 | 22.8775 | 21.8621 |
| QC0U-pH 9.5 | 53.615 | 52.5718 | 24.0616 | 23.0454 |

DESORPTION (UNCAPPED) 11/13/98 11/12/98 11/12/98

| Sample Name | 11/13/98 PP tubes wt. of vial, g | Desorption wt. Of vial + sample, g | 11/12/98 PC containers wt. of vial, g | 11/12/98 Desorption wt. Of vial + sample, g |
|---------------|--|--|---|--|
| QC0U-pH 3a | 7.6128 | 8.1109 | 7.6319 | 8.1479 |
| QC0U-pH 3b | 7.6332 | 8.1248 | 7.6100 | 8.1215 |
| QC0U-pH 3.5a | 7.6077 | 8.1081 | 7.6195 | 8.1336 |
| QC0U-pH 3.5b | 7.7021 | 8.1942 | 7.6862 | 8.1952 |
| QC0U-pH 4a | 7.7182 | 8.2098 | 7.6374 | 8.1428 |
| QC0U-pH 4b | 7.7623 | 8.2503 | 7.6217 | 8.1277 |
| QC0U-pH 4.5a | 7.7152 | 8.2247 | 7.5812 | 8.0908 |
| QC0U-pH 4.5b | 7.6936 | 8.1991 | 7.5669 | 8.0803 |
| QC0U-pH 4.75a | 7.7347 | 8.2376 | 7.6325 | 8.1338 |
| QC0U-pH 4.75b | 7.7445 | 8.2481 | 7.6093 | 8.1139 |
| QC0U-pH 5a | 7.7220 | 8.2255 | 7.6474, 7.5967 | 8.1498, 8.1003 |
| QC0U-pH 5b | 7.7301 | 8.2311 | 7.6042, 7.5967 | 8.1082, 8.1001 |
| QC0U-pH 5.25a | 7.6881 | 8.1949 | 7.6092 | 8.1158 |
| QC0U-pH 5.25b | 7.7201 | 8.2301/0 | 7.6496 | 8.1544 |
| QC0U-pH 5.5a | 7.7081 | 8.2110 | 7.6185 | 8.1246 |
| QC0U-pH 5.5b | 7.7282 | 8.2295 | 7.5961 | 8.1017 |
| QC0U-pH 5.75a | 7.7550 | 8.2598 | 7.6102 | 8.1122 |
| QC0U-pH 5.75b | 7.7103 | 8.2133 | 7.5853 | 8.0888 |
| QC0U-pH 6a | 7.7196 | 8.2239 | 7.6630 63 | 8.1670 |
| QC0U-pH 6b | 7.7192 | 8.2249 | 7.6372 | 8.1382 |
| QC0U-pH 6.25a | 7.7198 | 8.2176 | 7.5556 | 8.0589 |
| QC0U-pH 6.25b | 7.7370 | 8.2347 | 7.5969 | 8.0996 |
| QC0U-pH 6.5a | 7.7546 | 8.2550 | 7.6082 | 8.1184 |
| QC0U-pH 6.5b | 7.6972 | 8.1996 | 7.6099 | 8.1242 |
| QC0U-pH 6.75a | 7.7638 | 8.2691 | 7.5658 | 8.0670 |
| QC0U-pH 6.75b | 7.7634 | 8.2700 | 7.5742 | 8.0763 |
| QC0U-pH 7a | 7.7436 | 8.2574 | 7.6015 | 8.1018 |
| QC0U-pH 7b | 7.7259 | 8.2294 | 7.5727 | 8.0857 |
| QC0U-pH 7.25a | 7.7279 | 8.2382 | 7.5122 | 8.0171 |
| QC0U-pH 7.25b | 7.7228 | 8.2264 | 7.6356 | 8.1397 |
| QC0U-pH 7.5a | 7.7202 | 8.2259 | 7.5886 | 8.0924 |
| QC0U-pH 7.5b | 7.7196 | 8.2228 | 7.5977 | 8.0992 |
| QC0U-pH 7.75a | 7.7337 | 8.2401 | 7.6060 | 8.1068 |
| QC0U-pH 7.75b | 7.7906 | 8.2929 | 7.5951 | 8.0950 |
| QC0U-pH 8a | 7.7802 | 8.2847 | 7.6011 | 8.1023 |
| QC0U-pH 8b | 7.7126 | 8.2280 | 7.5984 | 8.0978 |

| | | | | |
|---------------|--------|--------|--------|--------|
| QCOU-pH 8.25a | 7.8764 | 8.3804 | 7.6224 | 8.1230 |
| QCOU-pH 8.25b | 7.7479 | 8.2513 | 7.6088 | 8.1091 |
| QCOU-pH 8.5a | 7.7592 | 8.2658 | 7.6060 | 8.1085 |
| QCOU-pH 8.5b | 7.7712 | 8.2796 | 7.6368 | 8.1387 |
| QCOU-pH 8.75a | 7.7016 | 8.2096 | 7.6100 | 8.1095 |
| QCOU-pH 8.75b | 7.7295 | 8.2435 | 7.5646 | 8.0656 |
| QCOU-pH 9a | 7.7049 | 8.2060 | 7.6408 | 8.1424 |
| QCOU-pH 9b | 7.7095 | 8.0790 | 7.5786 | 8.0826 |
| QCOU-pH 9.25a | 7.7422 | 8.2471 | 7.5980 | 8.0995 |
| QCOU-pH 9.25b | 7.6484 | 8.1503 | 7.6812 | 8.1821 |
| QCOU-pH 9.5a | 7.7131 | 8.2173 | 7.6382 | 8.1400 |
| QCOU-pH 9.5b | 7.7528 | 8.2611 | 7.5338 | 8.0754 |

*data recounted for initial counts (from page
on 2/10/99)*

(CPM)

QCOU-IVa 513.334

QCOU-IVb 521.267

231 MISSING TUBE(S) 2/10/99 Std.

| | | | | | | | | |
|----|------|------|-------|---------|---------|--------|--------|-------------|
| 31 | 8.60 | 2.80 | 116.9 | 513.334 | 3.02 | 515.93 | 3.09 | 631.76 |
| 23 | 32 | 8.47 | 1.15 | 277.5 | 521.267 | 3.02 | 523.01 | 3.09 633.89 |
| 23 | | | | | | | | |

Initial conc. recounted on 2/20/99

231 MISSING TUBE(S) Std.

| | | | | | | | | |
|-----|------|------|------|---------|---------|--------|--------|-------------|
| 103 | 8.55 | 0.10 | 3173 | 516.770 | 3.02 | 517.42 | 3.09 | 637.04 |
| 23 | 104 | 8.43 | 1.09 | 289.9 | 524.057 | 3.02 | 525.20 | 3.09 638.39 |

Time: 999.99

Data Mode: CPM

Background Subtract: 1st Vial

Nuclide: MANUAL

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.11 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.78 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.81 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

29 Oct., 1998

Protocol # 23

AT

QCOU - Sorption Data

UNCAPPED

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|-------|-----------------|------------|--------------|-------------|-------------------|
| P# 1 | 999.99 | 20.11 1.41 | 3.783 3.25 | 29.81 1.16 | 151.00 B |
| 23 2 | 8.63 | 1.09 288.3 | 511.512 3.02 | 511.56 3.10 | 617.35] std. |
| 23 3 | 8.35 | 0.37 859.7 | 528.432 3.02 | 530.19 3.09 | 612.42 |
| 231 | MISSING TUBE(S) | | | | |
| 5 | 9.14 | 2.53 124.7 | 482.431 3.02 | 486.60 3.09 | 618.59 29 |
| 23 6 | 8.74 | 0.94 331.8 | 505.141 3.02 | 506.00 3.10 | 613.68 26 QCO |
| 23 7 | 8.68 | 3.73 89.09 | 509.005 3.02 | 514.20 3.08 | 617.77 25.59 sorf |
| 23 8 | 8.41 | 4.26 80.16 | 525.111 3.02 | 530.36 3.08 | 617.75 25.56 |
| 23 9 | 8.61 | 0.00 0.00 | 513.058 3.02 | 510.37 3.10 | 628.59 39 |
| 23 10 | 8.81 | 3.27 100.0 | 500.871 3.02 | 504.47 3.09 | 623.48 36 |
| 23 11 | 8.75 | 2.17 147.5 | 504.217 3.02 | 506.42 3.09 | 619.82 3.59 |
| 23 12 | 8.78 | 2.78 116.6 | 502.367 3.02 | 505.27 3.09 | 625.51 |
| 23 13 | 9.14 | 1.77 175.7 | 482.431 3.02 | 483.54 3.10 | 621.91 |
| 23 14 | 8.81 | 0.00 0.00 | 501.552 3.02 | 502.99 3.09 | 625.67 |
| 23 15 | 9.98 | 0.00 0.00 | 441.808 3.02 | 441.03 3.12 | 629.38 |
| 23 16 | 9.68 | 1.68 179.0 | 455.514 3.02 | 456.55 3.11 | 620.04 |
| 23 17 | 19.54 | 0.00 0.00 | 223.648 3.05 | 222.44 3.23 | 626.49 |
| 23 18 | 19.65 | 1.46 144.4 | 222.426 3.05 | 224.08 3.21 | 621.51 |
| 23 19 | 32.72 | 0.00 0.00 | 132.067 3.09 | 131.89 3.38 | 632.04 |
| 23 20 | 31.31 | 0.23 706.4 | 138.184 3.08 | 139.27 3.35 | 623.68 |
| 23 21 | 42.68 | 0.11 1313 | 100.341 3.12 | 100.39 3.50 | 627.77 |
| 23 | | | | | |
| 22 | 41.63 | 0.28 508.7 | 102.991 3.11 | 103.94 3.46 | 625.22 |
| 23 23 | 45.85 | 0.34 396.1 | 93.142 3.12 | 93.59 3.53 | 624.90 |
| 23 24 | 45.29 | 0.00 0.00 | 94.340 3.12 | 94.08 3.54 | 626.88 |
| 23 25 | 42.99 | 1.54 93.82 | 99.590 3.12 | 102.03 3.45 | 620.27 |
| 23 26 | 42.83 | 1.02 140.9 | 99.976 3.12 | 101.24 3.47 | 619.87 |
| 23 27 | 42.76 | 1.38 104.9 | 100.146 3.12 | 101.92 3.46 | 619.18 |
| 23 28 | 42.49 | 1.07 134.8 | 100.830 3.11 | 101.80 3.47 | 619.97 |
| 23 29 | 42.49 | 0.53 270.0 | 100.853 3.11 | 101.70 3.48 | 622.63 |
| 23 30 | 43.27 | 0.18 785.8 | 98.944 3.12 | 99.54 3.49 | 621.19 |
| 23 31 | 43.46 | 0.00 0.00 | 98.472 3.12 | 97.96 3.52 | 628.09 |
| 23 32 | 42.63 | 0.65 220.2 | 100.486 3.12 | 101.39 3.48 | 623.25 |
| 23 33 | 33.99 | 0.60 265.0 | 126.961 3.09 | 128.26 3.37 | 625.84 |
| 23 34 | 34.16 | 0.17 902.3 | 126.340 3.09 | 126.72 3.39 | 626.07 |
| 23 35 | 33.55 | 0.39 403.8 | 128.706 3.09 | 129.44 3.38 | 622.10 |
| 23 36 | 33.46 | 0.99 163.5 | 129.032 3.09 | 130.56 3.36 | 623.62 |
| 23 37 | 29.70 | 1.03 165.9 | 145.948 3.08 | 148.30 3.31 | 619.79 |
| 23 38 | 29.78 | 0.00 0.00 | 145.445 3.08 | 144.74 3.35 | 632.98 |
| 23 39 | 24.11 | 0.00 0.00 | 180.539 3.06 | 180.43 3.28 | 623.27 |
| 23 40 | 24.36 | 2.22 87.26 | 178.647 3.06 | 180.86 3.26 | 616.25 |
| 23 41 | 17.47 | 0.00 0.00 | 250.596 3.05 | 249.24 3.21 | 627.38 |

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 16.86 | 0.17 | 1290 | 259.799 | 3.04 | 260.88 | 3.19 | 623.35 |
| 23 | 43 | 13.93 | 2.07 | 122.7 | 315.384 | 3.04 | 318.86 | 3.14 | 620.54 |
| 23 | 44 | 13.90 | 0.82 | 300.6 | 316.073 | 3.04 | 316.66 | 3.16 | 621.64 |
| 23 | 45 | 9.79 | 0.93 | 317.2 | 450.967 | 3.02 | 453.85 | 3.10 | 622.77 |
| 23 | 46 | 9.66 | 0.49 | 602.3 | 456.879 | 3.02 | 457.77 | 3.10 | 622.29 |
| 23 | 47 | 9.04 | 0.00 | 0.00 | 487.810 | 3.02 | 488.44 | 3.10 | 621.62 |
| 23 | 48 | 9.07 | 2.27 | 139.1 | 486.404 | 3.02 | 490.59 | 3.09 | 621.72 |
| 23 | 49 | 8.67 | 3.07 | 106.9 | 508.904 | 3.02 | 513.44 | 3.08 | 621.66 |
| 23 | 50 | 8.49 | 2.50 | 131.0 | 519.656 | 3.02 | 521.90 | 3.09 | 620.95 |
| 23 | 51 | 8.77 | 0.75 | 411.2 | 503.058 | 3.02 | 504.62 | 3.09 | 628.43 |
| 23 | 52 | 8.59 | 0.61 | 512.6 | 513.563 | 3.02 | 514.54 | 3.09 | 625.70 |
| 23 | 53 | 9.18 | 1.89 | 164.4 | 481.184 | 3.02 | 483.81 | 3.09 | 620.60 |
| 23 | 54 | 8.70 | 3.56 | 92.89 | 507.137 | 3.02 | 511.22 | 3.09 | 620.87 |
| 23 | 55 | 9.14 | 3.30 | 97.37 | 482.541 | 3.02 | 485.94 | 3.09 | 632.96 |
| 23 | 56 | 9.01 | 2.86 | 112.1 | 489.447 | 3.02 | 494.27 | 3.09 | 632.50 |

17 Nov 98 10:07 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.10 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.81 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.75 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|------|------------|------------|------------|---------------------------------|
| P# | 1 | 999.99 | 20.10 | 1.41 | 3.807 3.24 29.75 1.16 152.02 |
| 23 | 2 | 9.43 | 3.02 | 104.1 | 467.879 3.02 470.36 3.10 617.78 |
| 23 | 3 | 9.28 | 2.86 | 110.6 | 475.072 3.02 478.44 3.09 621.27 |
| 23 | 4 | 9.62 | 1.32 | 227.7 | 458.355 3.02 460.28 3.10 621.89 |
| 23 | 5 | 9.27 | 8.06 | 43.40 | 476.020 3.02 486.00 3.07 619.03 |
| 23 | 6 | 9.82 | 4.14 | 76.22 | 449.146 3.02 454.06 3.09 626.72 |
| 23 | 7 | 9.96 | 1.49 | 198.6 | 442.578 3.03 445.05 3.10 627.39 |
| 23 | 8 | 10.51 | 0.74 | 382.4 | 419.124 3.03 420.68 3.11 624.63 |
| 23 | 9 | 10.75 | 2.23 | 130.0 | 409.588 3.03 412.12 3.11 623.88 |
| 23 | 10 | 17.58 | 1.46 | 152.8 | 249.037 3.05 251.65 3.18 627.13 |
| 23 | 11 | 17.41 | 2.36 | 96.95 | 251.449 3.05 254.86 3.18 621.23 |
| 23 | 12 | 25.65 | 0.18 | 1025 | 169.487 3.07 169.79 3.29 629.40 |
| 23 | 13 | 25.58 | 1.91 | 98.15 | 169.922 3.07 171.86 3.27 619.02 |
| 23 | 14 | 25.12 | 1.44 | 130.2 | 173.223 3.07 175.35 3.26 623.04 |

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 15 | 25.11 | 2.24 | 85.00 | 173.174 | 3.07 | 176.03 | 3.26 | 622.87 |
| 23 | 16 | 34.82 | 2.99 | 55.23 | 123.849 | 3.09 | 127.58 | 3.34 | 614.34 |
| 23 | 17 | 34.37 | 2.45 | 67.08 | 125.492 | 3.09 | 128.79 | 3.35 | 618.93 |
| 23 | 18 | 33.76 | 0.49 | 324.4 | 127.828 | 3.09 | 128.52 | 3.38 | 621.52 |
| 23 | 19 | 33.65 | 1.54 | 105.9 | 128.258 | 3.09 | 130.55 | 3.35 | 618.04 |
| 23 | 20 | 36.94 | 1.80 | 86.84 | 116.523 | 3.10 | 118.96 | 3.39 | 619.15 |
| 23 | 21 | 35.34 | 1.07 | 147.3 | 121.943 | 3.10 | 123.96 | 3.38 | 619.70 |
| 23 | 22 | 35.57 | 1.16 | 135.9 | 121.130 | 3.10 | 122.26 | 3.39 | 617.48 |
| 23 | 23 | 36.95 | 1.47 | 105.5 | 116.464 | 3.10 | 118.27 | 3.40 | 620.03 |
| 23 | 24 | 33.29 | 1.02 | 158.6 | 129.687 | 3.09 | 131.71 | 3.35 | 620.96 |
| 23 | 25 | 32.47 | 2.39 | 70.78 | 133.119 | 3.09 | 136.41 | 3.33 | 616.03 |
| 23 | 26 | 32.58 | 0.04 | 4221 | 132.626 | 3.09 | 133.48 | 3.36 | 623.38 |
| 23 | 27 | 32.94 | 1.09 | 149.1 | 131.135 | 3.09 | 132.64 | 3.36 | 619.45 |
| 23 | 28 | 32.04 | 0.85 | 194.2 | 134.957 | 3.09 | 137.17 | 3.34 | 622.85 |
| 23 | 29 | 32.96 | 2.99 | 56.75 | 131.023 | 3.09 | 135.21 | 3.32 | 615.00 |
| 23 | 30 | 33.92 | 2.37 | 69.79 | 127.237 | 3.09 | 129.54 | 3.36 | 620.67 |
| 23 | 31 | 34.02 | 1.24 | 129.4 | 126.881 | 3.09 | 128.72 | 3.36 | 622.59 |
| 23 | 32 | 29.03 | 0.88 | 195.6 | 149.345 | 3.08 | 151.03 | 3.31 | 623.78 |
| 23 | 33 | 29.06 | 1.20 | 144.2 | 149.118 | 3.08 | 151.05 | 3.31 | 627.93 |
| 23 | 34 | 25.68 | 1.40 | 132.4 | 169.324 | 3.07 | 171.35 | 3.27 | 622.30 |
| 23 | 35 | 24.91 | 0.00 | 0.00 | 174.595 | 3.07 | 175.51 | 3.28 | 633.02 |
| 23 | 36 | 18.66 | 0.91 | 235.2 | 234.349 | 3.05 | 235.80 | 3.20 | 626.17 |
| 23 | 37 | 19.01 | 1.37 | 157.0 | 230.017 | 3.05 | 231.06 | 3.21 | 624.65 |
| 23 | 38 | 14.76 | 2.40 | 103.7 | 297.277 | 3.04 | 299.66 | 3.16 | 619.12 |
| 23 | 39 | 14.91 | 0.56 | 423.4 | 294.248 | 3.04 | 295.61 | 3.16 | 629.02 |
| 23 | 40 | 10.29 | 2.74 | 109.2 | 428.554 | 3.03 | 431.48 | 3.10 | 623.72 |
| 23 | 41 | 10.42 | 3.90 | 78.25 | 422.680 | 3.03 | 426.30 | 3.10 | 620.87 |

18 Nov 98 02:27 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 9.51 | 3.14 | 99.93 | 463.806 | 3.02 | 466.68 | 3.10 | 625.45 |
| 23 | 43 | 9.80 | 2.45 | 124.2 | 449.764 | 3.03 | 453.32 | 3.10 | 624.41 |
| 23 | 44 | 9.55 | 2.52 | 122.6 | 461.952 | 3.02 | 465.02 | 3.10 | 625.18 |
| 23 | 45 | 9.36 | 0.00 | 0.00 | 471.193 | 3.02 | 471.11 | 3.11 | 626.94 |
| 23 | 46 | 9.63 | 4.62 | 69.66 | 457.771 | 3.02 | 462.78 | 3.09 | 626.02 |
| 23 | 47 | 9.54 | 5.06 | 64.43 | 462.021 | 3.02 | 466.90 | 3.09 | 622.40 |
| 23 | 48 | 9.53 | 4.14 | 77.31 | 462.720 | 3.02 | 467.74 | 3.09 | 629.62 |
| 23 | 49 | 9.46 | 3.90 | 82.03 | 466.172 | 3.02 | 470.04 | 3.09 | 634.55 |
| 23 | 50 | 10.13 | 4.29 | 72.70 | 434.890 | 3.03 | 440.84 | 3.09 | 621.81 |
| 23 | 51 | 9.23 | 7.21 | 47.90 | 477.883 | 3.02 | 485.42 | 3.08 | 622.61 |

233 MISSING TUBE(S) QCOUD, PP tubes desorption

| | | | | | | | | | |
|-----------------|-----------|-------|-------------|---------|----------|--------|---------|--------|--------|
| MISSING TUBE(S) | ALCOA, PR | BASES | DESCRIPTION | | | | | | |
| 55 | 13.97 | 1.95 | 129.7 | 314.303 | 3.04 | 318.14 | 3.14 | 615.93 | |
| 23 | 56 | 14.21 | 1.01 | 241.9 | 308.930 | 3.04 | 310.72 | 3.15 | 618.86 |
| 23 | 57 | 12.82 | 2.76 | 97.38 | 342.839 | 3.03 | 346.62 | 3.13 | 609.40 |
| 23 | 58 | 13.67 | 2.95 | 88.67 | 321.504 | 3.03 | 325.78 | 3.13 | 609.51 |
| 23 | 59 | 10.85 | 0.00 | 0.00 | 406.239 | 3.03 | 406.66 | 3.12 | 617.43 |
| 23 | 60 | 10.84 | 2.87 | 101.8 | 406.525 | 3.03 | 411.58 | 3.10 | 620.80 |
| 23 | 61 | 7.94 | 3.20 | 107.3 | 556.646 | 3.02 | 559.17 | 3.08 | 612.34 |
| 23 | 62 | 8.21 | 2.56 | 130.3 | 537.849 | 3.02 | 540.90 | 3.08 | 612.37 |
| 23 | 63 | 3.41 | 10.40 | 57.57 | 1300.005 | 3.01 | 1308.08 | 3.03 | 612.78 |
| 23 | 64 | 3.42 | 5.63 | 97.50 | 1297.363 | 3.01 | 1304.17 | 3.03 | 615.17 |
| 23 | 65 | 2.72 | 1.50 | 354.0 | 1431.487 | 3.01 | 1433.12 | 3.03 | 612.84 |

QCOV, desorption for PP tubes

| | | | | | | | | | |
|----|-----|-------|-------|-------|----------|------|---------|------|--------|
| 23 | 66 | 2.69 | 5.93 | 105.1 | 1651.955 | 3.00 | 1657.62 | 3.02 | 616.29 |
| 23 | 67 | 2.49 | 9.62 | 71.87 | 1787.759 | 3.00 | 1796.36 | 3.02 | 609.79 |
| 23 | 68 | 2.56 | 8.03 | 82.65 | 1732.521 | 3.01 | 1741.35 | 3.02 | 612.85 |
| 23 | 69 | 2.55 | 12.06 | 58.94 | 1742.468 | 3.00 | 1754.96 | 3.01 | 610.11 |
| 23 | 70 | 2.61 | 9.02 | 74.11 | 1702.323 | 3.00 | 1708.57 | 3.02 | 612.95 |
| 23 | 71 | 2.57 | 7.14 | 91.27 | 1729.656 | 3.00 | 1734.85 | 3.02 | 613.11 |
| 23 | 72 | 2.60 | 1.83 | 318.4 | 1711.962 | 3.00 | 1715.25 | 3.02 | 616.37 |
| 23 | 73 | 2.25 | 2.13 | 296.1 | 1977.971 | 3.00 | 1983.59 | 3.02 | 612.65 |
| 23 | 74 | 2.23 | 2.77 | 231.2 | 1997.538 | 3.00 | 1998.95 | 3.02 | 611.58 |
| 23 | 75 | 2.77 | 10.23 | 64.76 | 1604.857 | 3.00 | 1617.19 | 3.02 | 611.56 |
| 23 | 76 | 2.71 | 7.21 | 88.15 | 1638.628 | 3.00 | 1644.42 | 3.02 | 617.85 |
| 23 | 77 | 2.51 | 6.60 | 98.97 | 1771.890 | 3.00 | 1779.02 | 3.02 | 621.68 |
| 23 | 78 | 2.50 | 12.30 | 58.57 | 1777.793 | 3.00 | 1791.85 | 3.01 | 612.59 |
| 23 | 79 | 2.53 | 7.18 | 91.60 | 1758.248 | 3.00 | 1763.93 | 3.02 | 611.88 |
| 23 | 80 | 2.47 | 7.03 | 94.39 | 1796.598 | 3.01 | 1805.48 | 3.02 | 620.46 |
| 23 | 81 | 2.67 | 3.87 | 154.9 | 1660.612 | 3.01 | 1665.76 | 3.03 | 607.18 |
| 23 | 82 | 2.69 | 8.90 | 73.86 | 1650.840 | 3.00 | 1661.33 | 3.02 | 610.56 |
| 23 | 83 | 2.56 | 5.68 | 111.8 | 1738.380 | 3.00 | 1747.60 | 3.02 | 605.69 |
| 23 | 84 | 2.50 | 10.70 | 65.64 | 1775.793 | 3.00 | 1786.65 | 3.02 | 609.06 |
| 23 | 85 | 3.13 | 8.66 | 70.10 | 1418.877 | 3.01 | 1426.48 | 3.02 | 610.84 |
| 23 | 86 | 3.14 | 8.88 | 68.47 | 1413.709 | 3.01 | 1422.48 | 3.02 | 612.40 |
| 23 | 87 | 3.43 | 6.43 | 86.57 | 1293.277 | 3.01 | 1299.70 | 3.03 | 611.87 |
| 23 | 88 | 3.58 | 9.23 | 62.08 | 1238.930 | 3.01 | 1250.98 | 3.02 | 609.20 |
| 23 | 89 | 4.75 | 3.90 | 115.4 | 932.193 | 3.01 | 937.83 | 3.04 | 614.53 |
| 23 | 90 | 4.49 | 4.85 | 97.43 | 985.948 | 3.01 | 990.52 | 3.04 | 612.85 |
| 23 | 91 | 8.35 | 4.09 | 83.43 | 529.127 | 3.02 | 536.00 | 3.07 | 598.37 |
| 23 | 92 | 8.60 | 4.90 | 69.79 | 513.286 | 3.02 | 518.86 | 3.08 | 608.71 |
| 23 | 93 | 10.36 | 4.90 | 63.63 | 425.633 | 3.03 | 431.64 | 3.09 | 612.12 |
| 23 | 94 | 10.46 | 2.46 | 119.7 | 421.050 | 3.03 | 423.60 | 3.11 | 612.73 |
| 23 | 95 | 12.50 | 4.54 | 62.13 | 351.873 | 3.03 | 356.65 | 3.12 | 611.87 |
| 23 | 96 | 13.05 | 0.00 | 0.00 | 336.883 | 3.03 | 338.15 | 3.14 | 621.04 |
| 23 | 97 | 12.35 | 2.57 | 105.8 | 356.031 | 3.03 | 359.00 | 3.13 | 616.52 |
| 23 | 98 | 12.92 | 1.88 | 139.3 | 340.311 | 3.03 | 341.77 | 3.14 | 615.71 |
| 23 | 99 | 12.84 | 4.20 | 65.83 | 342.377 | 3.03 | 347.28 | 3.12 | 612.90 |
| 23 | 100 | 13.16 | 1.33 | 192.9 | 333.883 | 3.03 | 335.98 | 3.14 | 621.33 |

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ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

Us

| | | | | | | | | | |
|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 101 | 12.68 | 0.57 | 454.4 | 346.824 | 3.03 | 347.86 | 3.14 | 624.32 |
| 23 | 102 | 12.49 | 3.60 | 76.89 | 352.078 | 3.03 | 356.80 | 3.12 | 618.61 |

UNCAPPED, $\text{Qtz} = 1.0 \text{ g}$, $\text{clinop} = 0.035 \text{ g}$

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C.

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma \text{U} = 50 \text{ ppb}$ (2.14×10^{-7})- equilibrium with atmospheric $\text{CO}_2(\text{g})$; $\text{pCO}_2 = 10^{-3.5}$

- pH range 4.0-9.5

- solution volume = 35 ml

- mass of solid 1, quartz = 1.000 g

- mass of solid 2, clinoptilolite = 35mg (0.035 g)

- M/V = 28.57 g/L

 $M/V \text{ for clinop} = 1.0 \text{ g/L}$

Equipment: Orion 920A pH meter and combination pH electrode

Gyratory shaker

Fisher Marathon 21K centrifuge

Mettler PM4600 and AE240 balances

Packard 2500 TR/AB LSC

Eppendorf micropipettors and tips

Oxford pipettes and tips

Repipettor for transfer of scintillation cocktail

Supplies:

$\text{WS10} \times \text{UR} \times \text{RL} \times \text{RFe} \times \text{HL} \times \leq 325 \text{ mesh}$ AJ
 $\leq 325 \text{ mesh}$ $\text{Qtz} / 100\% \text{ mesh clinoptilolite} \times \text{NaB}$ AJ

50 ppb ^{233}U stock solution

polycarbonate centrifuge tubes with caps (50 ml capacity)

polypropylene centrifuge tubes with caps (50 ml capacity)

pH buffer solutions

Ultima-Gold liquid scintillation cocktail

7 ml scintillation vials

reagent grade NaOH (lot NaHCO_3 (lot 936883) AJconcentrated HNO_3 (lot 983546) HNO_3 and NaHCO_3 or NaOH stock solutions at various concentrations

weighing paper/boats

deionized ultrapure water, DH_2O

Teflon beaker

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M NaNO_3 stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.
- Label 26 polycarbonate centrifuge tubes (50 ml capacity) QCU-pHi (where i is the approximate pH of each solution, see Table QCU).
 - Label 1 polycarbonate centrifuge tube QC1.0U-IU.
 - Weigh and record the weight of each container.
 - About 1L of ^{233}U solution will be required for this experiment.
 - Add ~35 g of the 50 ppb ^{233}U stock solution to each container. Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.

- Record weight of each container.
- B. Add quartz to sample containers.
 - Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the QC1.0-U-IU container.
 - Record weight of each sample container after addition of quartz.
- Adjust pH of each solution by adding HNO_3 or NaHCO_3 .
 - Add the volume of HNO_3 or NaHCO_3 to each experimental container as listed in Table QC1.0U. Take care to add HNO_3 or NaHCO_3 in the concentrations specified in Table CQ0U.
 - For sample QC1.0U-IU (not listed in Table QC1.0U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
 - Weigh and record the weight of each sample container.
- Sample QC10.0U-IU for initial [U].
 - Label two 7 ml LSC vials as QC1.0U-IUa and QC1.0U-IUb.
 - Add 0.5 ml of 0.02 M HNO_3 to each vial.
 - Record weight of each vial.
 - Transfer 0.5 ml of solution from QC1.0U-IU to each sample vial.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of container QC1.0U-IU.
- Wait for equilibrium.
 - Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
 - Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.
- Sample experimental solutions for [U] and pH.
 - For each sample solution label two 7 ml LSC vials (e.g., QC1.0U-pH3a and QC1.0U-pH3b).
 - Add 0.5 ml of 0.02 M HNO_3 to each vial.
 - Record weight of each vial.
 - Record weight of each sample container.
 - From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of each sample container.
 - Measure pH of each experimental solution.
 - Record weight of each sample container after pH measurement.
- Transfer mixture of quartz and clinoptilolite for desorption.

- Record weight of PC sample containers. *Note: this step can be skipped if the mixture transfer is done immediately after the last step in section F.*
 - Centrifuge polycarbonate containers containing experimental solutions at 10,000 rpm for 15 min to remove fine particles from suspension.
 - For each PC sample container, label two corresponding polypropylene (PP) test tubes A and B (50 ml capacity).
 - Record weight for both set of PP tubes A and B.
 - Add 3.0 ml of 0.1M HNO_3 to 50 ml PP tubes labeled as B-QC1.0Ud-pH.
 - Pour supernatant from PC containers into PP tubes with acid and labeled as B-QC1.0Ud-pH leaving behind about 7 to 8ml of solution in PC container with the solid mixture.
 - Record the weight of PC containers containing the solid mixture and some solution.
 - Stir the solid mixture in solution vigorously and immediately transfer all the solid and solution to the corresponding PP tubes marked as A-QC1.0Ud-pH.
 - Record the weight of PP tubes A-QC1.0Ud-pH.
 - Transfer supernatant with HNO_3 acid from the PP tubes B-QC1.0Ud-pH back to original PC containers.
 - Record weights of PC sample containers.
 - Add approximately 3 ml of 0.1 M HNO_3 to PP tubes containing solid mixture, A-QC1.0Ud-pH.
 - Record weights of PP tubes A-QC1.0Ud-pH and PC sample containers.
 - Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.
- L. Sample solutions for [U].
- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QC1.0Ud-pH3a and QC1.0Ud-pH3b).
 - Add 0.5 ml of 0.02 M HNO_3 to each vial.
 - Record weight of each vial.
 - Record weight of each sample container and PP test tube.
 - From each sample container and PP test tube withdraw two 0.5 ml aliquot of solution and transfer to the appropriately labeled LSC vials.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of each sample container and PP test tube.

Table QC1.0U: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with qtz/clinop (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |

UNCAPPED 10/29/98

| Sample Name | Original Container wt., g |
|----------------|---------------------------|
| QC1.0U-pH 3.0 | 22.3323 |
| QC1.0U-pH 3.5 | 22.5936 |
| QC1.0U-pH 4.0 | 22.2613 |
| QC1.0U-pH 4.25 | 22.3276 |
| QC1.0U-pH 4.5 | 22.5725 |
| QC1.0U-pH 4.75 | 22.3045 |
| QC1.0U-pH 5.0 | 22.3232 |
| QC1.0U-pH 5.25 | 22.2925 |
| QC1.0U-pH 5.5 | 22.3033 |
| QC1.0U-pH 5.75 | 22.2452 |
| QC1.0U-pH 6.0 | 22.4783 |
| QC1.0U-pH 6.25 | 22.6249 |
| QC1.0U-pH 6.5 | 22.3593 |
| QC1.0U-pH 6.75 | 22.3052 |
| QC1.0U-pH 7.0 | 22.3182 |
| QC1.0U-pH 7.25 | 22.2863 |
| QC1.0U-pH 7.5 | 22.2375 |
| QC1.0U-pH 7.75 | 22.3816 |
| QC1.0U-pH 8.0 | 22.4682 |
| QC1.0U-pH 8.25 | 22.2326 |
| QC1.0U-pH 8.5 | 22.3215 |
| QC1.0U-pH 8.75 | 22.3988 |
| QC1.0U-pH 9.0 | 22.5997 |
| QC1.0U-pH 9.25 | 22.3002 |
| QC1.0U-pH 9.5 | 22.3150 |

QC1.0U - IV

22.3536

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10/29/98

| Sample Name | Container wt. + U soln., g |
|----------------|----------------------------|
| QC1.0U-pH 3.0 | 57.3498 |
| QC1.0U-pH 3.5 | 57.6082 |
| QC1.0U-pH 4.0 | 57.2715 |
| QC1.0U-pH 4.25 | 57.3226 |
| QQC1.0U-pH 4.5 | 57.5683 |
| QC1.0U-pH 4.75 | 57.3194 |
| QC1.0U-pH 5.0 | 57.3303 |
| QC1.0U-pH 5.25 | 57.3048 |
| QC1.0U-pH 5.5 | 57.3172 |
| QC1.0U-pH 5.75 | 57.2456 |
| QC1.0U-pH 6.0 | 57.4883 |
| QC1.0U-pH 6.25 | 57.6329 |
| QC1.0U-pH 6.5 | 57.3567 |
| QC1.0U-pH 6.75 | 57.3074 |
| QC1.0U-pH 7.0 | 57.3292 |
| QC1.0U-pH 7.25 | 57.2989 |
| QC1.0U-pH 7.5 | 57.2472 |
| QC1.0U-pH 7.75 | 57.3776 |
| QC1.0U-pH 8.0 | 57.4807 |
| QC1.0U-pH 8.25 | 57.2246 |
| QC1.0U-pH 8.5 | 57.3261 |
| QC1.0U-pH 8.75 | 57.3937 |
| QC1.0U-pH 9.0 | 57.6052 |
| QC1.0U-pH 9.25 | 57.3033 |
| QC1.0U-pH 9.5 | 57.3746 |

QC1.0U-IV

57.3519

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| Sample Name | Container wt. + U soln. + quartz, g |
|----------------|-------------------------------------|
| QC1.0U-pH 3.0 | 58.3501 |
| QC1.0U-pH 3.5 | 58.6074 |
| QC1.0U-pH 4.0 | 58.2584 |
| QC1.0U-pH 4.25 | 58.3088 |
| QQC1.0U-pH 4.5 | 58.5582 |
| QC1.0U-pH 4.75 | 58.3055 |
| QC1.0U-pH 5.0 | 58.3179 |
| QC1.0U-pH 5.25 | 58.2924 |
| QC1.0U-pH 5.5 | 58.3058 |
| QC1.0U-pH 5.75 | 58.2311 |
| QC1.0U-pH 6.0 | 58.4688 |
| QC1.0U-pH 6.25 | 58.6205 |
| QC1.0U-pH 6.5 | 58.3415 |
| QC1.0U-pH 6.75 | 58.2986 |
| QC1.0U-pH 7.0 | 58.3151 |
| QC1.0U-pH 7.25 | 58.2867 |
| QC1.0U-pH 7.5 | 58.2346 |
| QC1.0U-pH 7.75 | 58.3625 |
| QC1.0U-pH 8.0 | 58.4670 |
| QC1.0U-pH 8.25 | 58.2119 |
| QC1.0U-pH 8.5 | 58.3095 |
| QC1.0U-pH 8.75 | 58.3805 |
| QC1.0U-pH 9.0 | 58.5926 |
| QC1.0U-pH 9.25 | 58.2903 |
| QC1.0U-pH 9.5 | 58.3620 |

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11/3/98

| Sample Name | Container wt. + U soln. + quartz + clinop, g |
|----------------|--|
| QC1.0U-pH 3.0 | 58.3604 |
| QC1.0U-pH 3.5 | 58.6108 |
| QC1.0U-pH 4.0 | 58.2833 |
| QC1.0U-pH 4.25 | 58.3387 |
| QQC1.0U-pH 4.5 | 58.5839 |
| QC1.0U-pH 4.75 | 58.3355 |
| QC1.0U-pH 5.0 | 58.3483 |
| QC1.0U-pH 5.25 | 58.3204 |
| QC1.0U-pH 5.5 | 58.3346 |
| QC1.0U-pH 5.75 | 58.2551 |
| QC1.0U-pH 6.0 | 58.4936 |
| QC1.0U-pH 6.25 | 58.6491 |
| QC1.0U-pH 6.5 | 58.3673 |
| QC1.0U-pH 6.75 | 58.3270 |
| QC1.0U-pH 7.0 | 58.3454 |
| QC1.0U-pH 7.25 | 58.3157 |
| QC1.0U-pH 7.5 | 58.2646 |
| QC1.0U-pH 7.75 | 58.3900 |
| QC1.0U-pH 8.0 | 58.4969 |
| QC1.0U-pH 8.25 | 58.2402 |
| QC1.0U-pH 8.5 | 58.3385 |
| QC1.0U-pH 8.75 | 58.4080 |
| QC1.0U-pH 9.0 | 58.6189 |
| QC1.0U-pH 9.25 | 58.3206 |
| QC1.0U-pH 9.5 | 58.3997 |

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11/4/98

| Sample Name | Container Wt. After additions of HNO3 and NaHCO3, g |
|----------------|---|
| QC1.0U-pH 3.0 | 58.7452 |
| QC1.0U-pH 3.5 | 58.7067 |
| QC1.0U-pH 4.0 | 58.3416 |
| QC1.0U-pH 4.25 | 58.4154 |
| QQC1.0U-pH 4.5 | 58.8661 |
| QC1.0U-pH 4.75 | 58.5315 |
| QC1.0U-pH 5.0 | 58.5808 |
| QC1.0U-pH 5.25 | 58.5730 |
| QC1.0U-pH 5.5 | 58.6044 |
| QC1.0U-pH 5.75 | 58.53840 |
| QC1.0U-pH 6.0 | 58.7796 |
| QC1.0U-pH 6.25 | 58.9588 |
| QC1.0U-pH 6.5 | 58.4221 |
| QC1.0U-pH 6.75 | 58.3991 |
| QC1.0U-pH 7.0 | 58.4373 |
| QC1.0U-pH 7.25 | 58.4479 |
| QC1.0U-pH 7.5 | 58.4668 |
| QC1.0U-pH 7.75 | 58.5466 |
| QC1.0U-pH 8.0 | 58.7709 |
| QC1.0U-pH 8.25 | 58.3415 |
| QC1.0U-pH 8.5 | 58.5696 58.5036 |
| QC1.0U-pH 8.75 | 58.9232 58.5696 |
| QC1.0U-pH 9.0 | 58.1999 9232 |
| QC1.0U-pH 9.25 | 59.1999 |
| QC1.0U-pH 9.5 | 59.6589 |
| QC1.0U-IV | 57.4526 |

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11/14/98

11/17/98

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| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|----------------|--|-------------|---|
| QC1.0U-pH 3.0 | 58.4578 | 2.96 | 57.4506 |
| QC1.0U-pH 3.5 | 58.4542 | 3.51 | 57.4467 |
| QC1.0U-pH 4.0 | 58.0686 | 4.16 | 57.0160 |
| QC1.0U-pH 4.25 | 58.1245 | 4.49 | 57.1252 |
| QC1.0U-pH 4.5 | 58.6345 | 4.84 | 57.6217 |
| QC1.0U-pH 4.75 | 58.2099 | 5.64 | 57.1951 |
| QC1.0U-pH 5.0 | 58.2613 | 6.09 | 57.2473 |
| QC1.0U-pH 5.25 | 58.3060 | 6.21 | 57.3009 |
| QC1.0U-pH 5.5 | 58.3167 | 6.34 | 57.2888 |
| QC1.0U-pH 5.75 | 58.1578 | 6.36 | 57.1462 |
| QC1.0U-pH 6.0 | 58.3896 | 6.47 | 57.3805 |
| QC1.0U-pH 6.25 | 58.7341 | 6.57 | 57.7327 |
| QC1.0U-pH 6.5 | 58.1691 | 6.58 | 57.1520 |
| QC1.0U-pH 6.75 | 58.0652 | 6.77 | 57.0488 |
| QC1.0U-pH 7.0 | 58.0944 | 7.05 | 57.0639 |
| QC1.0U-pH 7.25 | 58.1822 | 7.21 | 57.1822 |
| QC1.0U-pH 7.5 | 58.1020 | 7.39 | 57.1077 |
| QC1.0U-pH 7.75 | 58.1832 | 7.63 | 57.1763 |
| QC1.0U-pH 8.0 | 58.5972 | 7.85 | 57.5905 |
| QC1.0U-pH 8.25 | 58.0773 | 8.16 | 57.0687 |
| QC1.0U-pH 8.5 | 58.2145 | 8.36 | 57.1937 |
| QC1.0U-pH 8.75 | 58.3393 | 8.65 | 57.3441 |
| QC1.0U-pH 9.0 | 58.6522 | 8.86 | 57.6730 |
| QC1.0U-pH 9.25 | 58.9036 | 9.17 | 57.9134 |
| QC1.0U-pH 9.5 | 58.4413 | 9.09 | 58.4278 |
| QC0U-IU | 57.4063 | | 56.4068 |

UNCAPPED SORPTION

11/16/98

11/17/98 & 11/18/98

| Sample Name | wt. of vial, g | wt. Of vial + sample |
|-----------------|----------------|----------------------|
| QC1.0U-pH 3a | 7.6749 | 8.1639 |
| QC1.0U-pH 3b | 7.7212 | 8.2071 |
| QC1.0U-pH 3.5a | 7.412 | 8.2302 |
| QC1.0U-pH 3.5b | 7.7404 | 8.2300 |
| QC1.0U-pH 4a | 7.6736 | 8.1759 |
| QC1.0U-pH 4b | 7.7338 | 8.2493 |
| QC1.0U-pH 4.25a | 7.7604 | 8.2475 |
| QC1.0U-pH 4.25b | 7.6533 | 8.1390 |
| QC1.0U-pH 4.5a | 7.7470 | 8.2336 |
| QC1.0U-pH 4.5b | 7.6947 | 8.1852 |
| QC1.0U-pH 4.75a | 7.7123 | 8.2037 |
| QC1.0U-pH 4.75b | 7.6622 | 8.1556 |
| QC1.0U-pH 5a | 7.6953 | 8.1865 |
| QC1.0U-pH 5b | 7.7235 | 8.1848 |
| QC1.0U-pH 5.25a | 7.6947 | 8.2141 |
| QC1.0U-pH 5.25b | 7.7330 | 8.1847 |
| QC1.0U-pH 5.5a | 7.7005 | 8.2229 |
| QC1.0U-pH 5.5b | 7.6860 | 8.1916 |
| QC1.0U-pH 5.75a | 7.6626 | 8.1735 |
| QC1.0U-pH 5.75b | 7.7334 | 8.1509 |
| QC1.0U-pH 6a | 7.6933 | 8.2257 |
| QC1.0U-pH 6b | 7.7031 | 8.1840 |
| QC1.0U-pH 6.25a | 7.6848 | 8.1915 |
| QC1.0U-pH 6.25b | 7.7130 | 8.1704 |
| QC1.0U-pH 6.5a | 7.7672 | 8.2046 |
| QC1.0U-pH 6.5b | 7.7036 | 8.2546 |
| QC1.0U-pH 6.75a | 7.7234 | 8.1956 |
| QC1.0U-pH 6.75b | 7.7098 | 8.2136 |
| QC1.0U-pH 7a | 7.7537 | 8.1982 |
| QC1.0U-pH 7b | 7.7095 | 8.2454 |
| QC1.0U-pH 7.25a | 7.7085 | 8.1990 |
| QC1.0U-pH 7.25b | 7.7117 | 8.1956 |
| QC1.0U-pH 7.5a | 7.6967 | 8.1992 |
| QC1.0U-pH 7.5b | 7.7081 | 8.1835 |
| QC1.0U-pH 7.75a | 7.7035 | 8.1959 |
| QC1.0U-pH 7.75b | 7.6715 | 8.1942 |
| QC1.0U-pH 8a | 7.7261 | 8.1614 |
| QC1.0U-pH 8b | 7.6760 | 8.2172 |
| QC1.0U-pH 8.25a | 7.7275 | 8.1641 |
| QC1.0U-pH 8.25b | 7.7877 | 8.2159 |
| QC1.0U-pH 8.5a | 7.6621 | 8.2765 |
| QC1.0U-pH 8.5b | 7.7261 | 8.1468 |
| QC1.0U-pH 8.75a | 7.7254 | 8.2183 |
| QC1.0U-pH 8.75b | 7.7376 | 8.2108 |
| QC1.0U-pH 9a | 7.6924 | 8.2165 |
| QC1.0U-pH 9b | 7.7247 | 8.1661 |
| QC1.0U-pH 9.25a | 7.7199 | 8.2075 |
| QC1.0U-pH 9.25b | 7.7199 | 8.2010 |
| QC1.0U-pH 9.5a | 7.7182 | 8.2041 |
| QC1.0U-pH 9.5b | 7.6947 | 8.1816 |
| QC1.0U-Iua | 7.7230 | 8.2215 |
| QC1.0U-IUb | 7.6941 | 8.1970 |
| QC1.0U-IUc | 7.7227 | 8.2266 |

19 Nov 98 08:46

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

Pac

User :

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.34 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.77 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 30.19 | |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

Qc1.0 U Sorption

Qtz & Clinoptilolite (0.035g)

M/V = 1 g/L

UNCAPPED

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|------|------------|------------|------------|---------------------------------|
| P# | 1 | 999.99 | 20.34 | 1.40 | 3.770 3.26 30.19 1.15 149.31 |
| 23 | 2 | 8.56 | 0.80 | 392.7 | 515.973 3.02 519.11 3.09 637.70 |
| 23 | 3 | 8.28 | 2.61 | 128.2 | 532.945 3.02 536.96 3.08 617.88 |
| 23 | 4 | 8.37 | 1.28 | 251.4 | 527.532 3.02 529.91 3.09 632.39 |
| 23 | 5 | 8.75 | 2.06 | 156.0 | 504.573 3.02 506.73 3.09 630.49 |
| 23 | 6 | 9.27 | 0.00 | 0.00 | 476.273 3.02 475.10 3.11 635.98 |
| 23 | 7 | 8.86 | 0.43 | 721.4 | 498.262 3.02 500.51 3.09 635.72 |
| 23 | 8 | 8.85 | 4.40 | 76.20 | 498.377 3.02 502.58 3.09 629.81 |
| 23 | 9 | 9.24 | 0.87 | 349.5 | 477.182 3.02 478.79 3.10 632.55 |
| 23 | 10 | 9.02 | 0.83 | 369.0 | 489.135 3.02 490.77 3.10 631.39 |
| 23 | 11 | 10.62 | 0.19 | 1502 | 414.686 3.03 415.76 3.12 640.85 |
| 23 | 12 | 10.56 | 2.76 | 107.5 | 417.442 3.03 419.43 3.11 634.91 |
| 23 | 13 | 12.96 | 0.00 | 0.00 | 339.208 3.03 338.64 3.15 639.96 |
| 23 | 14 | 12.87 | 1.88 | 140.5 | 341.762 3.03 343.01 3.14 635.67 |
| 23 | 15 | 28.74 | 1.61 | 109.7 | 150.858 3.08 153.25 3.30 635.91 |
| 23 | 16 | 27.66 | 0.00 | 0.00 | 157.004 3.07 156.87 3.32 636.22 |
| 23 | 17 | 39.72 | 0.00 | 0.00 | 108.113 3.11 109.41 3.44 637.46 |
| 23 | 18 | 39.70 | 0.26 | 557.8 | 108.170 3.11 108.73 3.46 636.24 |
| 23 | 19 | 42.37 | 0.00 | 0.00 | 101.116 3.11 101.01 3.50 636.27 |
| 23 | 20 | 41.46 | 1.20 | 122.7 | 103.442 3.11 105.10 3.45 627.89 |
| 23 | 21 | 41.82 | 0.18 | 815.5 | 102.519 3.11 102.93 3.48 633.92 |
| 23 | 22 | 41.34 | 1.16 | 126.4 | 103.753 3.11 105.66 3.45 630.95 |
| 23 | 23 | 42.17 | 0.19 | 731.6 | 101.613 3.11 101.71 3.49 633.29 |
| 23 | 24 | 42.31 | 0.01 | 16476 | 101.335 3.11 101.93 3.48 633.84 |
| 23 | 25 | 40.54 | 1.14 | 129.7 | 105.850 3.11 107.85 3.44 631.05 |
| 23 | 26 | 38.76 | 0.76 | 197.0 | 110.884 3.10 112.02 3.43 628.76 |
| 23 | 27 | 44.66 | 0.66 | 211.6 | 95.760 3.12 96.75 3.50 630.71 |
| 23 | 28 | 43.12 | 0.69 | 205.7 | 99.291 3.12 100.75 3.48 633.04 |
| 23 | 29 | 37.44 | 0.00 | 0.00 | 114.927 3.10 115.22 3.43 636.91 |
| 23 | 30 | 37.86 | 0.84 | 180.9 | 113.715 3.10 114.50 3.43 635.43 |
| 23 | 31 | 34.81 | 2.70 | 61.22 | 123.894 3.09 127.41 3.35 626.28 |
| 23 | 32 | 35.49 | 1.69 | 94.59 | 121.448 3.09 123.04 3.39 628.70 |

B

std.

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 33 | 33.21 | 0.95 | 171.7 | 130.075 | 3.09 | 130.22 | 3.39 | 633.25 |
| 23 | 34 | 31.97 | 0.87 | 190.9 | 135.298 | 3.08 | 136.16 | 3.36 | 634.74 |
| 23 | 35 | 28.01 | 0.00 | 0.00 | 154.959 | 3.07 | 155.17 | 3.32 | 637.80 |
| 23 | 36 | 27.64 | 1.37 | 131.4 | 157.011 | 3.07 | 158.89 | 3.30 | 632.12 |
| 23 | 37 | 22.95 | 0.00 | 0.00 | 189.912 | 3.06 | 190.16 | 3.26 | 642.67 |
| 23 | 38 | 22.61 | 2.22 | 91.09 | 192.780 | 3.06 | 195.29 | 3.24 | 634.01 |
| 23 | 39 | 18.49 | 1.08 | 201.8 | 236.738 | 3.05 | 238.55 | 3.20 | 637.57 |
| 23 | 40 | 18.83 | 0.42 | 500.4 | 232.236 | 3.05 | 232.42 | 3.22 | 636.52 |
| 23 | 41 | 13.72 | 1.89 | 135.6 | 320.210 | 3.04 | 322.22 | 3.15 | 634.54 |

20 Nov 98 02:31

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 13.45 | 0.92 | 274.3 | 326.862 | 3.03 | 328.55 | 3.15 | 636.14 |
| 23 | 43 | 10.09 | 0.27 | 1051 | 436.963 | 3.03 | 436.91 | 3.12 | 630.48 |
| 23 | 44 | 10.58 | 0.83 | 342.3 | 416.268 | 3.03 | 417.92 | 3.12 | 636.81 |
| 23 | 45 | 9.30 | 2.45 | 128.1 | 474.510 | 3.02 | 476.26 | 3.10 | 635.52 |
| 23 | 46 | 9.18 | 0.79 | 385.0 | 480.653 | 3.02 | 481.25 | 3.10 | 636.69 |
| 23 | 47 | 8.86 | 4.38 | 76.60 | 497.923 | 3.02 | 502.54 | 3.09 | 623.47 |
| 23 | 48 | 8.90 | 0.00 | 0.00 | 495.781 | 3.02 | 495.77 | 3.10 | 638.60 |
| 23 | 49 | 9.03 | 4.35 | 76.24 | 488.367 | 3.02 | 492.29 | 3.09 | 636.93 |
| 23 | 50 | 9.08 | 2.79 | 115.0 | 486.208 | 3.02 | 488.97 | 3.09 | 638.73 |
| 23 | 51 | 9.30 | 0.52 | 579.7 | 474.725 | 3.02 | 475.40 | 3.10 | 638.70 |
| 23 | 52 | 9.12 | 1.81 | 173.1 | 483.620 | 3.02 | 485.27 | 3.10 | 636.86 |
| 23 | 53 | 9.11 | 1.61 | 193.3 | 484.814 | 3.02 | 487.16 | 3.09 | 645.97 |
| 23 | 54 | 9.19 | 0.22 | 1338 | 480.234 | 3.02 | 480.37 | 3.10 | 644.18 |

231 MISSING TUBE(S) 2/10/99

24 8.39 4.33 79.12 526.271 3.02 530.54 3.08 625.90

23 25 8.58 4.72 72.33 514.656 3.02 519.30 3.08 610.53

23 26 8.58 1.22 258.8 514.889 3.02 516.74 3.09 603.02

23 27 8.31 2.64 125.9 531.492 3.02 535.81 3.08 595.86

23 28 8.23 0.00 0.00 536.814 3.02 537.18 3.09 603.81

23 29 8.26 0.00 0.00 534.852 3.02 534.39 3.09 606.93

The samples were resampled from Qc1.0U-1V container & counted. The wts. are written below.

231 MISSING TUBE(S) 2/20/99

96 8.87 1.74 180.0 497.663 3.02 500.74 3.09 632.72

23 97 8.49 0.47 656.8 520.329 3.02 520.46 3.09 615.99

23 98 8.32 1.01 315.1 531.512 3.02 534.22 3.08 607.12

23 99 8.20 1.44 225.2 538.487 3.02 540.39 3.09 601.79

23 100 8.43 1.45 220.5 524.175 3.02 526.03 3.09 604.74

23 101 8.41 0.55 570.8 524.955 3.02 524.62 3.09 609.95

2/8/99

wt. of vial

wt. of vial + sample

Qc1.0U-IVc 7.8733

8.3816

Qc1.0U-IVd 7.8780

8.3842

Qc1.0U-IVe 7.8377

8.3458

Qc1.0U-IVf 7.9457

8.4528

Qc1.0U
uncapped
Recounted
on 2/10/99These samples
were recounted
on 2/20/99
No resampling
was done

DESORPTION, UNCAPPED

11/18/98

11/18/98

11/18/98

11/18/98

11/18/98

| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after transfer of qtz/clino, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. After acid add & supernatant retransfer, g |
|----------------|--------------------|--|-----------------------------------|--|--|
| QC1.0U-pH 3.0 | 13.6074 | 18.1869 | 21.2190 | 26.9476 | 55.8491 |
| QC1.0U-pH 3.5 | 13.2172 | 18.4885 | 21.5188 | 27.8934 | 55.1699 |
| QC1.0U-pH 4.0 | 13.6698 | 18.0004 | 21.0235 | 26.6494 | 55.6086 |
| QC1.0U-pH 4.25 | 13.2340 | 18.2879 | 21.3129 | 27.4393 | 54.9294 |
| QC1.0U-pH 4.5 | 13.2506 | 19.6177 | 22.6376 | 29.0415 | 54.1688 |
| QC1.0U-pH 4.75 | 13.6424 | 19.4899 | 22.5126 | 28.1950 | 54.2535 |
| QC1.0U-pH 5.0 | 13.6963 | 20.4197 | 23.4410 | 29.0820 | 53.4536 |
| QC1.0U-pH 5.25 | 13.9760 | 20.8086 | 23.8266 | 29.1618 | 53.3485 |
| QC1.0U-pH 5.5 | 13.2502 | 19.6975 | 22.7103 | 28.7897 | 53.7715 |
| QC1.0U-pH 5.75 | 13.8666 | 20.0620 | 23.0834 | 28.5026 | 53.8624 |
| QC1.0U-pH 6.0 | 13.6487 | 19.3171 | 22.3251 | 28.2655 | 54.5177 |
| QC1.0U-pH 6.25 | 13.7516 | 21.2215 | 24.2522 | 30.1779 | 53.2249 |
| QC1.0U-pH 6.5 | 13.6656 | 21.2442 | 24.2692 | 30.0263 | 52.5027 |
| QC1.0U-pH 6.75 | 13.7502 | 20.8399 | 23.8688 | 29.4774 | 52.9202 |
| QC1.0U-pH 7.0 | 13.2876 | 18.4619 | 21.4851 | 27.5609 | 54.8337 |
| QC1.0U-pH 7.25 | 13.7078 | 20.3383 | 23.3622 | 29.0057 | 53.5182 |
| QC1.0U-pH 7.5 | 13.1775 | 19.0609 | 22.0825 | 28.2165 | 54.2846 |
| QC1.0U-pH 7.75 | 13.1655 | 18.6224 | 21.6501 | 27.9263 | 54.6694 |
| QC1.0U-pH 8.0 | 13.1787 | 19.7807 | 22.8098 | 29.1449 | 53.9198 |
| QC1.0U-pH 8.25 | 13.2808 | 18.5994 | 21.6295 | 27.6297 | 54.7335 |
| QC1.0U-pH 8.5 | 13.6522 | 18.9538 | 21.9886 | 27.7047 | 54.8375 |
| QC1.0U-pH 8.75 | 13.1900 | 21.1200 | 24.1608 | 30.4509 | 52.3354 |
| QC1.0U-pH 9.0 | 13.2779 | 20.2215 | 23.2595 | 29.6807 | 53.6661 |
| QC1.0U-pH 9.25 | 13.7046 | 19.7087 | 22.7398 | 28.4268 | 54.7739 |
| QC1.0U-pH 9.5 | 13.1839 | 19.7935 | 22.8205 | 29.1532 | 54.7573 |
| QC0U-pH 9.25 | | | | | |
| QC0U-pH 9.5 | | | | | |

DESORPTION (UNCAPPED)

11/30/98

12/2/98

11/30/98

12/1/98

| Sample Name | Container wt. before sampling | Container wt. after sampling | wt. Of PP tubes before sampling | wt. Of PP tubes after sampling |
|----------------|-------------------------------|------------------------------|---------------------------------|--------------------------------|
| QC1.0U-pH 3.0 | 55.7510 | 54.742 | 21.2156 | 20.2399 |
| QC1.0U-pH 3.5 | 55.0891 | 54.0717 | 21.5070 | 20.5207 |
| QC1.0U-pH 4.0 | 55.5337 | 54.5242 | 21.0149 | 20.0290 |
| QC1.0U-pH 4.25 | 54.8747 | 54.8745 | 21.3076 | 20.3271 |
| QC1.0U-pH 4.5 | 54.0585 | 53.0461 | 22.6329 | 21.6454 |
| QC1.0U-pH 4.75 | 54.2045 | 53.2062 | 22.5054 | 20.5900 |
| QC1.0U-pH 5.0 | 53.4009 | 52.4016 | 22.4356 | 21.5124 |
| QC1.0U-pH 5.25 | 53.2642 | 52.4016 | 23.8220 | 22.8589 |
| QC1.0U-pH 5.5 | 53.7242 | 52.2617 | 23.7041 | 22.6934 |
| QC1.0U-pH 5.75 | 53.7459 | 52.7232 | 23.0758 | 22.0827 |
| QC1.0U-pH 6.0 | 54.4303 | 52.7499 | 22.2782 | 21.2710 |
| QC1.0U-pH 6.25 | 53.1504 | 53.4269 | 24.2281 | 23.2315 |
| QC1.0U-pH 6.5 | 52.4356 | 52.1440 | 24.2515 | 23.2518 |
| QC1.0U-pH 6.75 | 52.8512 | 51.4308 | 23.8650 | 22.8771 |
| QC1.0U-pH 7.0 | 54.7518 | 51.8475 | 21.4627 | 20.4793 |
| QC1.0U-pH 7.25 | 53.4725 | 53.7529 | 23.3532 | 22.3478 |
| QC1.0U-pH 7.5 | 54.2304 | 52.4725 | 22.0626 | 21.0621 |
| QC1.0U-pH 7.75 | 54.5932 | 53.2245 | 21.6423 | 20.6539 |
| QC1.0U-pH 8.0 | 53.8649 | 53.5711 | 22.7748 | 21.8359 |
| QC1.0U-pH 8.25 | 54.5927 | 52.8671 | 21.6241 | 20.6558 |
| QC1.0U-pH 8.5 | 54.7684 | 53.7621 | 21.7243 | 20.7702 |
| QC1.0U-pH 8.75 | 52.2589 | 51.2555 | 24.1296 | 23.1666 |
| QC1.0U-pH 9.0 | 53.5708 | 52.5604 | 23.2536 | 22.3062 |
| QC1.0U-pH 9.25 | 54.6178 | 53.6050 | 22.7312 | 21.7886 |
| QC1.0U-pH 9.5 | 54.6490 | 53.6358 | 22.8073 | 21.8445 |

| PP tubes | | Desorption | UNCAPPED | PC containers | | Desorption |
|-----------------|---------------|----------------------|----------|---------------|----------------------|------------|
| Sample Name | wt. of vial,g | wt. Of vial + sample | | wt. of vial,g | wt. Of vial + sample | |
| QC1.0U-pH 3a | 7.7046 | 8.1811 | | 7.7172 | 8.2103 | |
| QC1.0U-pH 3b | 7.7372 | 8.2138 | | 7.6990 | 8.1941 | |
| QC1.0U-pH 3.5a | 7.7297 | 8.2072 | | 7.6884 | 8.1844 | |
| QC1.0U-pH 3.5b | 7.7122 | 8.1947 | | 7.6954 | 8.1926 | |
| QC1.0U-pH 4a | 7.7444 | 8.2231 | | 7.7039 | 8.2062 | |
| QC1.0U-pH 4b | 7.7337 | 8.2166 | | 7.6942 | 8.1892 | |
| QC1.0U-pH 4.25a | 7.7075 | 8.1853 | | 7.6565 | 8.1509 | |
| QC1.0U-pH 4.25b | 7.7284 | 8.2090 | | 7.7308 | 8.2262 | |
| QC1.0U-pH 4.5a | 7.7223 | 8.2015 | | 7.7135 | 8.2090 | |
| QC1.0U-pH 4.5b | 7.6644 | 8.1447 | | 7.6628 | 8.1601 | |
| QC1.0U-pH 4.75a | 7.6758 | 8.1657 | | 7.7014 | 8.1960 | |
| QC1.0U-pH 4.75b | 7.6701 | 8.1334 | | 7.6758 | 8.1703 | |
| QC1.0U-pH 5a | 7.5932 | 8.0894 | | 7.6814 | 8.1761 | |
| QC1.0U-pH 5b | 7.6435 | 8.1416 | | 7.6976 | 8.1925 | |
| QC1.0U-pH 5.25a | 7.6899 | 8.1723 | | 7.6402 | 8.1334 | |
| QC1.0U-pH 5.25b | 7.6973 | 8.2135 | | 7.6953 | 8.1889 | |
| QC1.0U-pH 5.5a | 7.7089 | 8.1710 | | 7.6876 | 8.1821 | |
| QC1.0U-pH 5.5b | 7.6996 | 8.1290 | | 7.6655 | 8.1593 | |
| QC1.0U-pH 5.75a | 7.7021 | 8.1944 | | 7.6527 | 8.1476 | |
| QC1.0U-pH 5.75b | 7.7203 | 8.2179 | | 7.6447 | 8.1406 | |
| QC1.0U-pH 6a | 7.7457 | 8.1925 | | 7.6297 | 8.1260 | |
| QC1.0U-pH 6b | 7.7070 | 8.2362 | | 7.5936 | 8.0901 | |
| QC1.0U-pH 6.25a | 7.7137 | 8.1984 | | 7.6925 | 8.1903 | |
| QC1.0U-pH 6.25a | 7.7205 | 8.2040 | | 7.7158 | 8.2139 | |
| QC1.0U-pH 6.5a | 7.7623 | 8.2107 | | 7.8063 | 8.3008 | |
| QC1.0U-pH 6.5b | 7.6984 | 8.2548 | | 7.7380 | 8.2330 | |
| QC1.0U-pH 6.75a | 7.7437 | 8.1852 | | 7.6514 | 8.1507 | |
| QC1.0U-pH 6.75b | 7.8052 | 8.2326 | | 7.6745 | 8.1715 | |
| QC1.0U-pH 7a | 7.7063 | 8.2814 | | 7.7152 | 8.2122 | |
| QC1.0U-pH 7b | 7.7099 | 8.1846 | | 7.6836 | 8.1779 | |
| QC1.0U-pH 7.25a | 7.6696 | 8.1892 | | 7.7102 | 8.2072 | |
| QC1.0U-pH 7.25b | 7.6625 | 8.1452 | | 7.6961 | 8.1932 | |
| QC1.0U-pH 7.5a | 7.7073 | 8.1401 | | 7.7460 | 8.2473 | |
| QC1.0U-pH 7.5b | 7.7083 | 8.0547 | | 7.7422 | 8.2381 | |
| QC1.0U-pH 7.75a | 7.7478 | 8.2171 | | 7.7584 | 8.2574 | |
| QC1.0U-pH 7.75b | 7.6505 | 8.1810 | | 7.6751 | 8.1720 | |
| QC1.0U-pH 8a | 7.6858 | 8.1058 | | 7.7643 | 8.2608 | |
| QC1.0U-pH 8b | 7.7029 | 8.1376 | | 7.6958 | 8.1905 | |
| QC1.0U-pH 8.25a | 7.6918 | 8.1613 | | 7.6945 | 8.1916 | |
| QC1.0U-pH 8.25b | 7.7605 | 8.1495 | | 7.7017 | 8.1944 | |
| QC1.0U-pH 8.5a | 7.7299 | 8.2286 | | 7.7541 | 8.2542 | |
| QC1.0U-pH 8.5b | 7.7308 | 8.1984 | | 7.7564 | 8.2562 | |
| QC1.0U-pH 8.75a | 7.7623 | 8.1962 | | 7.6823 | 8.1801 | |
| QC1.0U-pH 8.75b | 7.6470 | 8.2268 | | 7.6862 | 8.1836 | |
| QC1.0U-pH 9a | 7.7381 | 8.1108 | | 7.7553 | 8.2530 | |
| QC1.0U-pH 9b | 7.6981 | 8.2077 | | 7.6511 | 8.1455 | |
| QC1.0U-pH 9.25a | 7.6767 | 8.1598 | | 7.6330 | 8.1322 | |
| QC1.0U-pH 9.25 | 7.6248 | 8.1358 | | 7.5915 | 8.0908 | |
| QC1.0U-pH 9.5a | 7.7487 | 8.0988 | | 7.6618 | 8.1608 | |
| QC1.0U-pH 9.5b | 7.7360 | 8.2194 | | 7.6809 | 8.1767 | |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 02-Dec-98 11:39

C14 Eff (0-156 keV) = 96.58 %

C14 CHI SQUARE IPA DATA PROCESSED - 02-Dec-98 11:50

C14 Chi Square = 23.43

H3 IPA DATA PROCESSED - 02-Dec-98 11:51

H3 Eff (0-18.6 keV) = 65.31 %

H3 CHI SQUARE IPA DATA PROCESSED - 02-Dec-98 12:01

H3 Chi Square = 13.88

BKG IPA DATA PROCESSED - 02-Dec-98 13:02

Bkg (0-18.6 keV) = 22.60 cpm

Bkg (0-156 keV) = 32.35 cpm

WARNING: Questionable C14 Background value - Please view historic data

WARNING: Questionable C14 Figure of Merit value - Please view historic

C14 E²/B (1-156 keV) = 374.01H3 E²/B (1-18.6 keV) = 187.68

03 Dec 98 05:51

ALPHA/BETA - 1.02

Page

Protocol #:23

U-233 3% 2 sigma

User : A

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.18 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.57 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.59 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

QC1.0U uncappped desorption

Q.C.1.0V, desorption tubes uncapped

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|--------|-------------|---------------|--------------|----------|
| P# 1 | 999.99 | 20.18 1.41 | 3.574 3.35 | 29.59 1.16 | 145.04 |
| 23 2 | 14.42 | 1.25 195.8 | 304.609 3.04 | 306.88 3.15 | 623.80 |
| 23 3 | 14.26 | 2.76 92.60 | 308.137 3.03 | 312.41 3.14 | 625.08 |
| 23 4 | 11.49 | 0.45 596.9 | 383.632 3.03 | 385.29 3.12 | 625.49 |
| 23 5 | 11.91 | 2.58 107.8 | 349.810 3.03 | 373.85 3.11 | 621.24 |
| 23 6 | 8.46 | 2.40 136.6 | 521.958 3.02 | 526.32 3.08 | 621.71 |
| 23 7 | 8.60 | 3.20 103.5 | 513.868 3.02 | 517.85 3.08 | 618.54 |
| 23 8 | 6.85 | 2.45 148.7 | 645.769 3.02 | 649.39 3.07 | 624.99 |
| 23 9 | 6.75 | 4.42 86.66 | 655.685 3.01 | 660.48 3.06 | 619.95 |
| 23 10 | 5.45 | 2.39 170.4 | 812.022 3.01 | 814.44 3.06 | 621.99 |
| 23 11 | 5.32 | 2.57 161.3 | 831.764 3.01 | 834.51 3.05 | 619.05 |
| 23 12 | 3.05 | 7.04 84.98 | 1455.115 3.01 | 1466.15 3.02 | 619.91 |
| 23 13 | 3.13 | 4.43 126.9 | 1416.234 3.01 | 1419.93 3.03 | 621.95 |
| 23 14 | 2.96 | 0.10 5513 | 1499.129 3.01 | 1503.18 3.03 | 623.18 |
| 23 15 | 3.01 | 10.39 61.40 | 1475.828 3.00 | 1486.35 3.02 | 618.96 |
| 23 16 | 2.78 | 6.08 101.1 | 1594.987 3.01 | 1602.06 3.02 | 620.27 |
| 23 17 | 2.77 | 5.10 118.7 | 1601.841 3.01 | 1609.76 3.02 | 616.95 |
| 23 18 | 3.00 | 6.49 91.96 | 1482.426 3.00 | 1489.07 3.02 | 623.53 |
| 23 19 | 2.89 | 6.12 98.65 | 1537.256 3.00 | 1548.95 3.02 | 624.11 |
| 23 20 | 2.93 | 8.15 76.36 | 1513.832 3.01 | 1522.97 3.02 | 623.20 |
| 23 21 | 2.98 | 3.65 155.1 | 1489.043 3.01 | 1493.23 3.03 | 625.26 |
| 23 22 | 2.56 | 4.43 140.0 | 1740.176 3.00 | 1750.88 3.01 | 619.41 |
| 23 23 | 2.59 | 7.24 89.98 | 1718.048 3.00 | 1727.16 3.02 | 620.71 |
| 23 24 | 2.44 | 8.51 80.62 | 1825.934 3.00 | 1832.70 3.01 | 619.59 |
| 23 25 | 2.50 | 6.62 98.94 | 1776.826 3.00 | 1785.21 3.02 | 623.15 |
| 23 26 | 2.94 | 5.34 110.6 | 1511.392 3.00 | 1520.41 3.02 | 624.30 |
| 23 27 | 2.95 | 4.57 126.9 | 1507.612 3.00 | 1512.78 3.02 | 620.78 |
| 23 28 | 3.06 | 4.33 130.7 | 1450.021 3.01 | 1456.68 3.03 | 623.23 |
| 23 29 | 3.01 | 9.73 64.88 | 1474.167 3.01 | 1483.36 3.02 | 620.59 |
| 23 30 | 2.88 | 4.82 122.3 | 1539.481 3.01 | 1544.71 3.03 | 620.36 |
| 23 31 | 2.89 | 3.70 155.5 | 1537.602 3.00 | 1541.00 3.03 | 621.32 |
| 23 32 | 2.42 | 10.40 68.39 | 1838.161 3.00 | 1850.57 3.01 | 610.39 |
| 23 33 | 2.35 | 8.76 80.17 | 1891.745 3.00 | 1899.77 3.02 | 621.18 |
| 23 34 | 2.91 | 5.25 112.7 | 1526.323 3.00 | 1534.67 3.02 | 622.52 |
| 23 35 | 2.99 | 0.00 0.00 | 1487.396 3.00 | 1490.14 3.03 | 622.43 |
| 23 36 | 2.97 | 12.15 54.36 | 1494.742 3.01 | 1509.13 3.02 | 618.00 |
| 23 37 | 4.09 | 3.30 145.6 | 1083.712 3.01 | 1089.48 3.04 | 639.97 |
| 23 38 | 3.09 | 9.27 66.64 | 1437.526 3.00 | 1449.37 3.02 | 621.34 |
| 23 39 | 3.11 | 4.26 131.7 | 1426.329 3.01 | 1431.50 3.03 | 621.03 |
| 23 40 | 4.73 | 6.46 73.57 | 937.018 3.01 | 944.19 3.04 | 620.14 |
| 23 41 | 4.65 | 3.91 116.6 | 954.706 3.01 | 960.95 3.04 | 632.37 |

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Protocol #:23ALPHA/BETA - 1.02
U-233 3% 2 sigma

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User : /

| | | | | | |
|--------------------------------|-------|------------|--------------|-------------|--------|
| 23 42 | 7.02 | 0.05 6462 | 630.044 3.02 | 632.23 3.07 | 631.34 |
| 23 43 | 7.03 | 6.00 64.51 | 629.427 3.02 | 637.55 3.06 | 624.07 |
| 23 44 | 10.46 | 4.11 74.51 | 421.378 3.03 | 427.48 3.09 | 623.48 |
| 23 45 | 10.46 | 1.62 178.8 | 421.569 3.02 | 425.09 3.10 | 623.12 |
| 23 46 | 11.73 | 0.88 305.5 | 375.284 3.03 | 377.06 3.12 | 627.06 |
| 23 47 | 11.73 | 2.67 105.0 | 375.454 3.03 | 379.44 3.11 | 619.97 |
| 23 48 | 12.44 | 1.69 157.8 | 353.902 3.03 | 355.62 3.13 | 628.20 |
| 23 49 | 12.58 | 2.80 97.13 | 349.765 3.03 | 353.48 3.12 | 626.89 |
| 23 50 | 13.37 | 0.17 1471 | 328.887 3.03 | 329.27 3.15 | 635.60 |
| 23 51 | 13.54 | 2.28 113.8 | 324.639 3.03 | 327.79 3.14 | 636.95 |
| 23 52 | 13.33 | 2.41 108.9 | 329.809 3.03 | 333.12 3.13 | 634.02 |
| 23 53 | 13.20 | 3.31 81.06 | 333.168 3.03 | 338.82 3.12 | 636.03 |
| 231 MISSING TUBE(S) Containers | | | | | |
| 55 | 9.43 | 4.11 78.41 | 468.218 3.02 | 474.23 3.08 | 627.94 |
| 23 56 | 9.50 | 0.04 8297 | 464.215 3.02 | 465.67 3.10 | 628.55 |
| 23 57 | 9.39 | 0.38 785.2 | 469.802 3.02 | 471.05 3.10 | 631.38 |
| 23 58 | 9.49 | 2.59 120.3 | 465.235 3.02 | 469.56 3.09 | 627.45 |
| 23 59 | 10.07 | 2.57 117.7 | 438.233 3.02 | 440.02 3.10 | 626.38 |
| 23 60 | 10.19 | 1.02 283.6 | 432.540 3.03 | 434.29 3.11 | 628.38 |
| 23 61 | 11.03 | 3.76 78.73 | 399.418 3.03 | 402.41 3.11 | 626.22 |
| 23 62 | 10.92 | 0.43 643.5 | 403.660 3.03 | 403.56 3.12 | 632.47 |
| 23 63 | 12.97 | 1.26 205.5 | 339.140 3.03 | 340.42 3.14 | 631.44 |
| 23 64 | 12.77 | 3.00 90.19 | 344.507 3.03 | 347.70 3.13 | 626.27 |
| 23 65 | 23.79 | 0.72 264.8 | 183.269 3.06 | 183.82 3.26 | 632.27 |
| 23 66 | 23.52 | 0.00 0.00 | 185.414 3.06 | 185.67 3.26 | 628.79 |
| 23 67 | 34.14 | 1.09 147.1 | 126.625 3.09 | 128.08 3.37 | 625.07 |
| 23 68 | 32.83 | 0.00 0.00 | 131.820 3.08 | 131.85 3.37 | 635.02 |
| 23 69 | 33.77 | 0.32 500.6 | 128.111 3.08 | 128.68 3.38 | 627.13 |
| 23 70 | 35.03 | 1.61 99.77 | 123.317 3.09 | 125.25 3.37 | 628.27 |
| 23 71 | 31.93 | 1.43 116.4 | 135.668 3.08 | 137.71 3.33 | 628.46 |
| 23 72 | 31.86 | 0.45 366.1 | 135.911 3.08 | 137.14 3.35 | 634.97 |
| 23 73 | 31.07 | 1.42 119.0 | 139.458 3.08 | 141.41 3.33 | 628.52 |
| 23 74 | 30.80 | 2.00 86.03 | 140.712 3.08 | 142.84 3.32 | 623.92 |
| 23 75 | 33.03 | 1.41 116.3 | 130.970 3.08 | 133.08 3.35 | 627.29 |
| 23 76 | 32.98 | 0.59 271.0 | 131.174 3.08 | 132.75 3.35 | 631.08 |
| 23 77 | 32.03 | 0.00 0.00 | 135.233 3.08 | 135.66 3.36 | 635.92 |
| 23 78 | 31.86 | 0.23 717.5 | 135.974 3.08 | 137.17 3.35 | 634.76 |
| 23 79 | 29.94 | 2.90 61.25 | 144.856 3.08 | 148.03 3.30 | 612.96 |
| 23 80 | 30.00 | 0.56 302.2 | 144.593 3.08 | 145.87 3.32 | 633.75 |
| 23 81 | 30.94 | 0.61 274.1 | 140.091 3.08 | 140.93 3.34 | 630.48 |
| 23 82 | 30.60 | 0.58 289.9 | 141.655 3.08 | 142.56 3.34 | 630.91 |
| 23 83 | 31.29 | 2.10 81.48 | 138.452 3.08 | 140.65 3.33 | 630.75 |
| 23 84 | 30.75 | 1.26 134.9 | 140.946 3.08 | 142.28 3.33 | 625.59 |
| 23 85 | 27.68 | 0.63 277.1 | 157.047 3.07 | 158.09 3.30 | 629.61 |
| 23 86 | 26.92 | 1.82 100.8 | 161.619 3.07 | 164.32 3.27 | 629.70 |
| 23 87 | 22.58 | 1.30 151.2 | 193.237 3.06 | 195.52 3.23 | 630.89 |
| 23 88 | 23.22 | 1.70 115.2 | 187.899 3.06 | 189.79 3.24 | 624.97 |

Uncapped QC1-00, desorption containers

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 89 | 19.54 | 1.68 | 127.2 | 224.010 | 3.05 | 224.91 | 3.21 | 626.62 |
| 23 | 90 | 19.87 | 0.00 | 0.00 | 220.080 | 3.05 | 220.43 | 3.22 | 635.31 |
| 23 | 91 | 14.38 | 1.80 | 138.3 | 305.605 | 3.03 | 308.24 | 3.15 | 636.44 |
| 23 | 92 | 14.59 | 0.94 | 259.0 | 301.155 | 3.04 | 301.73 | 3.16 | 634.52 |
| 23 | 93 | 11.32 | 1.20 | 229.7 | 389.094 | 3.03 | 390.46 | 3.12 | 631.22 |
| 23 | 94 | 10.97 | 1.70 | 166.7 | 401.713 | 3.03 | 404.23 | 3.11 | 635.08 |
| 23 | 95 | 9.50 | 2.56 | 121.3 | 464.321 | 3.02 | 466.41 | 3.10 | 627.52 |
| 23 | 96 | 9.78 | 3.96 | 79.74 | 451.232 | 3.02 | 456.09 | 3.09 | 630.05 |
| 23 | 97 | 9.33 | 1.15 | 263.2 | 472.739 | 3.02 | 474.69 | 3.10 | 636.26 |
| 23 | 98 | 9.65 | 1.59 | 190.2 | 457.151 | 3.02 | 458.80 | 3.10 | 632.52 |

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ALPHA/BETA - 1.02

U-233 3% 2 sigma

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| | | | | | | | | | |
|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 99 | 9.56 | 1.48 | 204.6 | 461.384 | 3.02 | 464.55 | 3.10 | 634.02 |
| 23 | 100 | 9.49 | 0.00 | 0.00 | 464.919 | 3.02 | 466.61 | 3.10 | 639.19 |
| 23 | 101 | 9.69 | 0.67 | 439.2 | 455.353 | 3.02 | 456.48 | 3.10 | 640.60 |
| 23 | 102 | 9.87 | 5.86 | 55.61 | 446.781 | 3.02 | 453.08 | 3.09 | 636.16 |
| 23 | 103 | 10.05 | 3.80 | 81.54 | 438.814 | 3.02 | 443.54 | 3.09 | 625.69 |
| 23 | 104 | 10.10 | 3.49 | 88.14 | 436.723 | 3.02 | 440.11 | 3.10 | 632.28 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 04-Dec-98 04:57

C14 Eff (0-156 keV) = 96.43 %

C14 CHI SQUARE IPA DATA PROCESSED - 04-Dec-98 05:07

C14 Chi Square = 20.94

H3 IPA DATA PROCESSED - 04-Dec-98 05:09

H3 Eff (0-18.6 keV) = 65.63 %

H3 CHI SQUARE IPA DATA PROCESSED - 04-Dec-98 05:19

H3 Chi Square = 34.64

BKG IPA DATA PROCESSED - 04-Dec-98 06:20

Bkg (0-18.6 keV) = 21.32 cpm

Bkg (0-156 keV) = 30.27 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 408.94

H3 E²/B (1-18.6 keV) = 201.63

[Data continued from page 75]

M/V (Qtz/U-233 son = 2 g/L), M/V (Clinop/U-233 = 0)
No Clinophtilolite was added, only Qtz is used as
substrate. The vials were kept capped during expts.

CAPPED (SORPTION)

12/15/98

NO Clinop (Qtz only) 12/15/98

| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|--------------|--|-------------|---|
| QC0U-pH 3.0 | 58.751 | 2.76 | 57.7308 |
| QC0U-pH 3.5 | 58.2928 | 3.33 | 57.2753 |
| QC0U-pH 4.0 | 58.387 | 3.81 | 57.3728 |
| QC0U-pH 4.25 | 58.7951 | 4.02 | 57.776 |
| QC0U-pH 4.5 | 58.6863 | 4.17 | 57.6716 |
| QC0U-pH4.75 | 58.8825 | 4.18 | 57.8639 |
| QC0U-pH 5.0 | 58.4776 | 6.42 | 57.4513 |
| QC0U-pH 5.25 | 58.3792 | 5.24 | 57.3691 |
| QC0U-pH 5.5 | 58.3364 | 5.63 | 57.3213 |
| QC0U-pH 5.75 | 58.4606 | 5.88 | 57.4401 |
| QC0U-pH 6.0 | 60.8091 | 4.17 | 59.7894 |
| QC0U-pH 6.25 | 58.9162 | 6.36 | 57.8957 |
| QC0U-pH 6.5 | 58.5745 | 6.49 | 57.5569 |
| QC0U-pH 6.75 | 58.8107 | 6.67 | 57.7926 |
| QC0U-pH 7.0 | 58.4751 | 6.76 | 57.4443 |
| QC0U-pH 7.25 | 59.4786 | 8.96 | 58.4402 |
| QC0U-pH 7.5 | 58.6428 | 6.46 | 57.6221 |
| QC0U-pH 7.75 | 58.4439 | 6.66 | 57.4205 |
| QC0U-pH 8.0 | 58.7237 | 7.11 | 57.7038 |
| QC0U-pH 8.25 | 58.761 | 6.89 | 57.7335 |
| QC0U-pH 8.5 | 58.8678 | 8.38 | 57.8484 |
| QC0U-pH 8.75 | 58.3256 | 7.64 | 57.3118 |
| QC0U-pH 9.0 | 58.3678 | 7.41 | 57.3518 |
| QC0U-pH 9.25 | 58.4654 | 7.6 | 57.4356 |
| QC0U-pH 9.5 | 58.6553 | 9.3 | 57.6418 |

No Clino (Quartz only) **CAPPED Sorption**

| Sample Name | wt. of vial | wt. Of vial + sample |
|----------------|-------------|----------------------|
| QC0U -pH 3a | 7.6277 | 8.1289 |
| QC0U -pH 3b | 7.6124 | 8.114 |
| QC0U--pH 3.5a | 7.6103 | 8.1094 |
| QC0U-pH 3.5b | 7.6141 | 8.1136 |
| QC0U--pH 4a | 7.6293 | 8.128 |
| QC0U--pH 4b | 7.5996 | 8.0969 |
| QC0U--pH 4.25a | 7.6643 | 8.1623 |
| QC0U--pH 4.25b | 7.6206 | 8.1212 |
| QC0U-pH 4.5a | 7.6567 | 8.1552 |
| QC0U-pH 4.5b | 7.5669 | 8.0655 |
| QC0U-pH 4.75a | 7.6373 | 8.1353 |
| QC0U-pH 4.75b | 7.5542 | 8.0542 |
| QC0U-pH 5a | 7.5661 | 8.0644 |
| QC0U-pH 5b | 7.6265 | 8.1254 |
| QC0U-pH 5.25a | 7.6174 | 8.1166 |
| QC0U-pH 5.25b | 7.6613 | 8.1612 |
| QC0U-pH 5.5a | 7.6273 | 8.1263 |
| QC0U-pH 5.5b | 7.626 | 8.1254 |
| QC0U-pH 5.75a | 7.6426 | 8.1399 |
| QC0U-pH 5.75b | 7.6356 | 8.1348 |
| QC0U-pH 6a | 7.613 | 8.1132 |
| QC0U-pH 6b | 7.6196 | 8.1242 |
| QC0U-pH 6.25a | 7.6704 | 8.1706 |
| QC0U-pH 6.25b | 7.5978 | 8.1022 |
| QC0U-pH 6.5a | 7.6094 | 8.1075 |
| QC0U-pH 6.5b | 7.6363 | 8.1339 |
| QC0U-pH 6.75a | 7.5789 | 8.0758 |
| QC0U-pH 6.75b | 7.6454 | 8.1433 |
| QC0U-pH 7a | 7.5346 | 8.036 |
| QC0U-pH 7b | 7.6585 | 8.1577 |
| QC0U-pH 7.25a | 7.6329 | 8.1337 |
| QC0U-pH 7.25b | 7.609 | 8.108 |
| QC0U-pH 7.5a | 7.5549 | 8.0539 |
| QC0U-pH 7.5b | 7.6114 | 8.1115 |
| QC0U-pH 7.75a | 7.5558 | 8.0558 |
| QC0U-pH 7.75b | 7.5858 | 8.0865 |
| QC0U-pH 8a | 7.5091 | 8.0092 |
| QC0U-pH 8b | 7.631 | 8.1305 |
| QC0U-pH 8.25a | 7.6081 | 8.1084 |
| QC0U-pH 8.25b | 7.6294 | 8.131 |
| QC0U-pH 8.5a | 7.6407 | 8.141 |
| QC0U-pH 8.5b | 7.5817 | 8.0808 |

| | | |
|---------------|--------|--------|
| QC0U-pH 8.75a | 7.6708 | 8.17 |
| QC0U-pH 8.75b | 7.6469 | 8.1481 |
| QC0U-pH 9a | 7.6431 | 8.1399 |
| QC0U-pH 9b | 7.5868 | 8.0852 |
| QC0U-pH 9.25a | 7.6081 | 8.1062 |
| QC0U-pH 9.25b | 7.6449 | 8.1425 |
| QC0U-pH 9.5a | 7.6848 | 8.183 |
| QC0U-pH 9.5b | 7.632 | 8.1298 |

CAPPED 12/16/98 *Desorption, No Clino (Quartz only)*

| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after transfer of qtz/clino, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer & acid add, g |
|--------------|--------------------|--|-----------------------------------|--|--|
| QC0U-pH 3.0 | 13.3781 | 19.929 | 22.9547 | 28.9298 | 54.1225 |
| QC0U-pH 3.5 | 13.7006 | 18.228 | 21.2486 | 26.9279 | 55.6617 |
| QC0U-pH 4.0 | 13.3093 | 19.6383 | 22.6548 | 28.6026 | 53.9925 |
| QC0U-pH 4.25 | 13.2509 | 19.7356 | 22.754 | 28.9859 | 54.1984 |
| QC0U-pH 4.5 | 13.4218 | 19.9782 | 22.9821 | 28.8807 | 54.012 |
| QC0U-pH 4.75 | 13.2197 | 20.1703 | 23.1964 | 28.3522 | 53.8527 |
| QC0U-pH 5.0 | 13.6255 | 20.1656 | 23.1766 | 28.9143 | 53.8014 |
| QC0U-pH 5.25 | 13.1447 | 20.6611 | 23.671 | 29.9456 | 52.7603 |
| QC0U-pH 5.5 | 13.3152 | 20.1629 | 23.1875 | 29.2199 | 53.3854 |
| QC0U-pH 5.75 | 13.7556 | 19.427 | 22.4403 | 28.0543 | 54.6243 |
| QC0U-pH 6.0 | 13.2029 | 20.7294 | 23.7356 | 29.917 | 55.1739 |
| QC0U-pH 6.25 | 13.2228 | 17.3315 | 20.3424 | 26.7841 | 56.479 |
| QC0U-pH 6.5 | 13.248 | 18.8222 | 21.8292 | 27.9732 | 54.8127 |
| QC0U-pH 6.75 | 13.6727 | 19.658 | 22.657 | 28.645 | 54.7056 |
| QC0U-pH 7.0 | 13.2973 | 19.1927 | 22.1966 | 28.2086 | 54.4132 |
| QC0U-pH 7.25 | 13.4084 | 19.7785 | 22.7829 | 28.9287 | 54.8841 |
| QC0U-pH 7.5 | 13.708 | 18.8564 | 21.8698 | 27.7738 | 55.3327 |
| QC0U-pH 7.75 | 13.6143 | 20.2859 | 23.2962 | 29.3197 | 53.6148 |
| QC0U-pH 8.0 | 13.2137 | 19.2373 | 22.2371 | 28.3196 | 54.4824 |
| QC0U-pH 8.25 | 13.8855 | 19.0705 | 22.0806 | 27.8605 | 55.3817 |
| QC0U-pH 8.5 | 13.6274 | 20.2118 | 23.2062 | 29.033 | 54.1125 |
| QC0U-pH 8.75 | 13.6811 | 19.6402 | 22.6486 | 28.2915 | 54.1677 |
| QC0U-pH 9.0 | 13.773 | 18.0081 | 21.6223 | 26.7945 | 55.9653 |
| QC0U-pH 9.25 | 13.7498 | 19.6771 | 22.672 | 28.552 | 54.18 |
| QC0U-pH 9.5 | 13.352 | 18.303 | 24.3285 | 27.4536 | 55.6034 |

CAPPED DESORPTION

12/28/98

*No Clinoptilactite
(Quartz only)*

| Sample Name | Container wt. before sampling | wt. Of PP tubes before sampling | Container wt. after sampling | wt. Of PP tubes after sampling |
|--------------|----------------------------------|------------------------------------|---------------------------------|-----------------------------------|
| QC0U-pH 3.0 | 54.0384 | 22.9487 | 53.0251 | 21.9376 |
| QC0U-pH 3.5 | 55.5330 | 21.1106 | 54.5260 | 20.1039 |
| QC0U-pH 4.0 | 53.9046 | 22.649 | 52.8944 | 21.6428 |
| QC0U-pH 4.25 | 54.0948 | 22.7458 | 53.0834 | 21.7419 |
| QC0U-pH 4.5 | 53.9312 | 22.9754 | 52.9205 | 21.9671 |
| QC0U-pH 4.75 | 53.7206 | 22.1894 | 52.7096 | 22.186 |
| QC0U-pH 5.0 | 53.6984 | 23.1557 | 52.6850 | 22.1512 |
| QC0U-pH 5.25 | 52.6334 | 23.6626 | 51.6527 | 22.6565 |
| QC0U-pH 5.5 | 53.2628 | 23.1642 | 52.2509 | 22.1635 |
| QC0U-pH 5.75 | 54.4061 | 22.3553 | 53.3938 | 21.3517 |
| QC0U-pH 6.0 | 55.0814 | 23.7064 | 54.0738 | 22.7052 |
| QC0U-pH 6.25 | 56.3376 | 20.3066 | 55.3291 | 19.305 |
| QC0U-pH 6.5 | 54.6909 | 21.7216 | 53.6846 | 20.7188 |
| QC0U-pH 6.75 | 54.5430 | 22.5339 | 53.5372 | 21.5314 |
| QC0U-pH 7.0 | 54.3314 | 22.1708 | 53.3239 | 21.158 |
| QC0U-pH 7.25 | 54.7348 | 22.7064 | 53.7248 | 21.7023 |
| QC0U-pH 7.5 | 55.1963 | 21.6659 | 54.1906 | 20.6688 |
| QC0U-pH 7.75 | 53.4834 | 23.2497 | 52.4740 | 22.2489 |
| QC0U-pH 8.0 | 54.4052 | 22.1968 | 53.3958 | 21.1946 |
| QC0U-pH 8.25 | 55.2194 | 22.0334 | 54.2088 | 21.0269 |
| QC0U-pH 8.5 | 53.9198 | 22.9139 | 52.9025 | 21.9049 |
| QC0U-pH 8.75 | 54.0234 | 22.55 | 53.0146 | 21.5409 |
| QC0U-pH 9.0 | 55.8171 | 20.967 | 54.8041 | 19.9622 |
| QC0U-pH 9.25 | 53.9557 | 22.6504 | 52.9465 | 21.6442 |
| QC0U-pH 9.5 | 55.3931 | 24.247 | 54.3624 | 23.2438 |

No Clinoptilactite (Quartz only) DESORPTION (CAPPED) Tubes & Containers
12/28/98

| PP tubes | | Desorption | | PC containers Desorption | |
|---------------|---------------|----------------------|--|--------------------------|----------------------|
| Sample Name | wt. of vial,g | wt. of vial+sample,g | | wt. of vial,g | wt. of vial+sample,g |
| QC0U-pH 3a | 7.8796 | 8.3799 | | 7.5608 | 8.0641 |
| QC0U-pH 3b | 7.8703 | 8.3719 | | 7.5461 | 8.0529 |
| QC0U-pH 3.5a | 7.8011 | 8.2999 | | 7.5379 | 8.0383 |
| QC0U-pH 3.5b | 7.9514 | 8.4509 | | 7.6211 | 8.1238 |
| QC0U-pH 4a | 7.8647 | 8.3641 | | 7.6590 | 8.1615 |
| QC0U-pH 4b | 7.8686 | 8.3674 | | 7.5590 | 8.0648 |
| QC0U-pH 4.25a | 7.8776 | 8.3753 | | 7.5976 | 8.1003 |
| QC0U-pH 4.25b | 7.9322 | 8.4329 | | 7.5803 | 8.0865 |
| QC0U-pH 4.5a | 7.9120 | 8.4099 | | 7.5867 | 8.0891 |
| QC0U-pH 4.5b | 7.8640 | 8.3640 | | 7.6206 | 8.1273 |
| QC0U-pH 4.75a | 7.8751 | 8.3722 | | 7.6248 | 8.1276 |
| QC0U-pH 4.75b | 8.0188 | 8.5184 | | 7.6409 | 8.1459 |
| QC0U-pH 5a | 7.9450 | 8.4418 | | 7.5390 | 8.0414 |
| QC0U-pH 5b | 8.0148 | 8.5142 | | 7.6086 | 8.1142 |
| QC0U-pH 5.25a | 7.8462 | 8.3436 | | 7.6007 | 8.1037 |
| QC0U-pH 5.25b | 7.9621 | 8.4611 | | 7.6342 | 8.1393 |
| QC0U-pH 5.5a | 7.8215 | 8.3162 | | 7.6163 | 8.1193 |
| QC0U-pH 5.5b | 7.8515 | 8.3484 | | 7.6021 | 8.1082 |
| QC0U-pH 5.75a | 7.8564 | 8.3536 | | 7.6514 | 8.1535 |
| QC0U-pH 5.75b | 7.8576 | 8.3552 | | 7.5688 | 8.0743 |
| QC0U-pH 6a | 7.8782 | 8.3731 | | 7.6376 | 8.1403 |
| QC0U-pH 6b | 7.9346 | 8.4306 | | 7.6388 | 8.1422 |
| QC0U-pH 6.25a | 7.8529 | 8.3473 | | 7.6447 | 8.1468 |
| QC0U-pH 6.25b | 7.8775 | 8.3744 | | 7.5836 | 8.0883 |
| QC0U-pH 6.5a | 7.9092 | 8.4044 | | 7.5871 | 8.0873 |
| QC0U-pH 6.5b | 7.9127 | 8.4107 | | 7.6106 | 8.1146 |
| QC0U-pH 6.75a | 7.8209 | 8.3165 | | 7.5998 | 8.1007 |
| QC0U-pH 6.75b | 7.8918 | 8.3877 | | 7.5100 | 8.0131 |
| QC0U-pH 7a | 7.7809 | 8.2782 | | 7.5898 | 8.0928 |
| QC0U-pH 7b | 7.8261 | 8.3243 | | 7.6497 | 8.1533 |
| QC0U-pH 7.25a | 7.8293 | 8.3277 | | 7.6066 | 8.1095 |
| QC0U-pH 7.25b | 7.9167 | 8.4135 | | 7.5833 | 8.0884 |
| QC0U-pH 7.5a | 7.8635 | 8.3586 | | 7.5728 | 8.0737 |
| QC0U-pH 7.5b | 7.8083 | 8.3051 | | 7.5628 | 8.066 |
| QC0U-pH 7.75a | 7.9811 | 8.4761 | | 7.6565 | 8.1587 |
| QC0U-pH 7.75b | 7.8928 | 8.3882 | | 7.6288 | 8.1339 |
| QC0U-pH 8a | 7.8571 | 8.3532 | | 7.5831 | 8.086 |
| QC0U-pH 8b | 7.8961 | 8.3941 | | 7.7187 | 8.2244 |
| QC0U-pH 8.25a | 7.8661 | 8.3642 | | 7.7471 | 8.2508 |
| QC0U-pH 8.25b | 7.9240 | 8.4236 | | 7.7682 | 8.2729 |
| QC0U-pH 8.5a | 7.9126 | 8.4098 | | 7.8132 | 8.3152 |

CAPPED DESORPTION (Quartz only), No Ulinap

| | | | | |
|---------------|--------|--------|--------|--------|
| QC0U-pH 8.5b | 7.8829 | 8.3837 | 7.8377 | 8.3449 |
| QC0U-pH 8.75a | 7.8157 | 8.3148 | 7.8688 | 8.3697 |
| QC0U-pH 8.75b | 7.8674 | 8.3685 | 7.8539 | 8.3561 |
| QC0U-pH 9a | 7.915 | 8.4114 | 7.8306 | 8.3305 |
| QC0U-pH 9b | 7.9266 | 8.4267 | 7.9264 | 8.4297 |
| QC0U-pH 9.25a | 7.8263 | 8.3249 | 7.8912 | 8.3922 |
| QC0U-pH 9.25b | 7.8200 | 8.3192 | 7.9250 | 8.4263 |
| QC0U-pH 9.5a | 7.9508 | 8.4466 | 7.9677 | 8.4768 |
| QC0U-pH 9.5b | 7.8765 | 8.3755 | 7.8930 | 8.4039 |

SYSTEM NORMALIZED

CAPPED SORPTION LSA, Qtz only

QCOU

C14 IPA DATA PROCESSED - 17-Dec-98 12:14

C14 Eff (0-156 keV) = 96.28 %

C14 CHI SQUARE IPA DATA PROCESSED - 17-Dec-98 12:24

C14 Chi Square = 20.85

H3 IPA DATA PROCESSED - 17-Dec-98 12:26

H3 Eff (0-18.6 keV) = 64.94 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves & view historic data

WARNING: Questionable H3 Chi square value - Please view historic data

BKG IPA DATA PROCESSED - 17-Dec-98 13:37

Bkg (0-18.6 keV) = 22.42 cpm

Bkg (0-156 keV) = 32.50 cpm

WARNING: Questionable C14 Background value - Please view historic data

WARNING: Questionable C14 Figure of Merit value - Please view historic data

17 Dec 98 13:37

Protocol #:23

ALPHA/BETA - 1.02

U-233 3% 2 sigma

Page #4

User : Alka

C14 E²/B (1-156 keV) = 372.47

H3 E²/B (1-18.6 keV) = 188.22

18 Dec 98 06:26

Protocol #:23

ALPHA/BETA - 1.02

U-233 3% 2 sigma

Page

User :

Time: 999.99

Data Mode: CPM

Background Subtract: 1st Vial

Nuclide: MANUAL

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 19.96 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.54 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.42 | |

Capped Sorption

QCOU

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|------|------------|------------|--------------|--------------------|
| P# | 1 | 999.99 | 19.96 1.42 | 3.544 3.36 | 29.42 1.17 145.20 |
| 23 | 2 | 8.83 | 5.18 65.36 | 499.967 3.02 | 506.02 3.08 630.04 |
| 23 | 3 | 8.96 | 1.25 248.1 | 492.885 3.02 | 494.57 3.09 632.52 |
| 23 | 4 | 8.85 | 2.30 138.5 | 498.716 3.02 | 501.31 3.09 628.26 |
| 23 | 5 | 8.73 | 3.52 93.48 | 506.078 3.02 | 511.01 3.08 629.71 |
| 23 | 6 | 9.00 | 3.26 98.87 | 490.567 3.02 | 495.91 3.08 629.19 |
| 23 | 7 | 9.19 | 2.78 113.6 | 480.134 3.02 | 482.66 3.09 629.69 |
| 23 | 8 | 9.43 | 1.25 241.3 | 468.354 3.02 | 471.43 3.09 632.28 |
| 23 | 9 | 9.45 | 2.90 107.8 | 467.144 3.02 | 471.53 3.09 627.54 |
| 23 | 10 | 9.20 | 4.17 77.97 | 479.499 3.02 | 484.05 3.09 626.47 |
| 23 | 11 | 9.02 | 1.33 232.7 | 489.250 3.02 | 490.53 3.10 627.94 |
| 23 | 12 | 9.38 | 3.49 90.88 | 470.443 3.02 | 474.63 3.09 625.60 |
| 23 | 13 | 9.09 | 6.22 54.74 | 485.565 3.02 | 491.81 3.08 621.35 |
| 23 | 14 | 50.38 | 1.85 72.60 | 84.666 3.13 | 86.69 3.52 622.85 |
| 23 | 15 | 52.27 | 2.37 56.52 | 81.514 3.13 | 84.33 3.52 617.17 |
| 23 | 16 | 16.84 | 3.02 77.91 | 260.351 3.04 | 264.10 3.16 624.49 |
| 23 | 17 | 16.17 | 1.50 154.8 | 271.348 3.04 | 272.74 3.17 630.84 |
| 23 | 18 | 32.65 | 3.32 51.62 | 132.566 3.08 | 136.33 3.31 619.85 |
| 23 | 19 | 32.77 | 1.86 89.11 | 132.159 3.08 | 135.33 3.32 627.61 |
| 23 | 20 | 47.01 | 1.27 108.2 | 90.989 3.12 | 92.93 3.49 624.98 |
| 23 | 21 | 46.47 | 1.52 91.60 | 92.088 3.12 | 93.71 3.49 623.09 |
| 23 | 22 | 10.44 | 0.44 635.6 | 422.222 3.03 | 424.03 3.11 631.96 |
| 23 | 23 | 9.82 | 2.24 134.9 | 449.104 3.02 | 451.94 3.10 629.00 |
| 23 | 24 | 50.99 | 0.59 219.4 | 83.630 3.13 | 84.56 3.56 631.18 |
| 23 | 25 | 51.98 | 0.80 162.3 | 81.950 3.13 | 83.72 3.55 627.46 |
| 23 | 26 | 54.78 | 1.62 79.57 | 77.617 3.14 | 79.78 3.57 621.79 |
| 23 | 27 | 54.72 | 0.65 192.7 | 77.669 3.14 | 78.69 3.60 628.03 |
| 23 | 28 | 52.86 | 1.64 79.65 | 80.527 3.14 | 82.21 3.56 620.65 |
| 23 | 29 | 51.79 | 0.62 207.4 | 82.264 3.13 | 83.80 3.55 632.54 |
| 23 | 30 | 44.26 | 1.30 108.8 | 96.863 3.11 | 98.48 3.47 624.16 |
| 23 | 31 | 45.05 | 0.37 368.3 | 95.102 3.11 | 95.93 3.50 632.36 |
| 23 | 32 | 31.49 | 3.13 55.51 | 137.802 3.08 | 141.68 3.30 620.53 |
| 23 | 33 | 31.59 | 3.05 56.67 | 137.165 3.08 | 140.44 3.31 620.38 |
| 23 | 34 | 42.09 | 2.37 62.54 | 102.158 3.10 | 104.84 3.42 619.71 |

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 35 | 40.39 | 1.88 | 79.79 | 106.483 | 3.10 | 108.88 | 3.41 | 624.26 |
| 23 | 36 | 40.71 | 1.53 | 96.55 | 105.643 | 3.10 | 107.57 | 3.43 | 622.11 |
| 23 | 37 | 41.53 | 2.00 | 74.08 | 103.487 | 3.10 | 105.80 | 3.43 | 621.87 |
| 23 | 38 | 36.56 | 0.53 | 289.3 | 118.010 | 3.09 | 118.99 | 3.40 | 628.80 |
| 23 | 39 | 37.10 | 1.79 | 86.91 | 116.267 | 3.09 | 117.94 | 3.39 | 627.14 |
| 23 | 40 | 36.67 | 0.93 | 165.3 | 117.645 | 3.09 | 119.06 | 3.39 | 625.54 |
| 23 | 41 | 36.13 | 1.55 | 101.5 | 119.456 | 3.09 | 121.48 | 3.38 | 630.53 |

Capped Sorption QCOU

| | | | | | | | | | | |
|---------------------|------|-------|-------------------|---------|---------|--------|--------|--------|--------|--------|
| 19 Dec 98 02:54 | | | ALPHA/BETA - 1.02 | | | | | | Pa | |
| Protocol #:23 | | | U-233 3% 2 sigma | | | | | | User : | |
| 23 | 42 | 31.43 | 0.31 | 531.1 | 137.881 | 3.08 | 138.51 | 3.35 | 634.36 | |
| 23 | 43 | 30.70 | 0.72 | 230.1 | 141.244 | 3.08 | 144.19 | 3.31 | 628.92 | |
| 23 | 44 | 38.13 | 0.34 | 438.7 | 113.005 | 3.10 | 113.51 | 3.42 | 636.87 | |
| 23 | 45 | 36.86 | 1.80 | 86.90 | 117.020 | 3.09 | 118.98 | 3.39 | 623.71 | |
| 23 | 46 | 30.54 | 1.59 | 107.5 | 141.970 | 3.08 | 144.32 | 3.31 | 630.74 | |
| 23 | 47 | 30.33 | 2.49 | 69.95 | 142.978 | 3.08 | 145.52 | 3.31 | 624.91 | |
| 23 | 48 | 22.56 | 0.00 | 0.00 | 193.442 | 3.06 | 193.67 | 3.25 | 633.06 | |
| 23 | 49 | 22.83 | 1.68 | 117.3 | 191.112 | 3.06 | 193.31 | 3.24 | 627.50 | |
| 23 | 50 | 34.44 | 2.77 | 59.44 | 125.492 | 3.09 | 129.00 | 3.34 | 622.55 | |
| 23 | 51 | 34.84 | 0.22 | 710.9 | 124.010 | 3.09 | 125.48 | 3.37 | 636.32 | |
| 231 MISSING TUBE(S) | | | | | | | | | | |
| 53 | 8.40 | 3.25 | 102.5 | 525.504 | 3.02 | 529.39 | 3.08 | 629.31 | Std. | |
| 23 | 54 | 8.40 | 0.99 | 319.6 | 526.099 | 3.02 | 527.24 | 3.09 | | 634.08 |
| 23 | | | | | | | | | | |

Standards Recounted on 2/10/99 AJ

Standards Recounted on 2/10/99 AJ

30 Dec 98 04:03
Protocol #:23

ALPHA/BETA - 1.02
U-233 3% 2 sigma

115
Page

User : A

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.39 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.68 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 30.01 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Purge(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|------|------------|------------|------------|---------------------------------|
| P# | 1 | 999.99 | 20.39 | 1.40 | 3.675 3.30 30.01 1.15 147.05 B |
| 23 | 2 | 9.62 | 2.47 | 125.1 | 458.695 3.02 461.57 3.10 623.00 |
| 23 | 3 | 9.67 | 3.39 | 92.89 | 456.304 3.02 459.96 3.10 621.59 |
| 23 | 4 | 9.54 | 0.00 | 0.00 | 462.467 3.02 463.39 3.10 629.96 |
| 23 | 5 | 9.63 | 5.25 | 62.35 | 457.799 3.02 462.93 3.09 623.99 |
| 23 | 6 | 9.79 | 2.59 | 118.9 | 450.462 3.02 453.14 3.10 625.48 |
| 23 | 7 | 9.92 | 1.58 | 189.1 | 444.410 3.02 446.10 3.11 627.16 |
| 23 | 8 | 9.70 | 2.80 | 110.9 | 454.779 3.02 458.86 3.10 624.14 |
| 23 | 9 | 9.65 | 1.06 | 283.5 | 456.843 3.02 458.70 3.10 627.50 |
| 23 | 10 | 10.16 | 2.15 | 139.5 | 434.120 3.02 436.82 3.10 628.65 |
| 23 | 11 | 9.85 | 3.46 | 90.24 | 447.594 3.02 450.50 3.10 625.14 |
| 23 | 12 | 9.85 | 0.82 | 357.9 | 448.305 3.02 449.48 3.11 629.71 |
| 23 | 13 | 9.71 | 4.63 | 69.60 | 454.203 3.02 458.56 3.09 622.83 |
| 23 | 14 | 25.54 | 0.79 | 233.9 | 170.327 3.07 170.85 3.29 625.42 |
| 23 | 15 | 26.37 | 0.00 | 0.00 | 165.001 3.07 164.87 3.30 630.26 |
| 23 | 16 | 16.01 | 0.00 | 0.00 | 273.964 3.04 274.24 3.18 630.09 |
| 23 | 17 | 15.94 | 0.94 | 249.1 | 275.309 3.04 276.89 3.17 626.11 |
| 23 | 18 | 21.27 | 2.74 | 76.91 | 205.305 3.05 208.21 3.22 621.50 |
| 23 | 19 | 22.00 | 1.15 | 173.7 | 198.416 3.06 199.81 3.24 625.65 |
| 23 | 20 | 24.09 | 0.00 | 0.00 | 180.841 3.06 180.45 3.28 633.92 |
| 23 | 21 | 23.88 | 1.67 | 116.1 | 182.590 3.06 185.28 3.25 626.38 |
| 23 | 22 | 10.63 | 2.65 | 111.5 | 414.575 3.03 417.59 3.11 627.74 |
| 23 | 23 | 10.82 | 3.45 | 86.44 | 407.046 3.03 411.21 3.11 623.88 |
| 23 | 24 | 25.59 | 1.06 | 174.9 | 169.987 3.07 171.24 3.28 628.39 |
| 23 | 25 | 25.77 | 1.88 | 100.1 | 168.812 3.07 171.54 3.27 627.02 |
| 23 | 26 | 24.72 | 2.79 | 70.28 | 176.179 3.06 179.13 3.25 625.39 |
| 23 | 27 | 24.63 | 2.26 | 85.77 | 176.796 3.06 180.26 3.25 623.50 |
| 23 | 28 | 23.70 | 1.72 | 113.8 | 183.836 3.06 185.86 3.25 625.52 |
| 23 | 29 | 23.22 | 1.35 | 144.5 | 187.712 3.06 190.15 3.24 623.38 |
| 23 | 30 | 28.55 | 1.78 | 100.5 | 152.017 3.07 154.23 3.30 619.88 |
| 23 | 31 | 27.57 | 0.86 | 206.7 | 157.623 3.07 159.44 3.30 627.82 |
| 23 | 32 | 28.69 | 1.63 | 108.6 | 151.222 3.07 152.49 3.32 624.29 |
| 23 | 33 | 29.30 | 1.18 | 148.0 | 147.997 3.08 149.17 3.32 623.29 |
| 23 | 34 | 25.45 | 0.00 | 0.00 | 170.981 3.07 171.37 3.29 627.68 |
| 23 | 35 | 24.96 | 1.12 | 167.7 | 174.410 3.06 175.80 3.27 624.26 |
| 23 | 36 | 22.33 | 2.04 | 99.17 | 195.385 3.06 198.12 3.23 625.68 |
| 23 | 37 | 22.48 | 0.82 | 238.2 | 194.012 3.06 195.44 3.25 627.07 |
| 23 | 38 | 29.04 | 1.78 | 99.37 | 149.355 3.08 152.26 3.30 622.53 |
| 23 | 39 | 28.52 | 0.00 | 0.00 | 152.251 3.07 152.60 3.32 624.21 |
| 23 | 40 | 25.58 | 1.38 | 135.2 | 170.094 3.07 171.09 3.28 620.17 |
| 23 | 41 | 25.39 | 1.94 | 97.93 | 171.355 3.07 173.02 3.27 617.64 |

Capped
QCOU Description Containers
Qtz only, No Clinop.

QCOU
cap

Desor
Conta

Qtz
No cl

30 Dec 98 17:22

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User : A

| | | | | | | | | | |
|-----|--|-------|-------|---------|----------|--------|---------|--------|--------|
| 23 | 42 | 28.49 | 1.40 | 126.3 | 152.345 | 3.07 | 154.62 | 3.30 | 618.87 |
| 23 | 43 | 28.58 | 2.94 | 62.16 | 151.923 | 3.07 | 155.30 | 3.29 | 608.33 |
| 23 | 44 | 28.28 | 3.72 | 50.21 | 153.468 | 3.07 | 157.58 | 3.28 | 611.69 |
| 23 | 45 | 29.05 | 2.02 | 88.30 | 149.303 | 3.08 | 152.19 | 3.30 | 615.12 |
| 23 | 46 | 22.44 | 4.92 | 43.58 | 194.409 | 3.06 | 199.58 | 3.21 | 609.50 |
| 23 | 47 | 22.40 | 4.65 | 45.89 | 194.852 | 3.06 | 199.10 | 3.22 | 611.78 |
| 23 | 48 | 20.06 | 4.73 | 47.70 | 217.910 | 3.05 | 222.58 | 3.19 | 609.57 |
| 23 | 49 | 20.87 | 3.47 | 62.22 | 209.310 | 3.05 | 213.74 | 3.20 | 616.85 |
| 23 | 50 | 29.04 | 4.57 | 41.04 | 149.390 | 3.07 | 153.46 | 3.28 | 611.72 |
| 23 | 51 | 28.48 | 3.31 | 55.85 | 152.364 | 3.07 | 155.07 | 3.30 | 610.57 |
| 233 | MISSING TUBE(S) Resolution tubes capped, No Clinop. Atz only | | | | | | | | |
| 55 | 13.20 | 6.20 | 46.04 | 333.598 | 3.03 | 340.29 | 3.11 | 603.46 | |
| 23 | 56 | 12.84 | 10.91 | 28.73 | 342.898 | 3.03 | 354.03 | 3.09 | 597.94 |
| 23 | 57 | 14.43 | 9.34 | 30.90 | 304.364 | 3.04 | 314.07 | 3.11 | 593.67 |
| 23 | 58 | 14.96 | 3.47 | 73.27 | 293.451 | 3.04 | 297.20 | 3.15 | 609.14 |
| 23 | 59 | 11.99 | 3.46 | 81.97 | 366.967 | 3.03 | 369.99 | 3.12 | 608.22 |
| 23 | 60 | 11.86 | 7.01 | 43.57 | 371.114 | 3.03 | 378.93 | 3.10 | 601.94 |
| 23 | 61 | 11.65 | 5.36 | 55.76 | 377.956 | 3.03 | 384.41 | 3.10 | 606.47 |
| 23 | 62 | 11.27 | 4.01 | 73.79 | 391.179 | 3.03 | 395.55 | 3.11 | 607.58 |
| 23 | 63 | 11.12 | 4.79 | 63.17 | 396.055 | 3.03 | 402.19 | 3.10 | 611.89 |
| 23 | 64 | 11.07 | 5.71 | 54.00 | 398.222 | 3.03 | 403.42 | 3.10 | 609.04 |
| 23 | 65 | 10.76 | 4.70 | 65.28 | 409.708 | 3.03 | 414.42 | 3.10 | 606.84 |
| 23 | 66 | 10.64 | 6.20 | 51.18 | 414.370 | 3.03 | 421.12 | 3.09 | 611.65 |
| 23 | 67 | 3.76 | 7.00 | 77.23 | 1182.229 | 3.00 | 1188.61 | 3.03 | 611.62 |
| 23 | 68 | 3.75 | 16.67 | 37.75 | 1184.592 | 3.01 | 1201.99 | 3.02 | 605.83 |
| 23 | 69 | 4.61 | 12.14 | 43.82 | 962.268 | 3.01 | 974.98 | 3.03 | 607.88 |
| 23 | 70 | 4.62 | 4.93 | 95.15 | 959.745 | 3.01 | 964.15 | 3.04 | 613.82 |
| 23 | 71 | 4.48 | 15.99 | 35.69 | 991.414 | 3.01 | 1008.61 | 3.02 | 602.51 |
| 23 | 72 | 4.50 | 9.61 | 53.84 | 985.880 | 3.01 | 996.88 | 3.03 | 608.55 |
| 23 | 73 | 4.31 | 10.93 | 49.41 | 1027.880 | 3.01 | 1039.60 | 3.03 | 606.04 |
| 23 | 74 | 4.28 | 13.25 | 42.37 | 1035.344 | 3.01 | 1048.73 | 3.03 | 604.80 |
| 23 | 75 | 11.26 | 5.54 | 55.05 | 391.085 | 3.03 | 396.63 | 3.11 | 608.74 |
| 23 | 76 | 10.97 | 4.13 | 72.78 | 401.430 | 3.03 | 406.55 | 3.10 | 615.16 |
| 23 | 77 | 2.28 | 20.40 | 41.50 | 1949.395 | 3.00 | 1968.68 | 3.01 | 604.93 |
| 23 | 78 | 2.24 | 21.12 | 40.78 | 1989.629 | 3.00 | 2007.49 | 3.00 | 603.37 |
| 23 | 79 | 2.85 | 15.40 | 46.07 | 1558.079 | 3.00 | 1575.25 | 3.01 | 605.67 |
| 23 | 80 | 2.86 | 15.97 | 44.69 | 1550.870 | 3.01 | 1565.80 | 3.02 | 605.48 |
| 23 | 81 | 2.93 | 24.66 | 31.83 | 1514.755 | 3.01 | 1540.30 | 3.01 | 599.05 |
| 23 | 82 | 2.94 | 18.72 | 39.00 | 1511.631 | 3.00 | 1532.24 | 3.01 | 604.96 |
| 23 | 83 | 2.72 | 15.64 | 46.59 | 1636.766 | 3.00 | 1654.92 | 3.01 | 605.76 |
| 23 | 84 | 2.75 | 9.79 | 67.76 | 1612.689 | 3.01 | 1622.72 | 3.02 | 607.25 |
| 23 | 85 | 2.86 | 15.97 | 44.69 | 1551.220 | 3.01 | 1568.94 | 3.01 | 605.41 |
| 23 | 86 | 2.89 | 23.55 | 33.14 | 1535.079 | 3.01 | 1558.23 | 3.01 | 604.00 |
| 23 | 87 | 2.66 | 13.06 | 54.34 | 1672.641 | 3.00 | 1685.78 | 3.01 | 606.42 |
| 23 | 88 | 2.76 | 12.94 | 53.76 | 1609.731 | 3.00 | 1625.06 | 3.01 | 604.66 |
| 23 | 89 | 3.24 | 11.40 | 55.03 | 1369.164 | 3.01 | 1380.79 | 3.02 | 612.25 |
| 23 | 90 | 3.46 | 15.15 | 42.34 | 1282.741 | 3.01 | 1299.18 | 3.02 | 611.23 |

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|----|-----|------|-------|-------|----------|------|---------|------|--------|
| 23 | 91 | 2.85 | 18.90 | 39.32 | 1561.939 | 3.00 | 1582.27 | 3.01 | 605.95 |
| 23 | 92 | 2.83 | 24.84 | 32.21 | 1572.297 | 3.00 | 1597.55 | 3.00 | 604.42 |
| 23 | 93 | 2.46 | 9.69 | 72.26 | 1805.675 | 3.00 | 1816.33 | 3.02 | 609.72 |
| 23 | 94 | 2.50 | 19.21 | 41.47 | 1775.925 | 3.00 | 1798.39 | 3.01 | 607.49 |
| 23 | 95 | 2.96 | 13.73 | 49.51 | 1499.703 | 3.01 | 1513.91 | 3.02 | 610.13 |
| 23 | 96 | 2.98 | 23.23 | 32.96 | 1491.291 | 3.00 | 1513.95 | 3.01 | 606.04 |
| 23 | 97 | 2.67 | 23.80 | 34.21 | 1662.992 | 3.00 | 1686.47 | 3.01 | 604.06 |
| 23 | 98 | 2.70 | 13.31 | 53.13 | 1645.955 | 3.00 | 1658.14 | 3.02 | 610.01 |
| 23 | 99 | 2.50 | 29.61 | 30.23 | 1776.725 | 3.00 | 1808.79 | 3.00 | 604.00 |
| 23 | 100 | 2.39 | 23.96 | 35.98 | 1855.739 | 3.01 | 1880.87 | 3.01 | 604.23 |

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desorption
tubes

31 Dec 98 02:32

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

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| | | | | | | | | | |
|----|-----|------|-------|-------|----------|------|---------|------|--------|
| 23 | 101 | 3.34 | 15.83 | 41.64 | 1328.062 | 3.01 | 1344.24 | 3.02 | 609.27 |
| 23 | 102 | 3.30 | 12.03 | 52.17 | 1346.931 | 3.00 | 1359.69 | 3.02 | 609.49 |
| 23 | 103 | 3.50 | 15.89 | 40.56 | 1266.611 | 3.01 | 1282.28 | 3.02 | 602.69 |
| 23 | 104 | 3.56 | 16.12 | 39.77 | 1248.010 | 3.00 | 1264.94 | 3.02 | 603.43 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 31-Dec-98 02:52

C14 Eff (0-156 keV) = 96.83 %

C14 CHI SQUARE IPA DATA PROCESSED - 31-Dec-98 03:03

C14 Chi Square = 32.02

H3 IPA DATA PROCESSED - 31-Dec-98 03:04

H3 Eff (0-18.6 keV) = 65.16 %

H3 CHI SQUARE IPA DATA PROCESSED - 31-Dec-98 03:15

H3 Chi Square = 21.04

BKG IPA DATA PROCESSED - 31-Dec-98 04:15

Bkg (0-18.6 keV) = 22.02 cpm

Bkg (0-156 keV) = 32.15 cpm

WARNING: Questionable C14 Background value - Please view historic data

12/22/98
AJ ① Preparations of 0.1 M NaNO_3 matrix to dilute the U-233 stock solutions.

Refer to page 34 for procedure

16.9968 g of NaNO_3 (lot # 930601) in 2000 g of nanopure water.

② Preparation of 50 PPb U stock soln.
Refer to page ③④ for procedure.

③ For CAPPED VIAL EXPERIMENTS, prepare all the reagents in degassed water.

Note 1- Degassed water was prepared by boiling 1000 ml of water in flasks and cooling it with mouth covered with rubber stopper. 3 batches of 1000 ml were prepared.

Prepare 1 M, 0.1 M, 0.001 M, 0.01 M HNO_3 stock solutions

④ 1 M HNO_3 :-
AJ 15.75 ml of conc. HNO_3 diluted it to 250 ml of degassed H_2O .

⑤ 0.1 M HNO_3

20 ml of 1 M HNO_3 & dilute it to 200 ml with degassed H_2O

⑥ 0.01 M HNO_3

2 ml of 1 M HNO_3 & dilute it to 200 ml with degassed H_2O

⑦ 0.001 M HNO_3

Dilute 20 ml of 0.01 M HNO_3 to 200 ml with degassed H_2O .

⑧ Degassed NaNO_3 soln.

Dilute 16.9968 g of NaNO_3 (lot # 930601) with 2000 g of nanopure water.

⑨ Boil 2 lts of nanopure H_2O for NaOH & HNO_3 stocks solution for capped vials.

12/30/98

AJ ① Degassed 50 PPb U-233 stock solution

Dilute 200 g of 500 PPb soln (28A Spike) to 2000 g with nanopure water. PH of starting U-233 solution = 4.28

② 1 M NaOH (degassed)

Dilute 40 g of NaOH pellets (lot #) to 2000 g with degassed H_2O .

③ 0.1 M NaOH

Dilute 10 ml of 1 M NaOH soln to 100 ml with degassed H_2O .

④ 0.05 NaOH

Dilute 10 ml of 1 M NaOH to 200 ml with degassed H_2O .

⑤ 0.02 M HNO_3

Dilute 2 ml of 1 M HNO_3 to 100 ml with degassed H_2O .

Qtz = 1g, Clinop = 0.35g, QC10U, UNCAPPED EXPTS.

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C.

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma U = 50 \text{ ppb}$ (2.14×10^{-7})
 - equilibrium with atmospheric $\text{CO}_2(\text{g})$; $p\text{CO}_2 = 10^{-3.5}$
 - pH range 4.0-9.5
 - solution volume = 35 ml
 - mass of solid 1, quartz = 1.000 g
 - mass of solid 2, clinoptilolite = 350mg (0.35 g)
 - $M/V = 28.57 \text{ g/L}$

Equipment: Orion 920A pH meter and combination pH electrode
 Gyrotory shaker
 Fisher Marathon 21K centrifuge
 Mettler PM4600 and AE240 balances
 Packard 2500 TR/AB LSC
 Eppendorf micropipetters and tips
 Oxford pipettes and tips
 Repipettor for transfer of scintillation cocktail

Supplies: *W510 * UC * RC * RFe * HL * $\leq 325 \text{ mesh AJ}$*
*CDV * UC * RC * RFe * HL * NaF * 100/200 mesh AJ*
 50 ppb ^{233}U stock solution
 polycarbonate centrifuge tubes with caps (50 ml capacity)
 polypropylene centrifuge tubes with caps (50 ml capacity)
 pH buffer solutions
 Ultima-Gold liquid scintillation cocktail
 7 ml scintillation vials
 reagent grade ~~NaOH~~ *NaHCO₃ (lot 936883) AJ*
 concentrated HNO_3 (lot 983546)
 HNO_3 and NaHCO_3 or NaOH stock solutions at various concentrations
 weighing paper/boats
 deionized ultrapure water, DH_2O
 Teflon beaker

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M NaNO_3 stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.
- Label 26 polycarbonate centrifuge tubes (50 ml capacity) QCU-pHi (where i is the approximate pH of each solution, see Table QCU).
 - Label 1 polycarbonate centrifuge tube QC10.0U-IU.
 - Weigh and record the weight of each container.
 - About 1L of ^{233}U solution will be required for this experiment.
 - Add ~35 g of the 50 ppb ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.*

- Record weight of each container.

Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the QC10.0-U-IU container.
- Record weight of each sample container after addition of quartz.

Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaHCO_3 to each experimental container as listed in Table QC10.0U. Take care to add HNO_3 or NaHCO_3 in the concentrations specified in Table CQ0U.
- For sample QC10.0U-IU (not listed in Table QC10.0U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

Sample QC10.0U-IU for initial [U].

- Label two 7 ml LSC vials as QC10.0U-IUa and QC10.0U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from QC10.0U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container QC10.0U-IU.

Wait for equilibrium.

- Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
- Place containers on gyrotory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.

Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., QC10.0U-pH3a and QC10.0U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

Transfer mixture of quartz and clinoptilolite for desorption.

- Record weight of PC sample containers. *Note: this step can be skipped if the mixture transfer is done immediately after the last step in section 7.4. A5*
- Centrifuge polycarbonate containers containing experimental solutions at 10,000 rpm for 15 min to remove fine particles from suspension.
- For each PC sample container, label two corresponding polypropylene (PP) test tubes A and B(50 ml capacity).
- Record weight for both set of PP tubes ~~A and B~~ **A5**
- Add 3.0 ml of 0.1M HNO3 to 50 ml PP tubes labeled as **B-QC10.0Ud-pH**.
- Pour supernatant from PC containers into PP tubes with acid and labeled as **B-QC10.0Ud-pH** leaving behind about 7 to 8ml of solution in PC container with the solid mixture.
- Record the weight of PC containers containing the solid mixture and some solution.
- Stir the solid mixture in solution vigorously and immediately transfer all the solid and solution to the corresponding PP tubes marked as **A-QC10.0Ud-pH**.
- Record the weight of PP tubes **A-QC10.0Ud-pH**.
- Transfer supernatant with HNO3 acid from the PP tubes **B-QC10.0Ud-pH** back to original PC containers.
- Record weights of PC sample containers.
- Add approximately 3 ml of 0.1 M HNO₃ to PP tubes containing solid mixture, **A-QC10.0Ud-pH**.
- Record weights of PP tubes **A-QC10.0Ud-pH** and PC sample containers.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

C. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QC10.0Ud-pH3a and QC10.0Ud-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquot of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table QC10.0U: Estimated solution pH and volumes of HNO3 or NaHCO3 solutions needed for adjustment of pH in 0.1 m NaNO3 solutions with 50 ppb U in contact with qtz/clinop (35 ml)

| Estimated solution pH | Volume of HNO3 added, ml | Molarity of HNO3 to use |
|-----------------------|----------------------------|---------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO3 added, ml | Molarity of NaHCO3 to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |

QC10.0U - IU

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| Sample Name | Original Container wt., g |
|----------------|---------------------------|
| QC10U-pH 3.0 | 22.2461 |
| QC10U-pH 3.5 | 22.2512 |
| QC10U-pH 4.0 | 22.2562 |
| QQC10U-pH 4.25 | 22.128 |
| QQC10U-pH 4.5 | 22.2214 |
| QC1.0U-pH4.75 | 22.2285 |
| QC10U-pH 5.0 | 22.2894 |
| QC10U-pH 5.25 | 22.2356 |
| QC10U-pH 5.5 | 22.1926 |
| QC10U-pH 5.75 | 22.2095 |
| QC10U-pH 6.0 | 22.2183 |
| QC10U-pH 6.25 | 22.1481 |
| QC10U-pH 6.5 | 22.2123 |
| QC10U-pH 6.75 | 22.1355 |
| QC10U-pH 7.0 | 22.1952 |
| QC10U-pH 7.25 | 22.3005 |
| QC10U-pH 7.5 | 22.5150 |
| QC10U-pH 7.75 | 22.1720 |
| QC10U-pH 8.0 | 22.2748 |
| QC10U-pH 8.25 | 22.1974 |
| QC10U-pH 8.5 | 22.2697 |
| QC10U-pH 8.75 | 22.1780 |
| QC10U-pH 9.0 | 22.2977 |
| QC10U-pH 9.25 | 22.2218 |
| QC10U-pH 9.5 | 22.4932 |
| QC10U-IU | |

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| Sample Name | Container wt. + U soln., g |
|----------------|----------------------------|
| QC10U-pH 3.0 | 57.2563 |
| QC10U-pH 3.5 | 57.2677 |
| QC10U-pH 4.0 | 57.2494 |
| QQC10U-pH 4.25 | 57.1432 |
| QQC10U-pH 4.5 | 57.2285 |
| QC1.0U-pH4.75 | 57.2555 |
| QC10U-pH 5.0 | 57.311 |
| QC10U-pH 5.25 | 57.2587 |
| QC10U-pH 5.5 | 57.2103 |
| QC10U-pH 5.75 | 57.2282 |
| QC10U-pH 6.0 | 57.24 |
| QC10U-pH 6.25 | 57.1674 |
| QC10U-pH 6.5 | 57.2249 |
| QC10U-pH 6.75 | 57.1419 |
| QC10U-pH 7.0 | 57.2183 |
| QC10U-pH 7.25 | 57.335 |
| QC10U-pH 7.5 | 57.536 |
| QC10U-pH 7.75 | 57.2002 |
| QC10U-pH 8.0 | 57.2982 |
| QC10U-pH 8.25 | 57.2287 |
| QC10U-pH 8.5 | 57.2952 |
| QC10U-pH 8.75 | 57.1998 |
| QC10U-pH 9.0 | 57.3223 |
| QC10U-pH 9.25 | 57.2453 |
| QC10U-pH 9.5 | 57.5162 |

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| Sample Name | Container wt. + U soln. + quartz, g |
|----------------|-------------------------------------|
| QC10U-pH 3.0 | 58.2531 |
| QC10U-pH 3.5 | 58.2708 |
| QC10U-pH 4.0 | 58.2519 |
| QQC10U-pH 4.25 | 58.1417 |
| QQC10U-pH 4.5 | 58.2303 |
| QC1.0U-pH4.75 | 58.2553 |
| QC10U-pH 5.0 | 58.3094 |
| QC10U-pH 5.25 | 58.2609 |
| QC10U-pH 5.5 | 58.2129 |
| QC10U-pH 5.75 | 58.2294 |
| QC10U-pH 6.0 | 58.2412 |
| QC10U-pH 6.25 | 58.1696 |
| QC10U-pH 6.5 | 58.2251 |
| QC10U-pH 6.75 | 58.1451 |
| QC10U-pH 7.0 | 58.2198 |
| QC10U-pH 7.25 | 58.3348 |
| QC10U-pH 7.5 | 58.5383 |
| QC10U-pH 7.75 | 58.1976 |
| QC10U-pH 8.0 | 58.296 |
| QC10U-pH 8.25 | 58.2291 |
| QC10U-pH 8.5 | 58.2974 |
| QC10U-pH 8.75 | 58.2014 |
| QC10U-pH 9.0 | 58.3217 |
| QC10U-pH 9.25 | 58.2444 |
| QC10U-pH 9.5 | 58.5163 |

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| Sample Name | Container wt. + U soln. + quartz + clinop, g |
|----------------|--|
| QC10U-pH 3.0 | 58.6105 |
| QC10U-pH 3.5 | 58.6291 |
| QC10U-pH 4.0 | 58.6044 |
| QQC10U-pH 4.25 | 58.5003 |
| QQC10U-pH 4.5 | 58.5894 |
| QC1.0U-pH4.75 | 58.6145 |
| QC10U-pH 5.0 | 58.6693 |
| QC10U-pH 5.25 | 58.6174 |
| QC10U-pH 5.5 | 58.5734 |
| QC10U-pH 5.75 | 58.5902 |
| QC10U-pH 6.0 | 58.6021 |
| QC10U-pH 6.25 | 58.5295 |
| QC10U-pH 6.5 | 58.5851 |
| QC10U-pH 6.75 | 58.5002 |
| QC10U-pH 7.0 | 58.5742 |
| QC10U-pH 7.25 | 58.6932 |
| QC10U-pH 7.5 | 58.8912 |
| QC10U-pH 7.75 | 58.5542 |
| QC10U-pH 8.0 | 58.6541 |
| QC10U-pH 8.25 | 58.586 |
| QC10U-pH 8.5 | 58.6555 |
| QC10U-pH 8.75 | 58.5556 |
| QC10U-pH 9.0 | 58.6783 |
| QC10U-pH 9.25 | 58.5961 |
| QC10U-pH 9.5 | 58.8698 |

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| Sample Name | Container Wt. After additions of HNO3 and NaHCO3, g |
|----------------|---|
| QC10U-pH 3.0 | 59.0062 |
| QC10U-pH 3.5 | 58.7378 |
| QC10U-pH 4.0 | 58.6758 |
| QCC10U-pH 4.25 | 58.5837 |
| QCC10U-pH 4.5 | 58.8808 |
| QC1.0U-pH4.75 | 58.8179 |
| QC10U-pH 5.0 | 58.9078 |
| QC10U-pH 5.25 | 58.8745 |
| QC10U-pH 5.5 | 58.8952 |
| QC10U-pH 5.75 | 58.9236 |
| QC10U-pH 6.0 | 58.9002 |
| QC10U-pH 6.25 | 58.8498 |
| QC10U-pH 6.5 | 58.6586 |
| QC10U-pH 6.75 | 58.5691 |
| QC10U-pH 7.0 | 58.6838 |
| QC10U-pH 7.25 | 58.8369 |
| QC10U-pH 7.5 | 59.1058 |
| QC10U-pH 7.75 | 58.723 |
| QC10U-pH 8.0 | 58.9372 |
| QC10U-pH 8.25 | 58.6958 |
| QC10U-pH 8.5 | 58.8348 |
| QC10U-pH 8.75 | 58.7331 |
| QC10U-pH 9.0 | 59.0121 |
| QC10U-pH 9.25 | 59.4747 |
| QC10U-pH 9.5 | 60.1318 |

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| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|----------------|--|-------------|---|
| QC10U-pH 3.0 | 58.2581 | 3.33 | 57.2281 |
| QC10U-pH 3.5 | 58.192 | 4.09 | 57.1605 |
| QC10U-pH 4.0 | 57.7203 | 4.95 | 56.6878 |
| QCC10U-pH 4.25 | 57.6966 | 5.37 | 56.6606 |
| QCC10U-pH 4.5 | 58.0359 | 5.94 | 56.9892 |
| QC1.0U-pH4.75 | 58.0791 | 6.17 | 57.0511 |
| QC10U-pH 5.0 | 58.1816 | 6.38 | 57.1589 |
| QC10U-pH 5.25 | 58.0076 | 6.4 | 56.9783 |
| QC10U-pH 5.5 | 58.2206 | 6.66 | 57.1848 |
| QC10U-pH 5.75 | 57.913 | 6.76 | 56.8819 |
| QC10U-pH 6.0 | 58.1497 | 6.58 | 57.1353 |
| QC10U-pH 6.25 | 57.8511 | 6.72 | 56.823 |
| QC10U-pH 6.5 | 57.945 | 6.67 | 56.9217 |
| QC10U-pH 6.75 | 57.6963 | 6.68 | 56.6692 |
| QC10U-pH 7.0 | 57.5858 | 7.07 | 56.5626 |
| QC10U-pH 7.25 | 57.7721 | 7.29 | 56.7502 |
| QC10U-pH 7.5 | 58.1379 | 7.46 | 57.1065 |
| QC10U-pH 7.75 | 57.9802 | 7.7 | 56.9411 |
| QC10U-pH 8.0 | 58.2211 | 7.94 | 57.1919 |
| QC10U-pH 8.25 | 57.7166 | 8.22 | 56.6868 |
| QC10U-pH 8.5 | 58.1778 | 8.42 | 57.1551 |
| QC10U-pH 8.75 | 58.2655 | 8.72 | 57.2298 |
| QC10U-pH 9.0 | 58.3952 | 8.96 | 57.3774 |
| QC10U-pH 9.25 | 58.8248 | 9.23 | 57.8061 |
| QC10U-pH 9.5 | 59.7944 | 9.16 | 58.7531 |

1/6/99
1/5/99

| Sample Name | wt. of vial , 9 | wt. Of vial + sample , 9 |
|----------------|-----------------|--------------------------|
| QC10U-pH 3a | 7.9815 | 8.4772 |
| QC10U-pH 3b | 7.8747 | 8.3721 |
| QC10U-pH 3.5a | 7.862 | 8.3645 |
| QC10U-pH 3.5b | 7.8448 | 8.3459 |
| QC10U-pH 4a | 7.8717 | 8.3662 |
| QC10U-pH 4b | 7.8578 | 8.3557 |
| QC10U-pH 4.25a | 7.9213 | 8.4163 |
| QC10U-pH 4.25b | 7.8476 | 8.3466 |
| QC10U-pH 4.5a | 7.9402 | 8.4421 |
| QC10U-pH 4.5b | 7.8646 | 8.3636 |
| QC10U-pH 4.75a | 7.8611 | 8.3636 |
| QC10U-pH4.75b | 7.9154 | 8.4158 |
| QC10U-pH 5a | 7.8724 | 8.3696 |
| QC10U-pH 5b | 7.8623 | 8.3592 |
| QC10U-pH 5.25a | 7.8578 | 8.3546 |
| QC10U-pH 5.25b | 7.9878 | 8.4829 |
| QC10U-pH 5.5a | 7.8973 | 8.401 |
| QC10U-pH 5.5b | 7.9184 | 8.4192 |
| QC10U-pH 5.75a | 7.8837 | 8.3813 |
| QC10U-pH 5.75b | 7.8296 | 8.3298 |
| QC10U-pH 6a | 7.939 | 8.4383 |
| QC10U-pH 6b | 7.928 | 8.4257 |
| QC10U-pH 6.25a | 7.8332 | 8.337 |
| QC10U-pH 6.25a | 7.9121 | 8.4093 |
| QC10U-pH 6.5a | 7.8774 | 8.3756 |
| QC10U-pH 6.5b | 7.928 | 8.4329 |
| QC10U-pH 6.75a | 7.837 | 8.4106 |
| QC10U-pH 6.75b | 7.8992 | 8.3235 |
| QC10U-pH 7a | 7.8211 | 8.3272 |
| QC10U-pH 7b | 7.8738 | 8.375 |
| QC10U-pH 7.25a | 7.926 | 8.4248 |
| QC10U-pH 7.25b | 7.9572 | 8.4626 |
| QC10U-pH 7.5a | 7.829 | 8.3343 |
| QC10U-pH 7.5b | 7.8752 | 8.3774 |
| QC10U-pH 7.75a | 7.909 | 8.4157 |
| QC10U-pH 7.75b | 7.7619 | 8.2686 |
| QC10U-pH 8a | 7.8921 | 8.3935 |
| QC10U-pH 8b | 7.9024 | 8.4034 |
| QC10U-pH 8.25a | 7.8461 | 8.3511 |
| QC10U-pH 8.25b | 7.8497 | 8.3451 |
| QC10U-pH 8.5a | 7.8705 | 8.373 |
| QC10U-pH 8.5b | 7.9854 | 8.4913 |
| QC10U-pH 8.75a | 7.9283 | 8.4294 |
| QC10U-pH 8.75b | 7.9191 | 8.4126 |
| QC10U-pH 9a | 7.7834 | 8.2791 |
| QC10U-pH 9b | 7.8361 | 8.3322 |
| QC10U-pH 9.25a | 7.859 | 8.3568 |
| QC10U-pH 9.25 | 7.9304 | 8.4264 |
| QC10U-pH 9.5a | 7.8259 | 8.3311 |
| QC10U-pH 9.5b | 7.8628 | 8.3653 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 07-Jan-99 15:11

C14 Eff (0-156 keV) = 96.88 %

C14 CHI SQUARE IPA DATA PROCESSED - 07-Jan-99 15:21

C14 Chi Square = 22.67

H3 IPA DATA PROCESSED - 07-Jan-99 15:22

H3 Eff (0-18.6 keV) = 65.00 %

H3 CHI SQUARE IPA DATA PROCESSED - 07-Jan-99 15:33

H3 Chi Square = 28.23

BKG IPA DATA PROCESSED - 07-Jan-99 16:33

Bkg (0-18.6 keV) = 22.07 cpm

Bkg (0-156 keV) = 30.93 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 391.15H3 E²/B (1-18.6 keV) = 190.00

08 Jan 99 09:23

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

Page

User : /

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.35 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.56 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.91 | |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|------|----------|------------|--------------|-------------|----------|
| P# 1 | 999.99 | 20.35 1.40 | 3.557 3.35 | 29.91 1.16 | 145.52 |
| 23 | 2 9.59 | 1.96 156.1 | 460.051 3.02 | 461.85 3.10 | 627.36 |
| 23 | 3 9.45 | 0.28 1050 | 467.237 3.02 | 467.44 3.10 | 625.73 |
| 23 | 4 12.45 | 0.00 0.00 | 353.551 3.03 | 353.86 3.14 | 622.93 |
| 23 | 5 12.28 | 0.58 455.9 | 358.658 3.03 | 360.48 3.13 | 627.70 |
| 23 | 6 29.32 | 1.37 127.0 | 148.012 3.07 | 149.62 3.32 | 622.26 |
| 23 | 7 28.23 | 0.00 0.00 | 153.900 3.07 | 153.01 3.33 | 630.77 |
| 23 | 8 50.82 | 1.27 104.9 | 83.889 3.13 | 85.53 3.55 | 621.26 |
| 23 | 9 51.32 | 0.21 632.7 | 83.037 3.13 | 82.83 3.60 | 624.81 |
| 23 | 10 80.43 | 0.81 131.6 | 51.721 3.21 | 52.65 3.90 | 617.03 |

QC10U Sorption
Uncapped

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 11 | 78.84 | 0.25 | 430.6 | 52.823 | 3.21 | 53.58 | 3.90 | 625.68 |
| 23 | 12 | 88.56 | 0.00 | 0.00 | 46.624 | 3.24 | 45.85 | 4.10 | 636.69 |
| 23 | 13 | 88.25 | 0.79 | 128.7 | 46.811 | 3.24 | 47.55 | 4.01 | 617.42 |
| 23 | 14 | 97.34 | 0.30 | 324.6 | 42.097 | 3.27 | 42.38 | 4.15 | 624.45 |
| 23 | 15 | 96.17 | 0.83 | 118.3 | 42.653 | 3.26 | 43.41 | 4.10 | 617.18 |
| 23 | 16 | 90.23 | 0.00 | 0.00 | 45.695 | 3.24 | 45.99 | 4.06 | 628.36 |
| 23 | 17 | 88.77 | 0.90 | 112.6 | 46.516 | 3.24 | 47.52 | 4.00 | 620.11 |
| 23 | 18 | 87.23 | 0.00 | 0.00 | 47.389 | 3.24 | 47.15 | 4.05 | 632.17 |
| 23 | 19 | 86.28 | 0.00 | 0.00 | 47.973 | 3.23 | 47.94 | 4.03 | 630.03 |
| 23 | 20 | 77.84 | 0.78 | 138.4 | 53.547 | 3.21 | 54.14 | 3.89 | 617.65 |
| 23 | 21 | 77.08 | 0.00 | 0.00 | 54.123 | 3.20 | 54.58 | 3.89 | 624.82 |
| 23 | 22 | 86.08 | 0.80 | 128.6 | 48.093 | 3.23 | 49.15 | 3.96 | 619.81 |
| 23 | 23 | 87.17 | 1.27 | 81.45 | 47.424 | 3.23 | 48.71 | 3.96 | 613.83 |
| 23 | 24 | 83.28 | 0.96 | 109.3 | 49.841 | 3.22 | 51.26 | 3.91 | 615.49 |
| 23 | 25 | 84.82 | 1.80 | 58.94 | 48.836 | 3.23 | 50.97 | 3.89 | 608.41 |
| 23 | 26 | 83.23 | 0.00 | 0.00 | 49.849 | 3.22 | 50.13 | 3.97 | 632.85 |
| 23 | 27 | 82.21 | 0.00 | 0.00 | 50.500 | 3.22 | 49.98 | 4.01 | 636.45 |
| 23 | 28 | 78.88 | 0.40 | 265.6 | 52.794 | 3.21 | 52.98 | 3.92 | 625.37 |
| 23 | 29 | 78.77 | 0.52 | 206.0 | 52.860 | 3.21 | 53.47 | 3.90 | 625.35 |
| 23 | 30 | 54.33 | 0.00 | 0.00 | 78.258 | 3.14 | 77.80 | 3.65 | 625.95 |
| 23 | 31 | 56.01 | 0.16 | 767.7 | 75.786 | 3.14 | 76.37 | 3.64 | 627.09 |
| 23 | 32 | 47.19 | 1.28 | 107.8 | 90.637 | 3.12 | 91.85 | 3.52 | 619.55 |
| 23 | 33 | 47.24 | 0.35 | 386.3 | 90.516 | 3.12 | 90.53 | 3.55 | 626.45 |
| 23 | 34 | 37.32 | 0.12 | 1263 | 115.521 | 3.09 | 115.40 | 3.43 | 624.65 |
| 23 | 35 | 37.95 | 2.39 | 65.92 | 113.544 | 3.10 | 115.99 | 3.39 | 613.43 |
| 23 | 36 | 28.90 | 0.51 | 336.0 | 150.249 | 3.07 | 150.33 | 3.33 | 624.07 |
| 23 | 37 | 28.25 | 0.53 | 327.2 | 153.753 | 3.07 | 153.80 | 3.32 | 620.56 |
| 23 | 38 | 19.95 | 1.80 | 118.0 | 219.400 | 3.05 | 221.21 | 3.21 | 624.91 |
| 23 | 39 | 20.01 | 0.00 | 0.00 | 218.582 | 3.05 | 218.11 | 3.23 | 630.17 |
| 23 | 40 | 11.94 | 0.00 | 0.00 | 368.972 | 3.03 | 367.66 | 3.14 | 625.80 |
| 23 | 41 | 12.59 | 1.17 | 224.3 | 349.580 | 3.03 | 350.94 | 3.14 | 624.75 |

10 Jan 99 00:24

ALPHA/BETA = 1.02

Protocol #:23

U-233 3% 2 sigma

Ue

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 10.33 | 0.46 | 619.0 | 426.840 | 3.02 | 428.17 | 3.11 | 625.59 |
| 23 | 43 | 10.42 | 0.95 | 301.6 | 422.930 | 3.03 | 424.98 | 3.11 | 622.95 |
| 23 | 44 | 9.55 | 4.88 | 66.83 | 461.888 | 3.02 | 466.53 | 3.09 | 624.61 |
| 23 | 45 | 10.01 | 3.12 | 98.46 | 440.599 | 3.02 | 444.21 | 3.10 | 626.18 |
| 23 | 46 | 9.30 | 2.01 | 154.7 | 474.723 | 3.02 | 477.18 | 3.10 | 624.46 |
| 23 | 47 | 9.26 | 1.14 | 268.9 | 476.357 | 3.02 | 478.19 | 3.10 | 626.47 |
| 23 | 48 | 9.10 | 2.18 | 145.3 | 484.904 | 3.02 | 487.23 | 3.10 | 627.28 |
| 23 | 49 | 9.41 | 5.47 | 60.78 | 468.813 | 3.02 | 474.23 | 3.09 | 625.63 |
| 23 | 50 | 9.20 | 0.00 | 0.00 | 479.486 | 3.02 | 478.24 | 3.11 | 630.38 |
| 23 | 51 | 9.52 | 0.97 | 309.6 | 463.670 | 3.02 | 464.10 | 3.11 | 633.32 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 10-Jan-99 02:08

C14 Eff (0-156 keV) = 96.57 %

C14 CHI SQUARE IPA DATA PROCESSED - 10-Jan-99 02:19

C14 Chi Square = 22.48

H3 IPA DATA PROCESSED - 10-Jan-99 02:20

H3 Eff (0-18.6 keV) = 65.12 %

UNCAPPED

| Sample Name | 1/6/99 wt. Of PP tubes, g | 1/6/99 wt. Of PP tubes after transfer of qtz/clino, g | 1/6/99 wt. Of PP tubes after acid add, g | 1/6/99 Container wt. after transfer of supernatant, g | 1/6/99 Container wt. after supernatant retransfer+acid added, g |
|----------------|---------------------------------|--|---|--|---|
| QC10U-pH 3.0 | 13.3882 | 20.0566 | 23.0702 | 29.1569 | 53.509 |
| QC10U-pH 3.5 | 14.2561 | 22.7533 | 25.7666 | 30.911 | 51.6603 |
| QC10U-pH 4.0 | 13.4387 | 21.2664 | 24.2717 | 30.253 | 51.7673 |
| QQC10U-pH 4.25 | 13.5213 | 21.8909 | 24.9018 | 30.7126 | 51.1656 |
| QQC10U-pH 4.5 | 13.8959 | 22.4032 | 25.4113 | 30.9227 | 51.3899 |
| QC1.0U-pH4.75 | 13.4965 | 20.7492 | 23.7661 | 29.8166 | 52.609 |
| QC10U-pH 5.0 | 13.84 | 20.8944 | 23.9058 | 29.5268 | 52.9498 |
| QC10U-pH 5.25 | 13.5259 | 22.5365 | 25.5492 | 31.3836 | 50.7881 |
| QC10U-pH 5.5 | 13.3544 | 21.4084 | 24.4173 | 30.3688 | 52.1227 |
| QC10U-pH 5.75 | 13.4256 | 20.2156 | 23.2238 | 29.185 | 53.0853 |
| QC10U-pH 6.0 | 13.3795 | 21.0142 | 24.0287 | 30.2244 | 52.3024 |
| QC10U-pH 6.25 | 13.8493 | 19.9505 | 22.9663 | 28.4309 | 53.5624 |
| QC10U-pH 6.5 | 14.0001 | 20.0864 | 23.091 | 28.5433 | 53.631 |
| QC10U-pH 6.75 | 13.4094 | 18.7669 | 21.7789 | 27.672 | 54.173 |
| QC10U-pH 7.0 | 13.4493 | 19.728 | 22.751 | 28.6904 | 53.062 |
| QC10U-pH 7.25 | 13.3919 | 20.901 | 23.9151 | 30.0244 | 52.0258 |
| QC10U-pH 7.5 | 13.8809 | 20.7569 | 23.7706 | 29.629 | 53.2148 |
| QC10U-pH 7.75 | 13.3392 | 21.0146 | 24.0246 | 30.157 | 52.2518 |
| QC10U-pH 8.0 | 13.4757 | 21.2954 | 24.3075 | 30.4301 | 52.166 |
| QC10U-pH 8.25 | 13.4952 | 20.7627 | 23.7792 | 29.6842 | 52.2201 |
| QC10U-pH 8.5 | 13.5342 | 18.6819 | 21.6887 | 27.7038 | 54.8316 |
| QC10U-pH 8.75 | 13.4621 | 20.9606 | 23.9696 | 29.9143 | 52.4546 |
| QC10U-pH 9.0 | 13.825 | 21.9107 | 24.9219 | 30.5274 | 52.0722 |
| QC10U-pH 9.25 | 13.8604 | 21.2474 | 24.2566 | 29.7688 | 53.2118 |
| QC10U-pH 9.5 | 13.3972 | 24.4548 | 27.467 | 33.6803 | 50.4637 |

| PP Tubes 1/8/99 Desorption UNCAPPED | | |
|-------------------------------------|---------------|----------------------|
| Sample Name | wt. of vial,g | wt. Of vial + sample |
| QC10U-pH 3a | 7.8441 | 8.3431 |
| QC10U-pH 3b | 7.8162 | 8.3165 |
| QC10U-pH 3.5a | 7.8097 | 8.3084 |
| QC10U-pH 3.5b | 7.7645 | 8.264 |
| QC10U-pH 4a | 7.8354 | 8.3334 |
| QC10U-pH 4b | 7.7852 | 8.2843 |
| QC10U-pH 4.25a | 7.7902 | 8.288 |
| QC10U-pH 4.25b | 7.7897 | 8.2889 |
| QC10U-pH 4.5a | 7.8273 | 8.3257 |
| QC10U-pH 4.5b | 7.8853 | 8.3846 |
| QC10U-pH 4.75a | 7.7454 | 8.2435 |
| QC10U-pH 4.75b | 7.7721 | 8.2716 |
| QC10U-pH 5a | 7.7646 | 8.2625 |
| QC10U-pH 5b | 7.7331 | 8.2324 |
| QC10U-pH 5.25a | 7.8044 | 8.3029 |
| QC10U-pH 5.25b | 7.8475 | 8.3484 |
| QC10U-pH 5.5a | 7.8739 | 8.3722 |
| QC10U-pH 5.5b | 7.7586 | 8.2586 |
| QC10U-pH 5.75a | 7.7142 | 8.2128 |
| QC10U-pH 5.75b | 7.7548 | 8.2545 |
| QC10U-pH 6a | 7.8036 | 8.3034 |
| QC10U-pH 6b | 7.8217 | 8.3222 |
| QC10U-pH 6.25a | 7.8287 | 8.3285 |
| QC10U-pH 6.25a | 7.838 | 8.339 |
| QC10U-pH 6.5a | 7.8437 | 8.3413 |
| QC10U-pH 6.5b | 7.7685 | 8.2681 |
| QC10U-pH 6.75a | 7.7051 | 8.203 |
| QC10U-pH 6.75b | 7.7788 | 8.2784 |
| QC10U-pH 7a | 7.8408 | 8.3386 |
| QC10U-pH 7b | 7.769 | 8.268 |
| QC10U-pH 7.25a | 7.8008 | 8.2986 |
| QC10U-pH 7.25b | 7.8011 | 8.3011 |
| QC10U-pH 7.5a | 7.8291 | 8.3281 |
| QC10U-pH 7.5b | 7.7744 | 8.2732 |
| QC10U-pH 7.75a | 7.8215 | 8.3191 |
| QC10U-pH 7.75b | 7.7816 | 8.2803 |
| QC10U-pH 8a | 7.8056 | 8.3029 |
| QC10U-pH 8b | 7.7656 | 8.2641 |
| QC10U-pH 8.25a | 7.7695 | 8.2664 |
| QC10U-pH 8.25b | 7.8101 | 8.3093 |
| QC10U-pH 8.5a | 7.8167 | 8.3138 |
| QC10U-pH 8.5b | 7.7944 | 8.2941 |
| QC10U-pH 8.75a | 7.7785 | 8.275 |
| QC10U-pH 8.75b | 7.7312 | 8.2301 |
| QC10U-pH 9a | 7.8504 | 8.3035 |
| QC10U-pH 9b | 7.7502 | 8.2515 |
| QC10U-pH 9.25a | 7.7603 | 8.258 |
| QC10U-pH 9.25 | 7.7748 | 8.2726 |
| QC10U-pH 9.5a | 7.8392 | 8.3385 |
| QC10U-pH 9.5b | 7.7281 | 8.2282 |

| 1/8/99 PC containers UNCAPPED Desorption | | |
|--|---------------|----------------------|
| Sample Name | wt. of vial,g | wt. Of vial + sample |
| QC10U-pH 3a | 7.7803 | 8.2853 |
| QC10U-pH 3b | 7.8482 | 8.3511 |
| QC10U-pH 3.5a | 7.8068 | 8.3038 |
| QC10U-pH 3.5b | 7.8119 | 8.3129 |
| QC10U-pH 4a | 7.745 | 8.2461 |
| QC10U-pH 4b | 7.7708 | 8.2719 |
| QC10U-pH 4.25a | 7.7823 | 8.281 |
| QC10U-pH 4.25b | 7.7472 | 8.2466 |
| QC10U-pH 4.5a | 7.8096 | 8.3097 |
| QC10U-pH 4.5b | 7.7568 | 8.2602 |
| QC10U-pH 4.75a | 7.8262 | 8.3256 |
| QC10U-pH 4.75b | 7.798 | 8.2992 |
| QC10U-pH 5a | 7.8346 | 8.334 |
| QC10U-pH 5b | 7.7508 | 8.2513 |
| QC10U-pH 5.25a | 7.7753 | 8.2763 |
| QC10U-pH 5.25b | 7.8278 | 8.3288 |
| QC10U-pH 5.5a | 7.8407 | 8.3402 |
| QC10U-pH 5.5b | 7.7697 | 8.27 |
| QC10U-pH 5.75a | 7.8084 | 8.3085 |
| QC10U-pH 5.75b | 7.7865 | 8.2891 |
| QC10U-pH 6a | 7.7326 | 8.2319 |
| QC10U-pH 6b | 7.7951 | 8.3032 |
| QC10U-pH 6.25a | 7.8627 | 8.3621 |
| QC10U-pH 6.25a | 7.8206 | 8.3224 |
| QC10U-pH 6.5a | 7.7389 | 8.2381 |
| QC10U-pH 6.5b | 7.8328 | 8.3345 |
| QC10U-pH 6.75a | 7.804 | 8.3052 |
| QC10U-pH 6.75b | 7.7915 | 8.293 |
| QC10U-pH 7a | 7.8314 | 8.3298 |
| QC10U-pH 7b | 7.8115 | 8.3182 |
| QC10U-pH 7.25a | 7.7523 | 8.2504 |
| QC10U-pH 7.25b | 7.8341 | 8.3348 |
| QC10U-pH 7.5a | 7.7532 | 8.253 |
| QC10U-pH 7.5b | 7.8058 | 8.3063 |
| QC10U-pH 7.75a | 7.7938 | 8.293 |
| QC10U-pH 7.75b | 7.8053 | 8.3061 |
| QC10U-pH 8a | 7.829 | 8.3282 |
| QC10U-pH 8b | 7.7638 | 8.2634 |
| QC10U-pH 8.25a | 7.7746 | 8.2723 |
| QC10U-pH 8.25b | 7.8279 | 8.3281 |
| QC10U-pH 8.5a | 7.8466 | 8.346 |
| QC10U-pH 8.5b | 7.8052 | 8.3081 |
| QC10U-pH 8.75a | 7.7873 | 8.2857 |
| QC10U-pH 8.75b | 7.8115 | 8.3135 |
| QC10U-pH 9a | 7.7743 | 8.2731 |
| QC10U-pH 9b | 7.828 | 8.3278 |
| QC10U-pH 9.25a | 7.7108 | 8.2086 |
| QC10U-pH 9.25 | 7.7956 | 8.297 |
| QC10U-pH 9.5a | 7.84 | 8.3379 |
| QC10U-pH 9.5b | 7.8142 | 8.315 |

UNCAPPED DESORPTION

| Sample Name | 1/18/99 Container wt. before sampling | 1/18/99 Container wt. after sampling | 1/18/99 wt. Of PP tubes before sampling | 1/18/99 wt. Of PP tubes after sampling |
|----------------|---|--|---|--|
| QC10U-pH 3.0 | 53.4050 | 52.3936 | 23.0291 | 22.0220 |
| QC10U-pH 3.5 | 51.5187 | 50.5175 | 25.7543 | 24.7494 |
| QC10U-pH 4.0 | 51.6495 | 50.6443 | 24.2478 | 23.2431 |
| QCC10U-pH 4.25 | 51.0448 | 50.0442 | 24.8573 | 23.8511 |
| QCC10U-pH 4.5 | 51.2532 | 50.2461 | 25.3549 | 24.3498 |
| QC1.0U-pH4.75 | 52.4787 | 51.4757 | 23.7543 | 22.7514 |
| QC10U-pH 5.0 | 52.8509 | 51.8476 | 23.8942 | 22.8872 |
| QC10U-pH 5.25 | 50.6711 | 49.6676 | 25.539 | 24.5332 |
| QC10U-pH 5.5 | 51.9925 | 50.9905 | 24.4097 | 23.3976 |
| QC10U-pH 5.75 | 52.9976 | 51.9918 | 23.2182 | 22.2142 |
| QC10U-pH 6.0 | 52.1927 | 51.1798 | 24.0186 | 23.0127 |
| QC10U-pH 6.25 | 53.4618 | 52.4555 | 22.9576 | 21.9498 |
| QC10U-pH 6.5 | 53.5266 | 52.521 | 23.0822 | 22.0796 |
| QC10U-pH 6.75 | 54.0993 | 53.0933 | 21.7703 | 20.7683 |
| QC10U-pH 7.0 | 52.9162 | 51.9075 | 22.7343 | 21.7325 |
| QC10U-pH 7.25 | 51.866 | 50.8649 | 23.9042 | 22.9016 |
| QC10U-pH 7.5 | 53.0183 | 52.0161 | 23.746 | 22.7434 |
| QC10U-pH 7.75 | 52.1199 | 51.1178 | 24.0128 | 23.0127 |
| QC10U-pH 8.0 | 52.0464 | 51.045 | 24.2992 | 23.2972 |
| QC10U-pH 8.25 | 52.0512 | 51.051 | 23.7733 | 22.7712 |
| QC10U-pH 8.5 | 54.696 | 53.6903 | 21.6696 | 20.6664 |
| QC10U-pH 8.75 | 52.295 | 51.291 | 23.9604 | 22.9546 |
| QC10U-pH 9.0 | 51.9474 | 50.9437 | 24.898 | 23.8962 |
| QC10U-pH 9.25 | 53.0564 | 52.0546 | 24.2437 | 23.2422 |
| QC10U-pH 9.5 | 50.3413 | 49.3402 | 24.4555 | 23.4468 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 19-Jan-99 10:52
 C14 Eff (0-156 keV) = 96.71 %
 C14 CHI SQUARE IPA DATA PROCESSED - 19-Jan-99 11:03
 C14 Chi Square = 13.26
 H3 IPA DATA PROCESSED - 19-Jan-99 11:04
 H3 Eff (0-18.6 keV) = 65.23 %
 H3 CHI SQUARE IPA DATA PROCESSED - 19-Jan-99 11:15
 H3 Chi Square = 19.48
 BKG IPA DATA PROCESSED - 19-Jan-99 12:15
 Bkg (0-18.6 keV) = 21.17 cpm
 Bkg (0-156 keV) = 30.75 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 380.09
 H3 E²/B (1-18.6 keV) = 198.70
 20 Jan 99 05:05 ALPHA/BETA - 1.02
 Protocol #:23 U-233 3% 2 sigma

Page #:

User : Alka

Time: 999.99
 Data Mode: CPM
 Background Subtract: 1st Vial
 Nuclide: MANUAL

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.20 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.39 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.57 | |

Quench Indicator: SIS
 alpha cpm U-233 1st vial bkgnd
 Coincidence Time(ns): 18
 Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|-------|--------|------------|--------------|------------|-------------|
| P# 1 | 999.99 | 20.20 1.41 | 3.386 3.44 | 29.57 | 1.16 141.21 |
| 23 2 | 10.45 | 4.01 76.16 | 422.260 3.02 | 425.26 | 3.10 616.93 |
| 23 3 | 10.22 | 2.02 147.0 | 431.448 3.02 | 434.32 | 3.10 618.29 |
| 23 4 | 13.58 | 1.60 159.3 | 323.860 3.03 | 324.85 | 3.15 624.42 |
| 23 5 | 13.70 | 3.45 76.53 | 321.066 3.03 | 324.37 | 3.14 618.04 |
| 23 6 | 29.31 | 0.89 193.5 | 148.269 3.07 | 149.51 | 3.31 610.13 |
| 23 7 | 28.66 | 0.00 0.00 | 151.673 3.07 | 151.03 | 3.33 622.55 |
| 23 8 | 48.15 | 0.12 1153 | 88.909 3.12 | 88.73 | 3.55 618.27 |
| 23 9 | 47.01 | 1.22 112.6 | 91.168 3.11 | 92.53 | 3.50 616.23 |
| 23 10 | 67.85 | 0.76 150.6 | 62.126 3.17 | 62.69 | 3.76 621.91 |
| 23 11 | 65.47 | 0.45 254.9 | 64.492 3.16 | 65.13 | 3.73 614.85 |
| 23 12 | 60.80 | 0.66 182.9 | 69.739 3.15 | 69.87 | 3.69 621.34 |
| 23 13 | 60.26 | 0.65 187.1 | 70.361 3.15 | 71.16 | 3.67 618.92 |
| 23 14 | 78.39 | 0.00 0.00 | 53.305 3.20 | 53.09 | 3.92 627.20 |
| 23 15 | 76.19 | 0.29 368.2 | 54.955 3.19 | 55.44 | 3.86 622.84 |

QC10U
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| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 16 | 81.38 | 0.55 | 192.1 | 51.222 | 3.21 | 51.70 | 3.92 | 616.02 |
| 23 | 17 | 81.62 | 0.90 | 117.1 | 51.098 | 3.21 | 51.79 | 3.91 | 617.73 |
| 23 | 18 | 81.64 | 0.00 | 0.00 | 51.048 | 3.21 | 51.39 | 3.93 | 627.43 |
| 23 | 19 | 80.17 | 1.01 | 105.8 | 52.084 | 3.20 | 53.73 | 3.85 | 613.42 |
| 23 | 20 | 71.47 | 0.86 | 130.2 | 58.836 | 3.18 | 59.95 | 3.78 | 626.45 |
| 23 | 21 | 68.24 | 0.07 | 1598 | 61.737 | 3.17 | 62.22 | 3.77 | 619.71 |
| 23 | 22 | 74.03 | 0.44 | 246.3 | 56.644 | 3.19 | 57.45 | 3.82 | 621.79 |
| 23 | 23 | 73.42 | 1.21 | 91.93 | 57.142 | 3.18 | 58.67 | 3.78 | 612.15 |
| 23 | 24 | 70.42 | 0.00 | 0.00 | 59.735 | 3.18 | 60.05 | 3.80 | 625.10 |
| 23 | 25 | 69.10 | 0.00 | 0.00 | 60.956 | 3.17 | 60.72 | 3.81 | 634.52 |
| 23 | 26 | 67.30 | 0.56 | 204.2 | 62.647 | 3.17 | 63.77 | 3.73 | 621.12 |
| 23 | 27 | 65.97 | 0.00 | 0.00 | 63.978 | 3.16 | 63.21 | 3.79 | 626.63 |
| 23 | 28 | 66.44 | 0.06 | 1819 | 63.516 | 3.17 | 63.64 | 3.76 | 616.25 |
| 23 | 29 | 63.19 | 1.69 | 71.64 | 66.957 | 3.16 | 69.27 | 3.64 | 610.97 |
| 23 | 30 | 53.15 | 0.69 | 186.8 | 80.226 | 3.13 | 81.34 | 3.58 | 619.38 |
| 23 | 31 | 47.70 | 0.00 | 0.00 | 89.780 | 3.12 | 90.05 | 3.54 | 619.65 |
| 23 | 32 | 54.65 | 1.10 | 116.1 | 77.931 | 3.13 | 79.49 | 3.58 | 616.13 |
| 23 | 33 | 46.41 | 1.87 | 75.37 | 92.412 | 3.11 | 94.84 | 3.47 | 617.28 |
| 23 | 34 | 38.88 | 1.00 | 150.8 | 110.914 | 3.09 | 112.74 | 3.41 | 621.61 |
| 23 | 35 | 38.42 | 0.50 | 301.3 | 112.283 | 3.09 | 113.04 | 3.42 | 627.76 |
| 23 | 36 | 29.24 | 3.16 | 57.24 | 148.666 | 3.07 | 151.38 | 3.29 | 611.81 |
| 23 | 37 | 30.80 | 0.13 | 1284 | 140.932 | 3.07 | 141.37 | 3.34 | 624.54 |
| 23 | 38 | 21.78 | 2.71 | 76.28 | 200.700 | 3.05 | 204.40 | 3.21 | 616.85 |
| 23 | 39 | 21.39 | 2.62 | 79.63 | 204.515 | 3.05 | 205.91 | 3.23 | 611.05 |
| 23 | 40 | 13.52 | 0.59 | 424.6 | 325.534 | 3.03 | 327.23 | 3.14 | 620.20 |
| 23 | 41 | 13.88 | 1.49 | 168.9 | 316.787 | 3.03 | 318.27 | 3.15 | 618.41 |

21 Jan 99 15:31
Protocol #:23

ALPHA/BETA - 1.02
U-233 3% 2 sigma

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User : A

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 11.30 | 3.34 | 86.75 | 389.888 | 3.03 | 393.26 | 3.11 | 626.10 |
| 23 | 43 | 11.62 | 3.81 | 75.74 | 379.058 | 3.03 | 383.42 | 3.11 | 612.70 |
| 23 | 44 | 10.50 | 1.23 | 233.0 | 420.233 | 3.02 | 422.52 | 3.11 | 617.18 |
| 23 | 45 | 10.53 | 3.55 | 85.08 | 418.741 | 3.02 | 423.61 | 3.10 | 622.01 |
| 23 | 46 | 10.58 | 3.91 | 77.63 | 417.030 | 3.02 | 420.24 | 3.10 | 620.27 |
| 23 | 47 | 10.56 | 3.95 | 76.87 | 417.447 | 3.02 | 423.17 | 3.10 | 618.14 |
| 23 | 48 | 10.58 | 1.73 | 167.1 | 416.935 | 3.02 | 419.29 | 3.11 | 627.80 |
| 23 | 49 | 10.26 | 3.68 | 83.20 | 430.045 | 3.02 | 434.27 | 3.10 | 626.58 |
| 23 | 50 | 10.32 | 0.73 | 390.0 | 427.234 | 3.02 | 428.67 | 3.11 | 631.44 |
| 23 | 51 | 10.29 | 0.41 | 700.0 | 428.781 | 3.02 | 428.93 | 3.11 | 628.18 |

79
7-259
76
7-256

contains

233 MISSING TUBE(S)

| | | | | | | | | | |
|-----|-----------------|-------|-------|---------|----------|--------|---------|--------|--------|
| 233 | MISSING TDD (S) | | | | | | | | |
| 55 | 9.06 | 2.65 | 120.3 | 487.232 | 3.02 | 490.63 | 3.09 | 621.00 | |
| 23 | 56 | 8.84 | 2.32 | 138.4 | 499.781 | 3.02 | 504.03 | 3.08 | 613.54 |
| 23 | 57 | 5.93 | 4.76 | 86.38 | 746.867 | 3.01 | 750.36 | 3.06 | 618.11 |
| 23 | 58 | 5.70 | 4.01 | 102.9 | 776.439 | 3.01 | 781.13 | 3.05 | 615.75 |
| 23 | 59 | 3.21 | 8.15 | 72.99 | 1383.218 | 3.01 | 1392.24 | 3.02 | 613.51 |
| 23 | 60 | 3.17 | 5.67 | 100.9 | 1399.453 | 3.01 | 1404.18 | 3.03 | 615.49 |
| 23 | 61 | 2.97 | 4.05 | 141.4 | 1494.594 | 3.01 | 1501.07 | 3.02 | 615.42 |
| 23 | 62 | 3.02 | 9.94 | 63.64 | 1471.448 | 3.00 | 1482.02 | 3.02 | 616.16 |
| 23 | 63 | 2.87 | 8.38 | 75.42 | 1546.091 | 3.01 | 1554.40 | 3.02 | 608.12 |
| 23 | 64 | 2.87 | 5.59 | 107.4 | 1547.485 | 3.00 | 1554.75 | 3.02 | 616.00 |
| 23 | 65 | 2.53 | 14.19 | 52.00 | 1754.322 | 3.00 | 1769.64 | 3.01 | 611.09 |
| 23 | 66 | 2.56 | 13.01 | 55.42 | 1735.677 | 3.00 | 1750.51 | 3.01 | 613.37 |
| 23 | 67 | 2.47 | 15.03 | 50.30 | 1798.233 | 3.00 | 1814.96 | 3.01 | 604.80 |
| 23 | 68 | 2.38 | 9.64 | 73.54 | 1866.362 | 3.00 | 1876.73 | 3.02 | 608.80 |
| 23 | 69 | 2.90 | 16.70 | 42.75 | 1533.855 | 3.00 | 1551.46 | 3.01 | 608.49 |
| 23 | 70 | 2.98 | 12.35 | 53.55 | 1488.225 | 3.01 | 1500.63 | 3.02 | 614.25 |
| 23 | 71 | 2.71 | 5.27 | 116.6 | 1643.108 | 3.00 | 1649.76 | 3.02 | 614.42 |
| 23 | 72 | 2.81 | 8.99 | 71.80 | 1583.091 | 3.00 | 1594.63 | 3.02 | 606.27 |
| 23 | 73 | 2.33 | 10.28 | 70.44 | 1904.339 | 3.01 | 1915.06 | 3.02 | 609.11 |
| 23 | 74 | 2.37 | 7.65 | 89.67 | 1879.315 | 3.00 | 1888.15 | 3.01 | 604.66 |
| 23 | 75 | 2.57 | 9.76 | 69.99 | 1733.190 | 3.00 | 1742.80 | 3.01 | 612.72 |
| 23 | 76 | 2.22 | 10.43 | 71.25 | 2000.218 | 3.00 | 2007.82 | 3.02 | 612.69 |
| 23 | 77 | 2.65 | 12.26 | 57.15 | 1674.350 | 3.01 | 1687.79 | 3.02 | 610.68 |
| 23 | 78 | 2.08 | 10.09 | 75.67 | 2134.114 | 3.00 | 2143.99 | 3.02 | 616.27 |
| 23 | 79 | 2.14 | 10.18 | 74.09 | 2076.988 | 3.00 | 2088.19 | 3.01 | 614.20 |
| 23 | 80 | 2.23 | 15.68 | 51.20 | 1995.269 | 3.00 | 2011.24 | 3.01 | 610.57 |
| 23 | 81 | 1.96 | 8.89 | 86.76 | 2271.614 | 3.00 | 2280.12 | 3.01 | 609.42 |
| 23 | 82 | 1.92 | 14.70 | 58.04 | 2311.198 | 3.00 | 2327.72 | 3.01 | 607.37 |
| 23 | 83 | 2.31 | 6.21 | 109.0 | 1923.887 | 3.00 | 1931.47 | 3.02 | 609.23 |
| 23 | 84 | 2.37 | 17.78 | 45.06 | 1873.407 | 3.00 | 1894.90 | 3.01 | 603.86 |
| 23 | 85 | 2.74 | 6.45 | 96.85 | 1621.797 | 3.00 | 1627.36 | 3.02 | 612.51 |
| 23 | 86 | 2.73 | 16.07 | 45.40 | 1626.651 | 3.00 | 1644.79 | 3.01 | 610.87 |
| 23 | 87 | 2.67 | 13.89 | 51.50 | 1666.277 | 3.00 | 1682.79 | 3.01 | 610.28 |
| 23 | 88 | 2.62 | 8.81 | 75.59 | 1695.851 | 3.00 | 1706.69 | 3.02 | 613.40 |
| 23 | 89 | 3.13 | 8.56 | 70.91 | 1420.576 | 3.00 | 1429.21 | 3.02 | 610.01 |
| 23 | 90 | 3.10 | 8.84 | 69.34 | 1433.388 | 3.00 | 1442.04 | 3.02 | 607.46 |
| 23 | 91 | 4.28 | 1.77 | 257.0 | 1035.866 | 3.01 | 1035.38 | 3.05 | 617.28 |
| 23 | 92 | 4.23 | 9.59 | 55.42 | 1048.387 | 3.01 | 1059.79 | 3.03 | 611.60 |
| 23 | 93 | 6.93 | 48.35 | 13.02 | 639.038 | 3.01 | 688.75 | 2.96 | 487.19 |
| 23 | 94 | 6.31 | 6.75 | 61.41 | 701.527 | 3.01 | 710.21 | 3.05 | 611.43 |
| 23 | 95 | 8.99 | 3.83 | 85.68 | 491.497 | 3.02 | 496.01 | 3.08 | 611.01 |
| 23 | 96 | 9.14 | 4.42 | 74.52 | 483.047 | 3.02 | 487.39 | 3.09 | 611.92 |
| 23 | 97 | 12.39 | 3.77 | 74.08 | 355.290 | 3.03 | 358.97 | 3.12 | 613.85 |
| 23 | 98 | 12.65 | 0.99 | 263.1 | 348.235 | 3.03 | 350.74 | 3.13 | 611.61 |
| 23 | 99 | 14.89 | 26.95 | 13.25 | 295.069 | 3.03 | 321.60 | 3.02 | 468.22 |
| 23 | 100 | 12.77 | 2.04 | 129.9 | 344.617 | 3.03 | 347.09 | 3.13 | 615.49 |

21 Jan 99 21:05 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

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| | | | | | | | | | |
|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 101 | 14.11 | 3.19 | 81.17 | 311.568 | 3.03 | 315.86 | 3.13 | 613.22 |
| 23 | 102 | 13.86 | 1.74 | 145.7 | 317.537 | 3.03 | 318.77 | 3.15 | 603.72 |
| 23 | 103 | 17.81 | 2.49 | 91.45 | 246.361 | 3.04 | 249.88 | 3.17 | 617.68 |
| 23 | 104 | 17.97 | 2.01 | 111.6 | 243.971 | 3.04 | 246.56 | 3.18 | 616.99 |

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23
SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 21-Jan-99 22:20

C14 Eff (0-156 keV) = 96.78 %

C14 CHI SQUARE IPA DATA PROCESSED - 21-Jan-99 22:30

C14 Chi Square = 17.40

H3 IPA DATA PROCESSED - 21-Jan-99 22:31

H3 Eff (0-18.6 keV) = 64.86 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves
& view historic data

H3 CHI SQUARE IPA DATA PROCESSED - 21-Jan-99 22:42

H3 Chi Square = 15.87

BKG IPA DATA PROCESSED - 21-Jan-99 23:42

Bkg (0-18.6 keV) = 22.17 cpm

Bkg (0-156 keV) = 32.03 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 382.47

H3 E²/B (1-18.6 keV) = 189.46

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CAPPED, $\alpha t_2 = 1.00 \text{ g}$, $\text{Clinop} = 0.35 \text{ g}$, $V = 35 \text{ ml}$

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C.

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma U = 50 \text{ ppb}$ (2.14×10^{-7})
- Capped containers
- pH range 4.0-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 350mg (0.35 g)
- M/V = 28.57 g/L

Equipment: Orion 920A pH meter and combination pH electrode
Gyratory shaker
Fisher Marathon 21K centrifuge
Mettler PM4600 and AE240 balances
Packard 2500 TR/AB LSC
Eppendorf micropipettors and tips
Oxford pipettes and tips
Repipettor for transfer of scintillation cocktail

Supplies: $4510 \times UL \times RC \times RFe \times HL \times S325 \text{ AJ}$
 $LDV \times UC \times RC \times RFe \times HL \times 100/200 \times Na6 \text{ AJ}$
50 ppb ²³³U stock solution
polycarbonate centrifuge tubes with caps (50 ml capacity)
polypropylene centrifuge tubes with caps (50 ml capacity)
pH buffer solutions
Ultima-Gold liquid scintillation cocktail
7 ml scintillation vials
reagent grade NaOH (lot 976631) AJ
concentrated HNO₃ (lot 983546)
HNO₃ and NaOH stock solutions at various concentrations prepared in degassed water.
0.1M NaNO₃ matrix prepared with degassed water.
weighing paper/boats
deionized ultrapure water, DH₂O
Teflon beaker

Note: Ultrapure water was degassed by boiling it in a flask and cooled with mouth covered with rubber stopper.

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
 - On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M degassed NaNO₃ stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ²³³U stock solution to experimental containers.
 - Label 26 polycarbonate centrifuge tubes (50 ml capacity) QCU-pHi (where i is the approximate pH of each solution, see Table QCU).
 - Label 1 polycarbonate centrifuge tube QC10.0U-IU.
 - Weigh and record the weight of each container.

- About 1L of ^{233}U solution will be required for this experiment.
- Add ~35 g of the 50 ppb ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.*
- Record weight of each container.

C. Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the QC10.0-U-IU container.
- Record weight of each sample container after addition of quartz.

D. Add clinoptilolite to sample containers.

- Add 0.35 g of clinoptilolite to each sample container. **Do not** add clinoptilolite to the QC10.0-U-IU container.
- Record weight of each sample container after addition of clinoptilolite.

E. Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaOH to each experimental container as listed in Table QC10.0U. Take care to add HNO_3 or NaOH in the concentrations specified in Table CQ10U.
- For sample QC10.0U-IU (not listed in Table QC10.0U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

F. Sample QC10.0U-IU for initial [U].

- Label two 7 ml LSC vials as QC10.0U-IUa and QC10.0U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from QC10.0U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container QC10.0U-IU.

G. Wait for equilibrium.

- Place caps on sample containers and tighten the caps.
- Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate for at least 10 days.

H. Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., QC10.0U-pH3a and QC10.0U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.

- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

I. Transfer mixture of quartz and clinoptilolite for desorption.

- Record weight of PC sample containers. *Note: this step can be skipped if the mixture transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate containers containing experimental solutions at 10,000 rpm for 15 min to remove fine particles from suspension.
- For each PC sample container, label two corresponding polypropylene (PP) test tubes A and B (50 ml capacity).
- Record weight for both set of PP tubes A and B.
- Add 3.0 ml of 0.1M HNO_3 to 50 ml PP tubes labeled as B-QC10.0Ud-pH.
- Pour supernatant from PC containers into PP tubes with acid and labeled as B-QC10.0Ud-pH leaving behind about 7 to 8ml of solution in PC container with the solid mixture.
- Record the weight of PC containers containing the solid mixture and some solution.
- Stir the solid mixture in solution vigorously and immediately transfer all the solid and solution to the corresponding PP tubes marked as A-QC10.0Ud-pH.
- Record the weight of PP tubes A-QC10.0Ud-pH.
- Transfer supernatant with HNO_3 acid from the PP tubes B-QC10.0Ud-pH back to original PC containers.
- Record weights of PC sample containers.
- Add approximately 3 ml of 0.1 M HNO_3 to PP tubes containing solid mixture, A-QC10.0Ud-pH.
- Record weights of PP tubes A-QC10.0Ud-pH and PC sample containers.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

J. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QC10.0Ud-pH3a and QC10.0Ud-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquot of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table CQ10U: Estimated solution pH and volumes of HNO₃ or NaOH solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with quartz and clinoptilolite (35ml).

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--------------------------------------|-------------------------------------|
| 3 | 0.060 | 1 |
| 3.5 | 0.205 | 0.1 |
| 4 | 0.385 | 0.01 |
| 4.25 | 0.190 | 0.01 |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use |
| 4.50 | 0.09 | 0.01 |
| 4.75 | 0.150 | 0.01 |
| 5 | 0.200 | 0.01 |
| 5.25 | 0.225 | 0.01 |
| 5.5 | 0.250 | 0.01 |
| 5.75 | 0.275 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.325 | 0.01 |
| 6.5 | 0.350 | 0.01 |
| 6.75 | 0.375 | 0.01 |
| 7 | 0.400 | 0.01 |
| 7.25 | 0.050 | 0.05 |
| 7.5 | 0.115 | 0.05 |
| 7.75 | 0.130 | 0.05 |
| 8 | 0.175 | 0.05 |
| 8.25 | 0.150 | 0.05 |
| 8.5 | 0.160 | 0.1 |
| 8.75 | 0.100 | 0.1 |
| 9 | 0.125 | 0.1 |
| 9.25 | 0.150 | 0.1 |
| 9.5 | 0.175 | 0.1 |
| QC10U-IU | 0.100 | 50% HNO ₃ |

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| Sample Name | Original Container wt., g |
|----------------|---------------------------|
| QC10U-pH 3.0 | 22.1594 |
| QC10U-pH 3.5 | 22.2558 |
| QC10U-pH 4.0 | 22.1769 |
| QQC10U-pH 4.25 | 22.2406 |
| QQC10U-pH 4.5 | 22.1356 |
| QC10U-pH 4.75 | 22.1431 |
| QC10U-pH 5.0 | 22.2298 |
| QC10U-pH 5.25 | 22.4727 |
| QC10U-pH 5.5 | 22.5386 |
| QC10U-pH 5.75 | 22.2069 |
| QC10U-pH 6.0 | 22.253 |
| QC10U-pH 6.25 | 22.133 |
| QC10U-pH 6.5 | 22.1541 |
| QC10U-pH 6.75 | 22.2216 |
| QC10U-pH 7.0 | 22.2538 |
| QC10U-pH 7.25 | 22.1982 |
| QC10U-pH 7.5 | 22.1939 |
| QC10U-pH 7.75 | 22.1712 |
| QC10U-pH 8.0 | 22.4646 |
| QC10U-pH 8.25 | 22.9947 |
| QC10U-pH 8.5 | 22.1262 |
| QC10U-pH 8.75 | 22.2643 |
| QC10U-pH 9.0 | 22.2225 |
| QC10U-pH 9.25 | 22.236 |
| QC10U-pH 9.5 | 22.303 |
| QC10U-IU | 22.2969 |

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| Sample Name | Container wt. + U soln., g |
|----------------|----------------------------|
| QC10U-pH 3.0 | 57.1645 |
| QC10U-pH 3.5 | 57.2676 |
| QC10U-pH 4.0 | 57.2032 |
| QQC10U-pH 4.25 | 57.2618 |
| QQC10U-pH 4.5 | 57.1745 |
| QC10U-pH 4.75 | 57.168 |
| QC10U-pH 5.0 | 57.2242 |
| QC10U-pH 5.25 | 57.4896 |
| QC10U-pH 5.5 | 57.5372 |
| QC10U-pH 5.75 | 57.2339 |
| QC10U-pH 6.0 | 57.2495 |
| QC10U-pH 6.25 | 57.1492 |
| QC10U-pH 6.5 | 57.148 |
| QC10U-pH 6.75 | 57.2192 |
| QC10U-pH 7.0 | 57.2629 |
| QC10U-pH 7.25 | 57.2304 |
| QC10U-pH 7.5 | 57.203 |
| QC10U-pH 7.75 | 57.1932 |
| QC10U-pH 8.0 | 57.479 |
| QC10U-pH 8.25 | 57.985 |
| QC10U-pH 8.5 | 57.1556 |
| QC10U-pH 8.75 | 57.2708 |
| QC10U-pH 9.0 | 57.2416 |
| QC10U-pH 9.25 | 57.2037 |
| QC10U-pH 9.5 | 57.3100 |
| QC10U-IU | 57.2885 |

CAPPED

12/29/98

| Sample Name | Container wt. + U soln. + quartz, g |
|----------------|-------------------------------------|
| QC10U-pH 3.0 | 58.1574 |
| QC10U-pH 3.5 | 58.2719 |
| QC10U-pH 4.0 | 58.205 |
| QQC10U-pH 4.25 | 58.2596 |
| QQC10U-pH 4.5 | 58.177 |
| QC10U-pH 4.75 | 58.1669 |
| QC10U-pH 5.0 | 58.2202 |
| QC10U-pH 5.25 | 58.4916 |
| QC10U-pH 5.5 | 58.539 |
| QC10U-pH 5.75 | 58.2328 |
| QC10U-pH 6.0 | 58.247 |
| QC10U-pH 6.25 | 58.1453 |
| QC10U-pH 6.5 | 58.1284 |
| QC10U-pH 6.75 | 58.2171 |
| QC10U-pH 7.0 | 58.2672 |
| QC10U-pH 7.25 | 58.227 |
| QC10U-pH 7.5 | 58.2011 |
| QC10U-pH 7.75 | 58.1912 |
| QC10U-pH 8.0 | 58.4745 |
| QC10U-pH 8.25 | 58.9797 |
| QC10U-pH 8.5 | 58.1518 |
| QC10U-pH 8.75 | 58.2675 |
| QC10U-pH 9.0 | 58.2408 |
| QC10U-pH 9.25 | 58.2 |
| QC10U-pH 9.5 | 58.3137 |

CAPPED

12/29/98

| Sample Name | Container wt. + U soln. + quartz + clinop, g |
|----------------|--|
| QC10U-pH 3.0 | 58.5081 |
| QC10U-pH 3.5 | 58.6227 |
| QC10U-pH 4.0 | 58.5577 |
| QQC10U-pH 4.25 | 58.6089 |
| QQC10U-pH 4.5 | 58.4393 |
| QC10U-pH 4.75 | 58.5199 |
| QC10U-pH 5.0 | 58.5719 |
| QC10U-pH 5.25 | 58.8402 |
| QC10U-pH 5.5 | 58.8874 |
| QC10U-pH 5.75 | 58.5831 |
| QC10U-pH 6.0 | 58.597 |
| QC10U-pH 6.25 | 58.4971 |
| QC10U-pH 6.5 | 58.4713 |
| QC10U-pH 6.75 | 58.5676 |
| QC10U-pH 7.0 | 58.6107 |
| QC10U-pH 7.25 | 58.5771 |
| QC10U-pH 7.5 | 58.5495 |
| QC10U-pH 7.75 | 58.5429 |
| QC10U-pH 8.0 | 58.8267 |
| QC10U-pH 8.25 | 58.3288 |
| QC10U-pH 8.5 | 58.5033 |
| QC10U-pH 8.75 | 58.6146 |
| QC10U-pH 9.0 | 58.5941 |
| QC10U-pH 9.25 | 58.5508 |
| QC10U-pH 9.5 | 58.6664 |

CAPPED

12/30/98

| Sample Name | Container Wt. After additions of HNO3 and NaHCO3, g |
|----------------|---|
| QC10U-pH 3.0 | 58.5608 |
| QC10U-pH 3.5 | 58.7677 |
| QC10U-pH 4.0 | 58.6958 |
| QQC10U-pH 4.25 | 58.622 |
| QQC10U-pH 4.5 | 58.5266 |
| QC10U-pH 4.75 | 58.6689 |
| QC10U-pH 5.0 | 58.771 |
| QC10U-pH 5.25 | 58.0645 |
| QC10U-pH 5.5 | 58.1359 |
| QC10U-pH 5.75 | 58.8564 |
| QC10U-pH 6.0 | 58.2195 |
| QC10U-pH 6.25 | 58.8439 |
| QC10U-pH 6.5 | 58.7953 |
| QC10U-pH 6.75 | 58.9417 |
| QC10U-pH 7.0 | 59.0095 |
| QC10U-pH 7.25 | 58.6277 |
| QC10U-pH 7.5 | 58.6227 |
| QC10U-pH 7.75 | 58.6404 |
| QC10U-pH 8.0 | 58.9507 |
| QC10U-pH 8.25 | 58.4763 |
| QC10U-pH 8.5 | 58.5767 |
| QC10U-pH 8.75 | 58.713 |
| QC10U-pH 9.0 | 58.719 |
| QC10U-pH 9.25 | 58.6997 |
| QC10U-pH 9.5 | 58.8413 |
| QC10U-IU | 57.4141 |

CAPPED

| Sample Name | 1/11/99 Container wt. before sampling & pH measurements, g | 1/11/99 Measured pH | 1/11/99 Container wt. after sampling & pH measurements, g |
|----------------|---|------------------------|--|
| QC10U-pH 3.0 | 58.4978 | 3.17 | 57.4708 |
| QC10U-pH 3.5 | 58.7498 | 3.8 | 57.7109 |
| QC10U-pH 4.0 | 58.7433 | 4.59 | 57.7105 |
| QQC10U-pH 4.25 | 58.7132 | 4.84 | 57.6836 |
| QQC10U-pH 4.5 | 58.6085 | 5.01 | 57.5768 |
| QC10U-pH 4.75 | 58.6975 | 5.35 | 57.6448 |
| QC10U-pH 5.0 | 58.7558 | 5.62 | 57.7464 |
| QC10U-pH 5.25 | 58.0156 | 5.75 | 57.9918 |
| QC10U-pH 5.5 | 58.1269 | 5.92 | 58.1054 |
| QC10U-pH 5.75 | 58.8535 | 6.01 | 57.8303 |
| QC10U-pH 6.0 | 58.3461 | 6.78 | 58.3204 |
| QC10U-pH 6.25 | 58.8238 | 6.47 | 57.8059 |
| QC10U-pH 6.5 | 58.7246 | 6.41 | 57.6867 |
| QC10U-pH 6.75 | 58.8426 | 6.64 | 57.7232 |
| QC10U-pH 7.0 | 58.9809 | 6.7 | 57.9601 |
| QC10U-pH 7.25 | 58.729 | 6.93 | 57.6996 |
| QC10U-pH 7.5 | 58.6124 | 7.04 | 57.5763 |
| QC10U-pH 7.75 | 58.7626 | 7.16 | 57.7164 |
| QC10U-pH 8.0 | 58.8764 | 7.27 | 57.8487 |
| QC10U-pH 8.25 | 58.4862 | 7.5 | 57.4569 |
| QC10U-pH 8.5 | 58.5491 | 7.6 | 57.5213 |
| QC10U-pH 8.75 | 58.6516 | 8.6 | 57.6339 |
| QC10U-pH 9.0 | 58.7752 | 8.85 | 57.7366 |
| QC10U-pH 9.25 | 58.6744 | 9.24 | 57.6430 |
| QC10U-pH 9.5 | 58.8578 | 9.26 | 57.8170 |
| QC10U-IU | 57.2861 | | 56.2850 |

actual amounts added

Table CQ10U: Estimated solution pH and volumes of HNO3 or NaOH solutions needed for adjustment of pH in 0.1 m NaNO3 solutions with 50 ppb U in contact with qz/clino (35 ml)

| Estimated solution pH | Volume of HNO3 added, ml | Molarity of HNO3 to use | |
|-----------------------|--------------------------|-------------------------|--------------------------------|
| 3 | 0.060 | 1 | |
| 3.5 | 0.205 | 0.1 | |
| 4 | 0.385 | 0.01 | |
| 4.25 | 0.190 | 0.01 | |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use | Volume of 0.01M HNO3 added, ml |
| 4.50 | 0.09 | 0.01 | 0.125 |
| 4.75 | 0.150 | 0.01 | 0.075 |
| 5 | 0.200 | 0.01 | 0.050 |
| 5.25 | 0.225 | 0.01 | 0.045 |
| 5.5 | 0.250 | 0.01 | 0.050 |
| 5.75 | 0.275 | 0.01 | 0.040 |
| 6 | 0.300 | 0.01 | 0.200 |
| 6.25 | 0.325 | 0.01 | 0.380 |
| 6.5 | 0.350 | 0.01 | 0.335 |
| 6.75 | 0.375 | 0.01 | |
| 7 | 0.400 | 0.01 | |
| 7.25 | 0.050 | 0.05 | |
| 7.5 | 0.115 | 0.05 | |
| 7.75 | 0.130 | 0.05 | |
| 8 | 0.175 | 0.05 | |
| 8.25 | 0.150 | 0.05 | |
| 8.5 | 0.160 | 0.1 | |
| 8.75 | 0.100 | 0.1 | |
| 9 | 0.125 | 0.1 | |
| 9.25 | 0.150 | 0.1 | 0.020 |
| 9.5 | 0.175 | 0.1 | |
| QC10U-IU | 0.100 | 50% HNO3 | |

15 Jan 99 06:47 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page
User : A

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | LL | UL | LCR | 2S% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 19.71 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.44 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 28.97 |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

*QC100, Capped
SORPTION*

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|----|----------|------------|--------------|-------------|----------|
| P# | 1 999.99 | 19.71 1.42 | 3.443 3.41 | 28.97 1.18 | 145.55 B |
| 23 | 2 8.74 | 0.77 399.1 | 505.024 3.02 | 506.50 3.09 | 630.16 |
| 23 | 3 8.75 | 2.80 114.8 | 504.786 3.02 | 507.26 3.09 | 625.77 |
| 23 | 4 9.91 | 2.59 116.3 | 444.993 3.02 | 448.83 3.10 | 623.79 |
| 23 | 5 9.69 | 6.09 53.79 | 455.277 3.02 | 462.57 3.08 | 621.24 |
| 23 | 6 15.99 | 0.99 231.5 | 274.543 3.04 | 277.35 3.16 | 624.14 |
| 23 | 7 15.57 | 3.86 64.15 | 282.106 3.04 | 286.38 3.15 | 621.72 |
| 23 | 8 19.08 | 0.99 211.9 | 229.471 3.05 | 230.67 3.20 | 624.63 |
| 23 | 9 18.59 | 2.34 93.68 | 235.718 3.04 | 238.92 3.18 | 622.39 |
| 23 | 10 29.03 | 1.41 123.0 | 149.674 3.07 | 151.15 3.30 | 620.49 |
| 23 | 11 29.47 | 1.36 125.9 | 147.490 3.07 | 149.11 3.31 | 624.19 |
| 23 | 12 39.80 | 0.31 459.2 | 108.240 3.10 | 108.64 3.44 | 619.02 |
| 23 | 13 40.27 | 1.20 122.5 | 106.962 3.10 | 109.05 3.41 | 619.05 |
| 23 | 14 53.80 | 0.85 149.6 | 79.159 3.13 | 80.60 3.57 | 621.27 |
| 23 | 15 54.04 | 1.51 84.79 | 78.792 3.13 | 80.19 3.57 | 616.62 |
| 23 | 16 62.13 | 0.44 266.0 | 68.101 3.16 | 69.18 3.67 | 625.40 |
| 23 | 17 64.40 | 1.35 87.51 | 65.563 3.16 | 67.89 3.65 | 618.19 |
| 23 | 18 63.74 | 0.89 131.7 | 66.435 3.16 | 68.21 3.65 | 627.09 |
| 23 | 19 64.11 | 1.07 109.9 | 65.875 3.16 | 67.71 3.66 | 620.21 |
| 23 | 20 68.45 | 0.45 248.9 | 61.480 3.17 | 62.09 3.76 | 621.86 |
| 23 | 21 69.21 | 0.20 553.8 | 60.767 3.18 | 61.78 3.75 | 626.69 |
| 23 | 22 42.03 | 1.66 87.79 | 102.362 3.10 | 104.65 3.42 | 621.18 |
| 23 | 23 41.35 | 1.64 89.07 | 104.054 3.10 | 106.58 3.41 | 620.99 |
| 23 | 24 67.54 | 0.94 120.8 | 62.355 3.17 | 63.67 3.72 | 619.12 |
| 23 | 25 64.68 | 1.38 85.33 | 65.295 3.16 | 66.64 3.68 | 616.24 |
| 23 | 26 67.50 | 0.99 115.9 | 62.394 3.17 | 64.11 3.70 | 617.24 |
| 23 | 27 67.12 | 0.97 118.1 | 62.767 3.17 | 64.68 3.69 | 617.31 |
| 23 | 28 66.04 | 0.88 130.4 | 63.865 3.17 | 65.29 3.70 | 625.39 |
| 23 | 29 67.83 | 0.00 0.00 | 62.103 3.17 | 62.23 3.77 | 628.69 |
| 23 | 30 62.29 | 1.99 60.84 | 67.901 3.16 | 70.66 3.61 | 611.24 |
| 23 | 31 62.96 | 0.00 0.00 | 67.173 3.16 | 67.43 3.70 | 632.67 |
| 23 | 32 76.83 | 0.33 317.1 | 54.399 3.20 | 54.67 3.87 | 623.59 |

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 33 | 74.13 | 0.00 | 0.00 | 56.506 | 3.19 | 56.92 | 3.83 | 630.32 |
| 23 | 34 | 59.22 | 1.25 | 98.14 | 71.599 | 3.15 | 73.43 | 3.61 | 614.10 |
| 23 | 35 | 62.78 | 2.18 | 55.79 | 67.344 | 3.16 | 69.71 | 3.63 | 612.25 |
| 23 | 36 | 46.88 | 1.49 | 92.04 | 91.352 | 3.12 | 93.64 | 3.47 | 618.96 |
| 23 | 37 | 45.33 | 1.56 | 89.86 | 94.594 | 3.11 | 97.35 | 3.45 | 618.09 |
| 23 | 38 | 38.50 | 0.00 | 0.00 | 111.986 | 3.09 | 112.33 | 3.42 | 629.04 |
| 23 | 39 | 39.37 | 2.13 | 71.04 | 109.435 | 3.10 | 112.23 | 3.39 | 618.22 |
| 23 | 40 | 52.73 | 0.52 | 242.0 | 80.854 | 3.13 | 82.50 | 3.55 | 627.56 |
| 23 | 41 | 52.49 | 2.05 | 64.40 | 81.221 | 3.13 | 83.72 | 3.52 | 614.99 |

16 Jan 99 15:00 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page
User : A

| | | | | | | | | | |
|----|----|--------|------|-------|--------|------|-------|------|--------|
| 23 | 42 | 47.26 | 1.26 | 108.1 | 90.590 | 3.12 | 92.42 | 3.49 | 619.25 |
| 23 | 43 | 48.80 | 0.74 | 178.9 | 87.623 | 3.12 | 88.59 | 3.52 | 619.65 |
| 23 | 44 | 94.02 | 0.62 | 157.9 | 43.898 | 3.24 | 45.59 | 3.98 | 624.93 |
| 23 | 45 | 90.18 | 0.85 | 117.3 | 45.836 | 3.24 | 46.88 | 3.98 | 615.50 |
| 23 | 46 | 108.30 | 1.46 | 63.44 | 37.591 | 3.29 | 40.24 | 4.06 | 604.93 |
| 23 | 47 | 109.78 | 0.71 | 127.3 | 37.038 | 3.29 | 38.13 | 4.20 | 614.07 |
| 23 | 48 | 126.08 | 1.97 | 44.52 | 31.812 | 3.34 | 34.73 | 4.21 | 601.19 |
| 23 | 49 | 131.70 | 0.11 | 767.0 | 30.300 | 3.36 | 31.40 | 4.45 | 632.82 |
| 23 | 50 | 53.87 | 1.23 | 104.0 | 79.070 | 3.13 | 81.17 | 3.55 | 617.50 |
| 23 | 51 | 53.99 | 1.16 | 109.5 | 78.887 | 3.13 | 80.20 | 3.57 | 620.13 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 17-Jan-99 05:46

C14 Eff (0-156 keV) = 96.57 %

C14 CHI SQUARE IPA DATA PROCESSED - 17-Jan-99 05:56

C14 Chi Square = 14.26

H3 IPA DATA PROCESSED - 17-Jan-99 05:58

H3 Eff (0-18.6 keV) = 65.55 %

H3 CHI SQUARE IPA DATA PROCESSED - 17-Jan-99 06:08

H3 Chi Square = 15.41

BKG IPA DATA PROCESSED - 17-Jan-99 07:08

Bkg (0-18.6 keV) = 22.27 cpm

Bkg (0-156 keV) = 31.42 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 397.03

H3 E²/B (1-18.6 keV) = 192.95

CAPPED SORPTION

1/11/99

1/11/99

| Sample Name | wt. of vial | wt. Of vial + sample |
|----------------|-------------|----------------------|
| QC10U-pH 3a | 7.7837 | 8.2866 |
| QC10U-pH 3b | 7.9141 | 8.4181 |
| QC10U-pH 3.5a | 7.862 | 8.369 |
| QC10U-pH 3.5b | 7.8844 | 8.3959 |
| QC10U-pH 4a | 7.8321 | 8.3379 |
| QC10U-pH 4b | 7.8863 | 8.3949 |
| QC10U-pH 4.25a | 7.9267 | 8.431 |
| QC10U-pH 4.25b | 7.861 | 8.3662 |
| QC10U-pH 4.5a | 7.8618 | 8.3649 |
| QC10U-pH 4.5b | 7.8541 | 8.3606 |
| QC10U-pH 4.75a | 7.7981 | 8.3004 |
| QC10U-pH4.75b | 7.8422 | 8.3447 |
| QC10U-pH 5a | 7.8548 | 8.3612 |
| QC10U-pH 5b | 7.824 | 8.3269 |
| QC10U-pH 5.25a | 7.9179 | 8.4209 |
| QC10U-pH 5.25b | 7.8766 | 8.3788 |
| QC10U-pH 5.5a | 7.8783 | 8.3807 |
| QC10U-pH 5.5b | 7.8646 | 8.3674 |
| QC10U-pH 5.75a | 7.9006 | 8.4029 |
| QC10U-pH 5.75b | 7.8196 | 8.3217 |
| QC10U-pH 6a | 7.8938 | 8.3956 |
| QC10U-pH 6b | 7.9612 | 8.4638 |
| QC10U-pH 6.25a | 8.7933 | 8.2939 |
| QC10U-pH 6.25a | 7.9327 | 8.436 |
| QC10U-pH 6.5a | 7.8427 | 8.3467 |
| QC10U-pH 6.5b | 7.9057 | 8.4109 |
| QC10U-pH 6.75a | 7.9143 | 8.4169 |
| QC10U-pH 6.75b | 7.8638 | 8.3675 |
| QC10U-pH 7a | 7.804 | 8.3006 |
| QC10U-pH 7b | 7.9084 | 8.4108 |
| QC10U-pH 7.25a | 7.8469 | 8.3526 |
| QC10U-pH 7.25b | 7.9181 | 8.4214 |
| QC10U-pH 7.5a | 7.8658 | 8.3669 |
| QC10U-pH 7.5b | 7.8466 | 8.3502 |
| QC10U-pH 7.75a | 7.8454 | 8.3459 |
| QC10U-pH 7.75b | 7.8453 | 8.3489 |
| QC10U-pH 8a | 7.9125 | 8.4159 |
| QC10U-pH 8b | 7.8734 | 8.3764 |
| QC10U-pH 8.25a | 7.9482 | 8.4523 |
| QC10U-pH 8.25b | 7.8902 | 8.3954 |
| QC10U-pH 8.5a | 7.8631 | 8.3641 |
| QC10U-pH 8.5b | 7.887 | 8.3889 |
| QC10U-pH 8.75a | 7.86 | 8.3605 |
| QC10U-pH 8.75b | 7.886 | 8.3876 |

| | | |
|----------------|--------|--------|
| QC10U-pH 9a | 7.9279 | 8.4303 |
| QC10U-pH 9b | 7.8859 | 8.3885 |
| QC10U-pH 9.25a | 7.9296 | 8.4316 |
| QC10U-pH 9.25 | 7.867 | 8.3685 |
| QC10U-pH 9.5a | 7.8299 | 8.3361 |
| QC10U-pH 9.5b | 7.8822 | 8.3873 |
| QC10U-lua | 7.9449 | 8.4445 |
| QC10U-lub | 7.9161 | 8.4172 |

CAPPED

| Sample Name | 1/13/99 wt. Of PP tubes, g | 1/13/99 wt. Of PP tubes after transfer of qtz/clino, g | 1/13/99 wt. Of PP tubes after acid add, g | 1/13/99 Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer+acid added ,g |
|----------------|----------------------------------|---|--|---|---|
| QC10U-pH 3.0 | 13.6813 | 19.8772 | 22.8917 | 28.5038 | 54.2301 |
| QC10U-pH 3.5 | 13.7468 | 22.2644 | 25.29 | 30.8796 | 52.1295 |
| QC10U-pH 4.0 | 13.8652 | 22.5024 | 25.5145 | 30.9919 | 52.0106 |
| QQC10U-pH 4.25 | 13.6692 | 23.2113 | 26.2413 | 31.9711 | 51.0112 |
| QQC10U-pH 4.5 | 13.6636 | 22.3014 | 25.3463 | 30.9296 | 51.7904 |
| QC1.0U-pH4.75 | 13.84 | 21.3549 | 24.3978 | 30.1765 | 52.6843 |
| QC10U-pH 5.0 | 13.8628 | 23.5048 | 26.5293 | 32.0717 | 51.0529 |
| QC10U-pH 5.25 | 13.673 | 20.4494 | 23.4915 | 29.4862 | 54.0945 |
| QC10U-pH 5.5 | 13.6769 | 20.7575 | 23.7842 | 29.8424 | 53.949 |
| QC10U-pH 5.75 | 13.1407 | 20.7956 | 23.8268 | 29.991 | 53.1365 |
| QC10U-pH 6.0 | 13.6871 | 21.29 | 24.3137 | 30.0156 | 53.6434 |
| QC10U-pH 6.25 | 13.7045 | 20.9043 | 23.9591 | 29.5488 | 53.584 |
| QC10U-pH 6.5 | 13.6684 | 22.1018 | 25.1402 | 30.747 | 52.1253 |
| QC10U-pH 6.75 | 13.3205 | 19.6056 | 22.6429 | 28.6153 | 54.3981 |
| QC10U-pH 7.0 | 13.6804 | 21.2994 | 24.3347 | 30.0281 | 53.3042 |
| QC10U-pH 7.25 | 13.6975 | 20.8965 | 23.9304 | 29.6246 | 53.4606 |
| QC10U-pH 7.5 | 13.6759 | 22.5802 | 25.668 | 31.2918 | 51.5506 |
| QC10U-pH 7.75 | 13.3118 | 20.2618 | 23.3082 | 29.2881 | 53.7104 |
| QC10U-pH 8.0 | 13.6778 | 20.1252 | 23.176 | 29.1026 | 54.3482 |
| QC10U-pH 8.25 | 13.2921 | 22.2529 | 25.2987 | 31.1902 | 51.3317 |
| QC10U-pH 8.5 | 13.29 | 20.2786 | 23.3274 | 29.2569 | 53.4716 |
| QC10U-pH 8.75 | 13.7101 | 21.8255 | 24.8739 | 30.7748 | 52.2201 |
| QC10U-pH 9.0 | 13.8633 | 19.9431 | 22.9744 | 28.41 | 54.5586 |
| QC10U-pH 9.25 | 13.7524 | 20.312 | 23.3641 | 28.9155 | 54.0762 |
| QC10U-pH 9.5 | 13.6349 | 21.4969 | 24.534 | 30.3054 | 52.8766 |

CAPPED DESORPTION

1/25/99

| Sample Name | Container wt. before sampling | Container wt. after sampling | wt. Of PP tubes before sampling | wt. Of PP tubes after sampling |
|----------------|-------------------------------|------------------------------|---------------------------------|--------------------------------|
| QC10U-pH 3.0 | 54.1277 | 53.1124 | 22.8862 | 21.8761 |
| QC10U-pH 3.5 | 52.0317 | 51.022 | 25.2837 | 24.2751 |
| QC10U-pH 4.0 | 51.8994 | 50.3873 | 25.5088 | 24.5033 |
| QCC10U-pH 4.25 | 50.9058 | 49.8977 | 26.2329 | 25.2196 |
| QCC10U-pH 4.5 | 51.704 | 50.6947 | 25.3348 | 24.312 |
| QC1.0U-pH4.75 | 52.5667 | 51.5566 | 24.3904 | 23.3772 |
| QC10U-pH 5.0 | 50.958 | 49.9483 | 26.5232 | 25.5193 |
| QC10U-pH 5.25 | 53.9638 | 52.9517 | 23.4841 | 22.4716 |
| QC10U-pH 5.5 | 53.8371 | 52.8188 | 23.7769 | 22.7664 |
| QC10U-pH 5.75 | 53.05 | 52.0445 | 23.8195 | 22.8145 |
| QC10U-pH 6.0 | 53.5398 | 52.5345 | 24.306 | 23.299 |
| QC10U-pH 6.25 | 53.491 | 52.4855 | 23.9528 | 22.9134 |
| QC10U-pH 6.5 | 52.0235 | 51.0201 | 25.1324 | 24.1279 |
| QC10U-pH 6.75 | 54.301 | 53.293 | 22.6291 | 21.6237 |
| QC10U-pH 7.0 | 53.2056 | 52.1962 | 24.3282 | 23.3231 |
| QC10U-pH 7.25 | 53.3751 | 52.3681 | 23.9245 | 22.9153 |
| QC10U-pH 7.5 | 51.4424 | 50.4379 | 25.6517 | 24.6146 |
| QC10U-pH 7.75 | 53.617 | 52.6087 | 23.3017 | 22.283 |
| QC10U-pH 8.0 | 54.201 | 53.1851 | 23.17 | 22.1531 |
| QC10U-pH 8.25 | 51.1777 | 50.1671 | 25.2923 | 24.2534 |
| QC10U-pH 8.5 | 53.3624 | 52.358 | 23.3005 | 22.2846 |
| QC10U-pH 8.75 | 52.1256 | 51.1172 | 24.8666 | 23.8545 |
| QC10U-pH 9.0 | 54.4779 | 53.4757 | 22.9668 | 21.9467 |
| QC10U-pH 9.25 | 53.9985 | 52.9899 | 23.3586 | 22.3241 |
| QC10U-pH 9.5 | 52.7837 | 51.7782 | 24.5277 | 23.5172 |

QC10U-IV 57.3295 56.3212

PP Tubes

1/25/99

Desorption

| Sample Name | wt. of vial,g | wt. Of vial + sample |
|----------------|---------------|----------------------|
| QC10U-pH 3a | 7.8669 | 8.3696 |
| QC10U-pH 3b | 7.8743 | 8.371 |
| QC10U-pH 3.5a | 7.8493 | 8.3473 |
| QC10U-pH 3.5b | 7.8779 | 8.3798 |
| QC10U-pH 4a | 7.7634 | 8.2647 |
| QC10U-pH 4b | 7.765 | 8.2666 |
| QC10U-pH 4.25a | 7.7082 | 8.2086 |
| QC10U-pH 4.25b | 7.7512 | 8.2538 |
| QC10U-pH 4.5a | 7.7637 | 8.2703 |
| QC10U-pH 4.5b | 7.8494 | 8.3455 |
| QC10U-pH 4.75a | 7.8378 | 8.3333 |
| QC10U-pH4.75b | 7.7837 | 8.2837 |
| QC10U-pH 5a | 7.7893 | 8.2862 |
| QC10U-pH 5b | 7.7769 | 8.2748 |
| QC10U-pH 5.25a | 7.7933 | 8.2929 |
| QC10U-pH 5.25b | 7.8065 | 8.3034 |
| QC10U-pH 5.5a | 7.8096 | 8.3022 |
| QC10U-pH 5.5b | 7.7594 | 8.254 |
| QC10U-pH 5.75a | 7.7142 | 8.2152 |
| QC10U-pH 5.75b | 7.8307 | 8.326 |
| QC10U-pH 6a | 7.83 | 8.3312 |
| QC10U-pH 6b | 7.8017 | 8.3006 |
| QC10U-pH 6.25a | 7.7968 | 8.3116 |
| QC10U-pH 6.25a | 7.7939 | 8.3094 |
| QC10U-pH 6.5a | 7.7628 | 8.259 |
| QC10U-pH 6.5b | 7.7576 | 8.2555 |
| QC10U-pH 6.75a | 7.7682 | 8.2633 |
| QC10U-pH 6.75b | 7.8103 | 8.312 |
| QC10U-pH 7a | 7.804 | 8.2995 |
| QC10U-pH 7b | 7.8072 | 8.3071 |
| QC10U-pH 7.25a | 7.794 | 8.2928 |
| QC10U-pH 7.25b | 7.7759 | 8.2723 |
| QC10U-pH 7.5a | 7.768 | 8.2776 |
| QC10U-pH 7.5b | 7.876 | 8.3806 |
| QC10U-pH 7.75a | 7.8258 | 8.3285 |
| QC10U-pH 7.75b | 7.8331 | 8.3309 |
| QC10U-pH 8a | 7.7667 | 8.27 |
| QC10U-pH 8b | 7.7781 | 8.2829 |
| QC10U-pH 8.25a | 7.7507 | 8.2659 |
| QC10U-pH 8.25b | 7.7777 | 8.2902 |
| QC10U-pH 8.5a | 7.7274 | 8.2317 |
| QC10U-pH 8.5b | 7.7499 | 8.2513 |
| QC10U-pH 8.75a | 7.7999 | 8.2971 |
| QC10U-pH 8.75b | 7.7392 | 8.2399 |
| QC10U-pH 9a | 7.7908 | 8.292 |
| QC10U-pH 9b | 7.8239 | 8.3333 |
| QC10U-pH 9.25a | 7.7544 | 8.2563 |
| QC10U-pH 9.25 | 7.7732 | 8.2908 |
| QC10U-pH 9.5a | 7.7401 | 8.2366 |
| QC10U-pH 9.5b | 7.8062 | 8.3087 |
| QC10U-IUa | 7.8134 | 8.3196 |
| QC10U-IUb | 7.8690 | 8.3742 |

1

| 1/25/99 | Capped Desorption | PC containers |
|----------------|-------------------|----------------------|
| Sample Name | wt. of vial,g | wt. Of vial + sample |
| QC10U-pH 3a | 7.7383 | 8.2441 |
| QC10U-pH 3b | 7.7099 | 8.215 |
| QC10U-pH 3.5a | 7.8322 | 8.3353 |
| QC10U-pH 3.5b | 7.7501 | 8.254 |
| QC10U-pH 4a | 7.7782 | 8.7836 |
| QC10U-pH 4b | 7.841 | 8.345 |
| QC10U-pH 4.25a | 7.8387 | 8.3414 |
| QC10U-pH 4.25b | 7.7984 | 8.3015 |
| QC10U-pH 4.5a | 7.727 | 8.2306 |
| QC10U-pH 4.5b | 7.8187 | 8.3226 |
| QC10U-pH 4.75a | 7.7813 | 8.2839 |
| QC10U-pH4.75b | 7.7808 | 8.2853 |
| QC10U-pH 5a | 7.9263 | 8.4279 |
| QC10U-pH 5b | 7.8005 | 8.3061 |
| QC10U-pH 5.25a | 7.8504 | 8.3524 |
| QC10U-pH 5.25b | 7.8584 | 8.3224 |
| QC10U-pH 5.5a | 7.7677 | 8.2803 |
| QC10U-pH 5.5b | 7.81 | 8.3126 |
| QC10U-pH 5.75a | 7.7868 | 8.2892 |
| QC10U-pH 5.75b | 7.8639 | 8.364 |
| QC10U-pH 6a | 7.6968 | 8.1983 |
| QC10U-pH 6b | 7.7414 | 8.2431 |
| QC10U-pH 6.25a | 7.8165 | 8.3172 |
| QC10U-pH 6.25a | 7.796 | 8.2974 |
| QC10U-pH 6.5a | 7.7706 | 8.2722 |
| QC10U-pH 6.5b | 7.7554 | 8.2554 |
| QC10U-pH 6.75a | 7.8287 | 8.3346 |
| QC10U-pH 6.75b | 7.7785 | 8.2792 |
| QC10U-pH 7a | 7.761 | 8.2643 |
| QC10U-pH 7b | 7.7912 | 8.2931 |
| QC10U-pH 7.25a | 7.8094 | 8.3141 |
| QC10U-pH 7.25b | 7.8738 | 8.3753 |
| QC10U-pH 7.5a | 7.8439 | 8.3458 |
| QC10U-pH 7.5b | 7.8019 | 8.3058 |
| QC10U-pH 7.75a | 7.7764 | 8.2788 |
| QC10U-pH 7.75b | 7.7612 | 8.2634 |
| QC10U-pH 8a | 7.8035 | 8.3107 |
| QC10U-pH 8b | 7.8393 | 8.3437 |
| QC10U-pH 8.25a | 7.7642 | 8.2666 |
| QC10U-pH 8.25b | 7.7892 | 8.2913 |
| QC10U-pH 8.5a | 7.7893 | 8.2912 |
| QC10U-pH 8.5b | 7.7983 | 8.2995 |
| QC10U-pH 8.75a | 7.8469 | 8.3507 |
| QC10U-pH 8.75b | 7.7606 | 8.2628 |
| QC10U-pH 9a | 7.7772 | 8.2784 |
| QC10U-pH 9b | 7.7918 | 8.2907 |
| QC10U-pH 9.25a | 7.8441 | 8.3455 |
| QC10U-pH 9.25 | 7.7647 | 8.2696 |
| QC10U-pH 9.5a | 7.8145 | 8.3157 |
| QC10U-pH 9.5b | 7.8219 | 8.3205 |

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BKG IPA DATA PROCESSED - 27-Jan-99 10:42
Bkg (0-18.6 keV) = 22.30 cpm
Bkg (0-156 keV) = 31.62 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 384.06
H3 E²/B (1-18.6 keV) = 187.83

QC10U
Capped
Desorption

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 28-Jan-99 13:23
C14 Eff (0-156 keV) = 96.25 %
C14 CHI SQUARE IPA DATA PROCESSED - 28-Jan-99 13:34
C14 Chi Square = 14.31
H3 IPA DATA PROCESSED - 28-Jan-99 13:35
H3 Eff (0-18.6 keV) = 65.04 %
H3 CHI SQUARE IPA DATA PROCESSED - 28-Jan-99 13:45
H3 Chi Square = 16.29
BKG IPA DATA PROCESSED - 28-Jan-99 14:46
Bkg (0-18.6 keV) = 21.95 cpm
Bkg (0-156 keV) = 31.55 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 387.54
H3 E²/B (1-18.6 keV) = 191.79

29 Jan 99 07:35 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page
User : A

Time: 999.99
Data Mode: CPM
Background Subtract: 1st Vial
Nuclide: MANUAL

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.02 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.52 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.48 |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

Container Description, Capped QCI0U

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|----|------|------------|--------------------------|--------------------|--|
| P# | 1 | 999.99 | 20.02 1.41 3.524 3.37 | 29.48 1.16 144.10 | |
| 23 | 2 | 7.80 | 11.77 34.38 566.732 3.02 | 579.62 3.05 597.62 | B Std. ↑ * QCI0 Cap Cont desor |
| 23 | 3 | 7.75 | 3.08 112.6 570.799 3.02 | 575.94 3.07 608.19 | |
| 23 | 4 | 9.63 | 3.03 102.5 458.158 3.02 | 462.52 3.09 599.84 | |
| 23 | 5 | 9.45 | 0.00 0.00 467.693 3.02 | 467.87 3.10 608.40 | |
| 23 | 6 | 11.16 | 5.25 57.59 394.953 3.03 | 399.64 3.10 605.39 | |
| 23 | 7 | 11.39 | 3.77 76.99 386.731 3.03 | 391.06 3.11 599.31 | |
| 23 | 8 | 8.75 | 1.12 278.1 505.047 3.02 | 507.09 3.09 573.75 | |
| 23 | 9 | 17.72 | 0.52 416.4 247.322 3.04 | 248.28 3.19 608.17 | |
| 23 | 10 | 21.62 | 2.18 93.81 202.119 3.05 | 205.07 3.22 603.10 | |
| 23 | 11 | 20.92 | 0.44 455.4 208.952 3.05 | 209.67 3.23 613.53 | |
| 23 | 12 | 31.43 | 0.69 238.0 137.901 3.08 | 139.27 3.34 604.11 | |
| 23 | 13 | 30.73 | 1.13 148.7 141.090 3.08 | 142.53 3.33 608.31 | |
| 23 | 14 | 41.59 | 0.66 218.6 103.329 3.10 | 104.52 3.45 610.39 | |
| 23 | 15 | 41.94 | 1.56 93.84 102.461 3.11 | 104.31 3.44 602.02 | |
| 23 | 16 | 48.66 | 1.83 75.04 87.865 3.12 | 89.90 3.51 604.25 | |
| 23 | 17 | 48.26 | 0.00 0.00 88.561 3.12 | 88.61 3.55 612.30 | |
| 23 | 18 | 55.27 | 0.91 138.3 76.899 3.14 | 78.33 3.59 603.61 | |
| 23 | 19 | 62.05 | 2.17 56.60 68.112 3.16 | 70.16 3.65 586.48 | |
| 23 | 20 | 50.78 | 1.84 73.00 84.010 3.13 | 85.72 3.54 599.28 | |
| 23 | 21 | 51.67 | 0.26 489.6 82.483 3.13 | 83.12 3.58 608.95 | |
| 23 | 22 | 62.29 | 0.69 172.2 67.820 3.16 | 68.77 3.69 608.61 | |
| 23 | 23 | 63.92 | 0.18 655.3 66.016 3.17 | 66.75 3.71 613.12 | |
| 23 | 24 | 40.99 | 0.00 0.00 104.893 3.10 | 103.67 3.49 619.30 | |
| 23 | 25 | 41.36 | 0.00 0.00 103.923 3.10 | 103.30 3.48 612.58 | |
| 23 | 26 | 57.92 | 0.00 0.00 73.220 3.15 | 73.38 3.66 621.21 | |
| 23 | 27 | 60.66 | 0.00 0.00 69.737 3.16 | 69.41 3.71 619.58 | |
| 23 | 28 | 56.52 | 0.45 274.5 75.103 3.14 | 76.20 3.62 608.13 | |
| 23 | 29 | 57.44 | 0.85 145.1 73.844 3.15 | 74.82 3.63 608.61 | |
| 23 | 30 | 60.64 | 0.12 1032 69.777 3.16 | 70.19 3.69 615.64 | |
| 23 | 31 | 61.84 | 0.03 3720 68.339 3.16 | 68.90 3.69 614.18 | |
| 23 | 32 | 58.53 | 0.79 155.2 72.420 3.15 | 73.06 3.65 611.50 | |
| 23 | 33 | 58.63 | 0.00 0.00 72.273 3.15 | 72.14 3.68 621.03 | |
| 23 | 34 | 64.89 | 0.14 839.5 64.961 3.17 | 65.31 3.74 614.11 | |
| 23 | 35 | 64.99 | 0.28 418.9 64.871 3.17 | 64.93 3.75 618.86 | |
| 23 | 36 | 58.63 | 0.17 693.0 72.273 3.15 | 73.06 3.65 618.47 | |
| 23 | 37 | 60.16 | 0.00 0.00 70.396 3.15 | 69.69 3.72 626.01 | |
| 23 | 38 | 46.75 | 0.00 0.00 91.578 3.12 | 90.75 3.55 629.23 | |
| 23 | 39 | 46.96 | 0.55 245.9 91.110 3.12 | 92.90 3.50 612.61 | |
| 23 | 40 | 39.88 | 0.00 0.00 107.935 3.10 | 107.43 3.46 624.56 | |
| 23 | 41 | 40.07 | 0.84 174.4 107.382 3.10 | 108.13 3.44 611.39 | |

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ALPHA/BETA - 1.02

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Protocol #:23

U-233 3% 2 sigma

User

| | | | | | |
|-----|--|------------|--------------|---------------|---------------------|
| 23 | 42 | 49.66 | 0.00 0.00 | 85.964 3.13 | 85.92 3.57 620.91 |
| 23 | 43 | 49.54 | 1.03 129.1 | 86.181 3.13 | 87.47 3.53 612.31 |
| 23 | 44 | 47.20 | 0.00 0.00 | 90.629 3.12 | 90.98 3.53 611.86 |
| 23 | 45 | 46.60 | 0.00 0.00 | 91.862 3.12 | 92.41 3.52 610.54 |
| 23 | 46 | 91.04 | 0.21 462.9 | 45.290 3.24 | 46.27 4.01 605.02 |
| 23 | 47 | 89.98 | 0.75 133.4 | 45.865 3.24 | 46.91 4.00 605.11 |
| 23 | 48 | 112.33 | 0.00 0.00 | 36.038 3.31 | 36.03 4.34 612.36 |
| 23 | 49 | 115.94 | 0.34 263.9 | 34.806 3.32 | 35.43 4.33 604.74 |
| 23 | 50 | 131.10 | 0.02 4636 | 30.404 3.37 | 30.76 4.55 609.58 |
| 23 | 51 | 128.79 | 0.00 0.00 | 30.990 3.36 | 31.12 4.54 611.82 |
| 23 | 52 | 59.25 | 0.62 195.4 | 71.497 3.15 | 72.36 3.65 601.31 |
| 23 | 53 | 59.09 | 0.63 194.1 | 71.700 3.15 | 72.31 3.66 606.59 |
| 231 | MISSING TUBE(S) PP tubes desorption, QCI0U, Capped Vials | | | | |
| 55 | 9.44 | 1.80 169.5 | 467.768 3.02 | 469.57 3.10 | 607.07 |
| 23 | 56 | 9.41 | 2.19 140.9 | 469.165 3.02 | 473.07 3.09 606.39 |
| 23 | 57 | 7.32 | 2.66 132.9 | 604.126 3.02 | 606.17 3.08 607.85 |
| 23 | 58 | 6.87 | 4.43 85.34 | 643.492 3.02 | 649.12 3.06 603.81 |
| 23 | 59 | 4.25 | 2.57 179.9 | 1042.594 3.01 | 1046.99 3.04 599.52 |
| 23 | 60 | 4.31 | 4.34 109.7 | 1028.727 3.01 | 1033.86 3.04 602.45 |
| 23 | 61 | 4.03 | 2.56 185.2 | 1102.431 3.01 | 1105.26 3.04 618.62 |
| 23 | 62 | 3.98 | 3.10 156.0 | 1113.059 3.01 | 1116.00 3.04 605.54 |
| 23 | 63 | 3.21 | 4.28 128.8 | 1384.015 3.00 | 1389.52 3.03 601.38 |
| 23 | 64 | 3.32 | 9.50 62.86 | 1335.030 3.01 | 1346.12 3.02 604.51 |
| 23 | 65 | 2.64 | 0.06 9940 | 1685.491 3.00 | 1685.67 3.02 603.04 |
| 23 | 66 | 2.64 | 8.01 81.44 | 1680.946 3.01 | 1690.59 3.02 604.54 |
| 23 | 67 | 3.22 | 6.38 89.90 | 1378.774 3.01 | 1385.11 3.03 603.54 |
| 23 | 68 | 3.22 | 4.51 122.5 | 1379.706 3.00 | 1385.11 3.03 605.40 |
| 23 | 69 | 2.41 | 8.61 80.13 | 1845.024 3.00 | 1855.17 3.01 598.50 |
| 23 | 70 | 2.38 | 3.51 179.4 | 1864.543 3.01 | 1868.00 3.02 606.11 |
| 23 | 71 | 2.54 | 9.11 74.39 | 1751.988 3.00 | 1762.25 3.01 605.26 |
| 23 | 72 | 2.53 | 5.28 120.0 | 1760.112 3.00 | 1767.75 3.02 602.52 |
| 23 | 73 | 2.55 | 5.86 108.8 | 1741.574 3.00 | 1748.95 3.02 600.78 |
| 23 | 74 | 2.54 | 15.81 47.55 | 1746.082 3.01 | 1765.01 3.01 601.48 |
| 23 | 75 | 2.82 | 7.64 82.08 | 1576.263 3.00 | 1588.25 3.02 599.11 |
| 23 | 76 | 2.59 | 2.76 215.2 | 1716.939 3.00 | 1725.34 3.02 608.29 |
| 23 | 77 | 2.48 | 2.56 236.0 | 1789.218 3.01 | 1792.70 3.02 604.62 |
| 23 | 78 | 2.47 | 2.65 228.7 | 1804.978 3.00 | 1810.60 3.01 599.52 |
| 23 | 79 | 2.66 | 0.28 1972 | 1669.032 3.00 | 1671.27 3.03 606.68 |
| 23 | 80 | 2.75 | 3.98 148.6 | 1614.294 3.01 | 1617.06 3.03 598.44 |
| 23 | 81 | 2.14 | 8.02 90.37 | 2079.654 3.00 | 2089.68 3.01 606.30 |
| 23 | 82 | 2.12 | 6.39 110.5 | 2094.589 3.00 | 2103.07 3.02 602.00 |
| 23 | 83 | 2.51 | 4.68 134.2 | 1766.994 3.01 | 1772.91 3.02 605.76 |
| 23 | 84 | 2.50 | 0.78 740.7 | 1777.676 3.00 | 1778.52 3.02 609.95 |
| 23 | 85 | 2.44 | 7.03 94.82 | 1820.246 3.00 | 1827.90 3.02 606.03 |
| 23 | 86 | 2.44 | 10.72 66.29 | 1818.607 3.01 | 1828.72 3.02 606.33 |
| 23 | 87 | 3.02 | 3.82 147.3 | 1469.986 3.01 | 1475.15 3.03 610.89 |

QC
Cap
PP
des

| | | | | | | | | | |
|----|----|------|------|-------|----------|------|---------|------|--------|
| 23 | 88 | 2.96 | 2.95 | 188.9 | 1502.557 | 3.00 | 1507.01 | 3.02 | 602.23 |
| 23 | 89 | 2.50 | 7.58 | 87.75 | 1777.276 | 3.00 | 1789.32 | 3.01 | 603.95 |
| 23 | 90 | 2.47 | 5.89 | 110.1 | 1800.929 | 3.00 | 1808.98 | 3.02 | 603.21 |
| 23 | 91 | 2.43 | 1.38 | 430.9 | 1825.282 | 3.01 | 1826.90 | 3.03 | 601.28 |
| 23 | 92 | 2.49 | 6.08 | 106.5 | 1785.633 | 3.00 | 1791.80 | 3.02 | 604.83 |
| 23 | 93 | 2.94 | 7.19 | 84.71 | 1511.442 | 3.00 | 1517.12 | 3.02 | 600.04 |
| 23 | 94 | 3.00 | 5.31 | 109.5 | 1481.476 | 3.00 | 1486.19 | 3.02 | 603.69 |
| 23 | 95 | 2.47 | 7.92 | 85.05 | 1802.144 | 3.00 | 1813.03 | 3.01 | 608.29 |
| 23 | 96 | 2.45 | 2.02 | 297.2 | 1817.700 | 3.00 | 1820.72 | 3.02 | 599.25 |
| 23 | 97 | 2.61 | 4.12 | 147.9 | 1702.223 | 3.00 | 1707.68 | 3.02 | 606.26 |
| 23 | 98 | 2.53 | 5.28 | 120.0 | 1757.346 | 3.00 | 1762.61 | 3.02 | 600.52 |

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ALPHA/BETA - 1.02

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Protocol #:23

U-233 3% 2 sigma

User: AJ

| | | | | | | | | | |
|----|-----|------|-------|-------|----------|------|---------|------|--------|
| 23 | 99 | 2.03 | 8.06 | 92.37 | 2196.969 | 3.00 | 2208.45 | 3.01 | 595.81 |
| 23 | 100 | 1.89 | 9.61 | 82.46 | 2349.386 | 3.00 | 2358.88 | 3.01 | 598.66 |
| 23 | 101 | 2.06 | 4.74 | 146.5 | 2157.641 | 3.00 | 2161.30 | 3.02 | 602.86 |
| 23 | 102 | 2.07 | 20.56 | 43.09 | 2148.167 | 3.00 | 2170.52 | 3.00 | 604.58 |
| 23 | 103 | 2.59 | 3.53 | 170.9 | 1717.712 | 3.00 | 1721.48 | 3.02 | 606.75 |
| 23 | 104 | 2.56 | 0.00 | 0.00 | 1734.367 | 3.00 | 1730.28 | 3.03 | 605.72 |
| 23 | 105 | 8.16 | 0.08 | 4049 | 541.206 | 3.02 | 540.74 | 3.09 | 604.35 |
| 23 | 106 | 8.47 | 1.35 | 236.4 | 521.151 | 3.02 | 522.94 | 3.09 | 600.35 |
| 23 | 107 | 8.57 | 3.55 | 93.76 | 515.379 | 3.02 | 521.74 | 3.08 | 597.04 |
| 23 | 108 | 8.61 | 2.28 | 141.7 | 512.620 | 3.02 | 516.63 | 3.08 | 596.31 |
| 23 | | | | | | | | | |

1/26/99
AJ

stock U-soln recounted for QC10U uncapped expts.

| wt. of original Container | wt. of container + U soln. | wt. of container + U-soln + Acid |
|------------------------------|-------------------------------|-------------------------------------|
| 19.6480 | 54.6673 | 57.3693 |

PH of starting solution = 4.16

| | wt of vials | wt of vials + sample |
|------------|-------------|----------------------|
| QC10U - Ia | 7.7576 | 8.2599 |
| QC10U - Ib | 7.7941 | 8.2949 |

UNCAPPED, Qtz = 1.0 g, Clinop = 0.700 g.

1/28/99
AJ

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma U = 50$ ppb (2.14×10^{-7})

- equilibrium with atmospheric $CO_2(g)$; $pCO_2 = 10^{-3.5}$
- pH range 4.0-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 700mg (0.70 g)
- M/V = 28.57 g/L

Equipment: Orion 920A pH meter and combination pH electrode
Gyratory shaker
Fisher Marathon 21K centrifuge
Mettler PM4600 and AE240 balances
Packard 2500 TR/AB LSC
Eppendorf micropipettors and tips
Oxford pipettes and tips
Repipettor for transfer of scintillation cocktail

Supplies: ≤ 325 mesh prepared Qtz & 100/200 mesh Clinoptilolite in NaCl
50 ppb ^{233}U stock solution
polycarbonate centrifuge tubes with caps (50 ml capacity)
polypropylene centrifuge tubes with caps (50 ml capacity)
pH buffer solutions
Ultima-Gold liquid scintillation cocktail
7 ml scintillation vials
reagent grade NaOH (lot 936883) AT
concentrated HNO_3 (lot 983546)
 HNO_3 and NaOH stock solutions at various concentrations prepared in degassed water.
0.1M $NaNO_3$ matrix prepared with degassed water.
weighing paper/boats
deionized ultrapure water, DH_2O
Teflon beaker

Note: Ultrapure water was degassed by boiling it in a flask and cooled with mouth covered with rubber stopper.

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M degassed $NaNO_3$ stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.
- Label 26 polycarbonate centrifuge tubes (50 ml capacity) QCU-pHi (where i is the approximate pH of each solution, see Table QCU).
 - Label 1 polycarbonate centrifuge tube QC20.0U-IU.
 - Weigh and record the weight of each container.

- About 1L of ^{233}U solution will be required for this experiment.
- Add ~35 g of the 50 ppb ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.*
- Record weight of each container.

C. Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the QC20.0-U-IU container.
- Record weight of each sample container after addition of quartz.

D. Add clinoptilolite to sample containers.

- Add 0.70 g of clinoptilolite to each sample container. **Do not** add clinoptilolite to the QC20.0-U-IU container.
- Record weight of each sample container after addition of clinoptilolite.

E. Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaOH to each experimental container as listed in Table QC20.0U. Take care to add HNO_3 or NaOH in the concentrations specified in Table CQ20U.
- For sample QC20.0U-IU (as listed in Table QC20.0U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

F. Sample QC20.0U-IU for initial [U].

- Label two 7 ml LSC vials as QC20.0U-IUa and QC20.0U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from QC20.0U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container QC20.0U-IU.

G. Wait for equilibrium.

- Place caps on sample containers and tighten the caps.
- Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate for at least 10 days.

H. Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., QC20.0U-pH3a and QC20.0U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.

- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

I.

Transfer mixture of quartz and clinoptilolite for desorption.

- Record weight of PC sample containers. *Note: this step can be skipped if the mixture transfer is done immediately after the last step in section H.*
- Centrifuge polycarbonate containers containing experimental solutions at 10,000 rpm for 15 min to remove fine particles from suspension.
- For each PC sample container, label two corresponding polypropylene (PP) test tubes A and B (50 ml capacity).
- Record weight for the set of PP tubes A.
- Add 3.0 ml of 0.1M HNO_3 to 50 ml PP tubes labeled as B-QC20.0Ud-pH.
- Pour supernatant from PC containers into PP tubes with acid and labeled as B-QC20.0Ud-pH leaving behind about 7 to 8ml of solution in PC container with the solid mixture.
- Record the weight of PC containers containing the solid mixture and some solution.
- Stir the solid mixture in solution vigorously and immediately transfer all the solid and solution to the corresponding PP tubes marked as A-QC20.0Ud-pH.
- Record the weight of PP tubes A-QC20.0Ud-pH.
- Transfer supernatant with HNO_3 acid from the PP tubes B-QC20.0Ud-pH back to original PC containers.
- Record weights of PC sample containers.
- Add approximately 3 ml of 0.1 M HNO_3 to PP tubes containing solid mixture, A-QC20.0Ud-pH.
- Record weights of PP tubes A-QC20.0Ud-pH and PC sample containers.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

J.

Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QC20.0Ud-pH3a and QC20.0Ud-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquot of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table QC20.0U: Estimated solution pH and volumes of HNO3 or NaHCO3 solutions needed for adjustment of pH in 0.1 m NaNO3 solutions with 50 ppb U in contact with qtz/clinop (35 ml)

| Estimated solution pH | Volume of HNO3 added, ml | Molarity of HNO3 to use |
|-----------------------|----------------------------|---------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO3 added, ml | Molarity of NaHCO3 to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |
| QC20U-IU | 0.100 | 50% HNO3 |

1/29/99

AJ

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1/29/99

| Sample Name | Original Container wt., g |
|---------------|---------------------------|
| QC20U-pH 3.0 | 22.1799 |
| QC20U-pH 3.5 | 22.2045 |
| QC20U-pH 4.0 | 22.3451 |
| QC20U-pH 4.25 | 22.2656 |
| QC20U-pH 4.5 | 22.1422 |
| QC20U-pH 4.75 | 22.3608 |
| QC20U-pH 5.0 | 22.2403 |
| QC20U-pH 5.25 | 22.1384 |
| QC20U-pH 5.5 | 22.2506 |
| QC20U-pH 5.75 | 22.5185 |
| QC20U-pH 6.0 | 22.3267 |
| QC20U-pH 6.25 | 22.5800 |
| QC20U-pH 6.5 | 22.2974 |
| QC20U-pH 6.75 | 22.2558 |
| QC20U-pH 7.0 | 22.1861 |
| QC20U-pH 7.25 | 22.1911 |
| QC20U-pH 7.5 | 22.1452 |
| QC20U-pH 7.75 | 22.2309 |
| QC20U-pH 8.0 | 22.3018 |
| QC20U-pH 8.25 | 22.2579 |
| QC20U-pH 8.5 | 22.5666 |
| QC20U-pH 8.75 | 22.5728 |
| QC20U-pH 9.0 | 22.3169 |
| QC20U-pH 9.25 | 22.1572 |
| QC20U-pH 9.5 | 22.6105 |
| QC20U-IU | 22.2603 |

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1/29/99

| Sample Name | Container wt. + U soln., g |
|---------------|----------------------------|
| QC20U-pH 3.0 | 57.1809 |
| QC20U-pH 3.5 | 57.2135 |
| QC20U-pH 4.0 | 57.3418 |
| QC20U-pH 4.25 | 57.2782 |
| QC20U-pH 4.5 | 57.1402 |
| QC20U-pH 4.75 | 57.3554 |
| QC20U-pH 5.0 | 57.2654 |
| QC20U-pH 5.25 | 57.1329 |
| QC20U-pH 5.5 | 57.2592 |
| QC20U-pH 5.75 | 57.5194 |
| QC20U-pH 6.0 | 57.3422 |
| QC20U-pH 6.25 | 57.5858 |
| QC20U-pH 6.5 | 57.3012 |
| QC20U-pH 6.75 | 57.2558 |
| QC20U-pH 7.0 | 57.1861 |
| QC20U-pH 7.25 | 57.1969 |
| QC20U-pH 7.5 | 57.1483 |
| QC20U-pH 7.75 | 57.2423 |
| QC20U-pH 8.0 | 57.3022 |
| QC20U-pH 8.25 | 57.2586 |
| QC20U-pH 8.5 | 57.5659 |
| QC20U-pH 8.75 | 57.5686 |
| QC20U-pH 9.0 | 57.3288 |
| QC20U-pH 9.25 | 57.1686 |
| QC20U-pH 9.5 | 57.6046 |
| | |
| QC20U-IU | 57.2523 |

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1/29/99

| Sample Name | Container wt. + U soln. + quartz, g |
|---------------|-------------------------------------|
| QC20U-pH 3.0 | 58.1781 |
| QC20U-pH 3.5 | 58.2146 |
| QC20U-pH 4.0 | 58.3428 |
| QC20U-pH 4.25 | 58.2783 |
| QC20U-pH 4.5 | 58.1415 |
| QC20U-pH 4.75 | 58.3553 |
| QC20U-pH 5.0 | 58.2660 |
| QC20U-pH 5.25 | 58.1315 |
| QC20U-pH 5.5 | 58.2451 |
| QC20U-pH 5.75 | 58.5171 |
| QC20U-pH 6.0 | 58.3420 |
| QC20U-pH 6.25 | 58.5857 |
| QC20U-pH 6.5 | 58.3014 |
| QC20U-pH 6.75 | 58.2581 |
| QC20U-pH 7.0 | 58.1850 |
| QC20U-pH 7.25 | 58.1973 |
| QC20U-pH 7.5 | 58.1483 |
| QC20U-pH 7.75 | 58.2407 |
| QC20U-pH 8.0 | 58.3019 |
| QC20U-pH 8.25 | 58.2580 |
| QC20U-pH 8.5 | 58.5674 |
| QC20U-pH 8.75 | 58.5691 |
| QC20U-pH 9.0 | 58.3288 |
| QC20U-pH 9.25 | 58.1717 |
| QC20U-pH 9.5 | 58.6042 |

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2/2/99

| Sample Name | Container wt. + U soln. + quartz + clinop, g |
|---------------|--|
| QC20U-pH 3.0 | 58.8489 |
| QC20U-pH 3.5 | 58.8669 |
| QC20U-pH 4.0 | 59.0073 |
| QC20U-pH 4.25 | 58.9372 |
| QC20U-pH 4.5 | 58.8042 |
| QC20U-pH 4.75 | 59.0169 |
| QC20U-pH 5.0 | 58.9379 |
| QC20U-pH 5.25 | 58.8068 |
| QC20U-pH 5.5 | 58.9199 |
| QC20U-pH 5.75 | 59.1655 |
| QC20U-pH 6.0 | 59.0138 |
| QC20U-pH 6.25 | 59.2514 |
| QC20U-pH 6.5 | 58.9760 |
| QC20U-pH 6.75 | 58.9317 |
| QC20U-pH 7.0 | 58.8596 |
| QC20U-pH 7.25 | 58.8734 |
| QC20U-pH 7.5 | 58.8136 |
| QC20U-pH 7.75 | 58.9077 |
| QC20U-pH 8.0 | 58.9649 |
| QC20U-pH 8.25 | 58.9291 |
| QC20U-pH 8.5 | 59.2289 |
| QC20U-pH 8.75 | 59.2438 |
| QC20U-pH 9.0 | 58.9978 |
| QC20U-pH 9.25 | 58.8387 |
| QC20U-pH 9.5 | 59.2655 |

Actual Volumes added for PH adjust

Table QC20U: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with quartz and clinoptilolite (35ml).

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 3 | 0.394 0.300 + 0.075 + 0.020 | 0.1 |
| 3.25 | 0.209 0.200 + 0.010 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 0.050 + 0.020 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 0.075 | 0.005 |
| 4.5 | 0.292 0.250 | 0.005 |
| 4.75 | 0.205 0.150 | 0.01 |
| 5 | 0.239 0.200 | 0.01 |
| 5.25 | 0.260 0.200 + 0.025 | 0.01 |
| 5.5 | 0.274 0.200 + 0.040 | 0.01 |
| 5.75 | 0.286 0.250 | 0.01 |
| 6 | 0.300 0.275 | 0.01 |
| 6.25 | 0.322 0.300 | 0.01 |
| 6.5 | 0.071 0.065 | 0.05 |
| 6.75 | 0.084 0.095 | 0.05 |
| 7 | 0.106 0.105 | 0.05 |
| 7.25 | 0.145 0.100 + 0.020 + 0.025 | 0.05 |
| 7.5 | 0.214 0.100 + 0.075 + 0.020 + 0.020 | 0.05 |
| 7.75 | 0.169 0.175 | 0.1 |
| 8 | 0.282 0.285 | 0.1 |
| 8.25 | 0.097 0.100 | 0.5 |
| 8.5 | 0.174 0.175 | 0.5 |
| 8.75 | 0.160 0.100 + 0.050 + 0.010 | 1 |
| 9 | 0.305 0.310 | 1 |
| 9.25 | 0.825 | 1 |
| 9.5 | 1.220 | 1 |
| QC20U-IU | 0.100 | 50% HNO ₃ |

UNCAPPED

2/2/99

| Sample Name | Container Wt. After additions of HNO ₃ and NaHCO ₃ , g |
|---------------|--|
| QC20U-pH 3.0 | 59.2335 |
| QC20U-pH 3.5 | 59.0733 |
| QC20U-pH 4.0 | 59.1498 |
| QC20U-pH 4.25 | 59.0076 |
| QC20U-pH 4.5 | 59.0504 |
| QC20U-pH 4.75 | 59.1630 |
| QC20U-pH 5.0 | 59.1349 |
| QC20U-pH 5.25 | 59.0293 |
| QC20U-pH 5.5 | 59.1552 |
| QC20U-pH 5.75 | 59.4115 |
| QC20U-pH 6.0 | 59.2865 |
| QC20U-pH 6.25 | 59.5492 |
| QC20U-pH 6.5 | 59.0380 |
| QC20U-pH 6.75 | 59.0245 |
| QC20U-pH 7.0 | 59.9627 |
| QC20U-pH 7.25 | 59.0159 |
| QC20U-pH 7.5 | 59.0255 |
| QC20U-pH 7.75 | 59.0783 |
| QC20U-pH 8.0 | 59.2482 |
| QC20U-pH 8.25 | 59.0290 |
| QC20U-pH 8.5 | 59.4061 |
| QC20U-pH 8.75 | 59.4121 |
| QC20U-pH 9.0 | 59.3223 |
| QC20U-pH 9.25 | 59.7157 |
| QC20U-pH 9.5 | 60.5643 |
| QC20U-IU | 57.3522 |

18 Feb 99 11:38

ALPHA/BETA - 1.02

Page

Protocol #:23

U-233 3% 2 sigma

User : F

H3 Eff (0-18.6 keV) = 65.10 %

H3 CHI SQUARE IPA DATA PROCESSED - 18-Feb-99 11:48

H3 Chi Square = 31.04

BKG IPA DATA PROCESSED - 18-Feb-99 12:49

Bkg (0-18.6 keV) = 21.12 cpm

Bkg (0-156 keV) = 30.72 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 394.48H3 E²/B (1-18.6 keV) = 200.12

19 Feb 99 05:38

ALPHA/BETA - 1.02

Page

Protocol #:23

U-233 3% 2 sigma

User : F

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 2S% | BKG |
| Region A: | 0.0 - 100 | 0 | 0.3 | 19.90 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.46 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.24 | |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

QC20U
uncapped
Sorption

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|--------|------------|--------------|------------|---------------|
| P# 1 | 999.99 | 19.90 1.42 | 3.464 3.40 | 29.24 | 1.17 143.08 B |
| 23 2 | 8.06 | 1.56 209.9 | 548.645 3.02 | 550.41 | 3.08 608.96 |
| 23 3 | 8.10 | 1.58 207.1 | 545.919 3.02 | 548.04 | 3.08 599.18 |
| 23 4 | 8.05 | 0.00 0.00 | 548.834 3.02 | 548.89 | 3.09 607.15 |
| 23 5 | 8.18 | 2.59 128.5 | 539.934 3.02 | 542.03 | 3.08 606.26 |
| 23 6 | 8.13 | 1.38 236.0 | 544.014 3.02 | 546.16 | 3.08 609.82 |
| 23 7 | 8.42 | 0.05 6272 | 524.921 3.02 | 527.17 | 3.08 608.16 |
| 23 8 | 10.94 | 2.49 115.4 | 402.752 3.03 | 405.31 | 3.11 612.84 |
| 23 9 | 11.14 | 0.00 0.00 | 395.459 3.03 | 396.34 | 3.12 609.13 |
| 23 10 | 13.99 | 0.00 0.00 | 314.191 3.03 | 313.93 | 3.16 618.59 |
| 23 11 | 14.21 | 0.29 817.1 | 309.273 3.03 | 310.16 | 3.15 618.99 |
| 23 12 | 53.55 | 0.73 174.0 | 79.524 3.13 | 80.02 | 3.60 610.89 |
| 23 13 | 53.52 | 1.66 78.39 | 79.570 3.13 | 81.44 | 3.56 603.56 |
| 23 14 | 74.98 | 1.09 100.6 | 55.805 3.19 | 56.97 | 3.81 602.60 |
| 23 15 | 74.92 | 0.00 0.00 | 55.853 3.19 | 55.34 | 3.89 619.16 |
| 23 16 | 87.30 | 1.19 86.30 | 47.441 3.23 | 49.26 | 3.91 603.11 |
| 23 17 | 84.93 | 0.20 517.6 | 48.861 3.22 | 49.55 | 3.95 611.44 |
| 23 18 | 88.26 | 0.70 144.8 | 46.910 3.23 | 48.21 | 3.95 606.86 |
| 23 19 | 86.99 | 0.00 0.00 | 47.622 3.23 | 47.68 | 4.01 613.99 |
| 23 20 | 61.82 | 0.79 151.5 | 68.422 3.16 | 69.51 | 3.67 609.77 |

QC20U
Sorption

| | | | | | | | | | | |
|----|----|--------|-------|-------|---------|------|--------|------|--------|------------------|
| 23 | 21 | 61.80 | 0.86 | 139.2 | 68.445 | 3.16 | 69.72 | 3.66 | 606.65 | 2001 unlapped |
| 23 | 22 | 69.06 | 0.00 | 0.00 | 60.886 | 3.18 | 60.78 | 3.80 | 615.76 | |
| 23 | 23 | 68.77 | 0.19 | 579.6 | 61.201 | 3.17 | 61.30 | 3.78 | 604.08 | |
| 23 | 24 | 81.67 | 0.51 | 204.4 | 50.950 | 3.21 | 51.87 | 3.90 | 606.21 | |
| 23 | 25 | 81.15 | 0.01 | 9774 | 51.299 | 3.21 | 52.12 | 3.90 | 614.67 | |
| 23 | 26 | 84.94 | 0.59 | 172.2 | 48.867 | 3.22 | 49.66 | 3.94 | 604.68 | |
| 23 | 27 | 83.71 | 0.17 | 613.6 | 49.624 | 3.22 | 50.20 | 3.94 | 610.31 | |
| 23 | 28 | 74.30 | 0.45 | 242.7 | 56.388 | 3.19 | 57.34 | 3.81 | 609.01 | |
| 23 | 29 | 72.24 | 0.18 | 597.8 | 58.053 | 3.19 | 57.95 | 3.84 | 610.02 | |
| 23 | 30 | 77.22 | 0.00 | 0.00 | 54.086 | 3.20 | 53.75 | 3.91 | 623.91 | |
| 23 | 31 | 77.47 | 0.41 | 256.4 | 53.913 | 3.20 | 54.41 | 3.87 | 612.67 | |
| 23 | 32 | 75.13 | 0.34 | 314.8 | 55.687 | 3.19 | 56.42 | 3.83 | 605.72 | |
| 23 | 33 | 76.20 | 0.50 | 213.0 | 54.856 | 3.20 | 55.41 | 3.85 | 608.18 | |
| 23 | 34 | 107.41 | 10.29 | 10.66 | 37.910 | 3.29 | 47.88 | 3.61 | 453.42 | |
| 23 | 35 | 81.94 | 0.01 | 7426 | 50.771 | 3.21 | 50.56 | 3.96 | 612.98 | |
| 23 | 36 | 74.02 | 0.02 | 4509 | 56.574 | 3.19 | 57.00 | 3.83 | 613.15 | |
| 23 | 37 | 72.58 | 0.94 | 117.6 | 57.779 | 3.19 | 59.17 | 3.78 | 603.83 | |
| 23 | 38 | 59.99 | 0.18 | 649.2 | 70.632 | 3.15 | 71.07 | 3.67 | 611.52 | |
| 23 | 39 | 59.44 | 0.64 | 189.4 | 71.300 | 3.15 | 71.40 | 3.68 | 606.77 | |
| 23 | 40 | 40.44 | 0.40 | 362.6 | 106.452 | 3.10 | 107.18 | 3.44 | 604.64 | |
| 23 | 41 | 39.71 | 0.27 | 542.1 | 108.473 | 3.10 | 109.34 | 3.43 | 610.98 | |

U Feb 99 20:17
Protocol #:23

ALPHA/BETA - 1.02
U-233 3% 2 sigma

Page 1
User : Al

| | | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|-------------------------------|
| 23 | 42 | 29.56 | 1.38 | 125.1 | 146.908 | 3.07 | 148.56 | 3.31 | 607.01 | 2020V unlapped sorption |
| 23 | 43 | 28.76 | 0.00 | 0.00 | 151.160 | 3.07 | 150.90 | 3.32 | 614.68 | |
| 23 | 44 | 20.61 | 0.91 | 222.6 | 212.208 | 3.05 | 213.07 | 3.22 | 609.34 | |
| 23 | 45 | 19.79 | 0.31 | 660.4 | 221.195 | 3.05 | 221.39 | 3.22 | 616.20 | |
| 23 | 46 | 13.37 | 1.86 | 137.9 | 328.997 | 3.03 | 331.04 | 3.14 | 600.69 | |
| 23 | 47 | 13.34 | 0.86 | 291.5 | 330.119 | 3.03 | 331.48 | 3.14 | 610.83 | |
| 23 | 48 | 10.11 | 1.26 | 230.0 | 436.398 | 3.02 | 439.00 | 3.10 | 613.86 | |
| 23 | 49 | 9.78 | 0.00 | 0.00 | 451.035 | 3.02 | 452.86 | 3.10 | 614.67 | |
| 23 | 50 | 8.55 | 2.79 | 117.3 | 516.419 | 3.02 | 519.18 | 3.09 | 611.27 | |
| 23 | 51 | 8.69 | 0.93 | 336.0 | 508.389 | 3.02 | 509.08 | 3.09 | 613.09 | |
| 23 | 52 | 8.51 | 2.89 | 113.5 | 519.333 | 3.02 | 522.34 | 3.08 | 610.20 | |
| 23 | 53 | 8.45 | 3.41 | 97.76 | 522.927 | 3.02 | 526.97 | 3.08 | 610.12 | |
| 23 | 54 | 8.65 | 2.99 | 109.3 | 510.293 | 3.02 | 514.57 | 3.08 | 615.98 | |
| 23 | 55 | 8.42 | 1.83 | 176.2 | 525.039 | 3.02 | 526.81 | 3.09 | 618.02 | |
| 23 | 56 | 8.66 | 0.00 | 0.00 | 510.277 | 3.02 | 510.02 | 3.10 | 628.65 | |
| 23 | 57 | 8.69 | 0.23 | 1302 | 508.274 | 3.02 | 508.27 | 3.10 | 625.53 | |

21 Feb 99 04:59 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Pa
User :

C14 IPA DATA PROCESSED - 21-Feb-99 04:59
C14 Eff (0-156 keV) = 96.61 %
C14 CHI SQUARE IPA DATA PROCESSED - 21-Feb-99 05:09
C14 Chi Square = 18.56
H3 IPA DATA PROCESSED - 21-Feb-99 05:11
H3 Eff (0-18.6 keV) = 65.56 %
H3 CHI SQUARE IPA DATA PROCESSED - 21-Feb-99 05:21
H3 Chi Square = 14.62
BKG IPA DATA PROCESSED - 21-Feb-99 06:21
Bkg (0-18.6 keV) = 21.43 cpm
Bkg (0-156 keV) = 30.90 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 397.20
H3 E²/B (1-18.6 keV) = 198.97

Unlapped Sorption 2/11/99

| Sample Name | wt. of vial,g | wt. Of vial + sample |
|----------------|---------------|----------------------|
| QC20U-pH 3a | 7.7628 | 8.2582 |
| QC20U-pH 3b | 7.8019 | 8.3004 |
| QC20U-pH 3.5a | 7.8327 | 8.3303 |
| QC20U-pH 3.5b | 7.8903 | 8.3846 |
| QC20U-pH 4a | 7.837 | 8.3300 |
| QC20U-pH 4b | 7.8545 | 8.3384 |
| QC20U-pH 4.25a | 7.8277 | 8.3212 |
| QC20U-pH 4.25b | 7.8298 | 8.3186 |
| QC20U-pH 4.5a | 7.8404 | 8.3167 |
| QC20U-pH 4.5b | 7.8539 | 8.3492 |
| QC20U-pH 4.75a | 7.8177 | 8.3105 |
| QC20U-pH 4.75b | 7.863 | 8.3538 |
| QC20U-pH 5a | 7.8199 | 8.319 |
| QC20U-pH 5b | 7.807 | 8.2979 |
| QC20U-pH 5.25a | 7.7695 | 8.2598 |
| QC20U-pH 5.25b | 7.8102 | 8.2961 |
| QC20U-pH 5.5a | 7.8573 | 8.3489 |
| QC20U-pH 5.5b | 7.8911 | 8.3833 |
| QC20U-pH 5.75a | 7.84 | 8.3127 |
| QC20U-pH 5.75b | 7.8248 | 8.3161 |
| QC20U-pH 6a | 7.7909 | 8.2829 |
| QC20U-pH 6b | 7.8295 | 8.3215 |
| QC20U-pH 6.25a | 7.77 | 8.2515 |
| QC20U-pH 6.25a | 7.8376 | 8.3073 |
| QC20U-pH 6.5a | 7.8545 | 8.3472 |
| QC20U-pH 6.5b | 7.8158 | 8.2971 |
| QC20U-pH 6.75a | 7.839 | 8.3235 |
| QC20U-pH 6.75b | 7.8111 | 8.3048 |
| QC20U-pH 7a | 7.869 | 8.3505 |
| QC20U-pH 7b | 7.8024 | 8.2948 |
| QC20U-pH 7.25a | 7.8416 | 8.3164 |
| QC20U-pH 7.25b | 7.7714 | 8.2626 |

| | | |
|----------------|--------|--------|
| QC20U-pH 7.5a | 7.8579 | 8.3318 |
| QC20U-pH 7.5b | 7.8721 | 8.3478 |
| QC20U-pH 7.75a | 7.7799 | 8.2722 |
| QC20U-pH 7.75b | 7.8305 | 8.3227 |
| QC20U-pH 8a | 7.8593 | 8.3521 |
| QC20U-pH 8b | 7.9129 | 8.3987 |
| QC20U-pH 8.25a | 7.7583 | 8.2487 |
| QC20U-pH 8.25b | 7.8536 | 8.3457 |
| QC20U-pH 8.5a | 7.8247 | 8.3166 |
| QC20U-pH 8.5b | 7.8294 | 8.3064 |
| QC20U-pH 8.75a | 7.8038 | 8.2795 |
| QC20U-pH 8.75b | 7.8108 | 8.3021 |
| QC20U-pH 9a | 7.8252 | 8.2989 |
| QC20U-pH 9b | 7.8408 | 8.3306 |
| QC20U-pH 9.25a | 7.8819 | 8.3703 |
| QC20U-pH 9.25 | 7.888 | 8.3789 |
| QC20U-pH 9.5a | 7.8466 | 8.3219 |
| QC20U-pH 9.5b | 7.823 | 8.2963 |
| QC1.0U-lua | 7.7876 | 8.2933 |
| QC1.0U-IUb | 7.812 | 8.3168 |
| QC1.0U-IUc | 7.812 | 8.3168 |
| QC1.0U-IUd | 7.7876 | 8.2933 |
| QC1.0U-IUe | 7.8399 | 8.3457 |
| QC1.0U-IUf | 7.8136 | 8.3189 |
| QC1.0U-IUg | 7.7792 | 8.2779 |
| QC1.0U-IUh | 7.7917 | 8.2897 |

UNCAPPED

| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|---------------|--|-------------|---|
| QC20U-pH 3.0 | 58.9597 | 3.64 | 57.9176 |
| QC20U-pH 3.5 | 58.8334 | 4.06 | 57.7767 |
| QC20U-pH 4.0 | 58.9038 | 5.3 | 57.8241 |
| QC20U-pH 4.25 | 58.6626 | 5.73 | 57.6393 |
| QC20U-pH 4.5 | 58.8128 | 6.02 | 57.7857 |
| QC20U-pH 4.75 | 58.6372 | 6.15 | 57.6121 |
| QC20U-pH 5.0 | 58.8768 | 5.47 | 57.8448 |
| QC20U-pH 5.25 | 58.7585 | 5.61 | 57.6945 |
| QC20U-pH 5.5 | 58.8617 | 6.55 | 57.7117 |
| QC20U-pH 5.75 | 59.0804 | 6.62 | 58.0551 |
| QC20U-pH 6.0 | 59.0198 | 5.69 | 57.999 |
| QC20U-pH 6.25 | 59.2660 | 6.74 | 58.2475 |
| QC20U-pH 6.5 | 58.7645 | 5.87 | 57.7414 |
| QC20U-pH 6.75 | 58.7672 | 6.53 | 57.7409 |
| QC20U-pH 7.0 | 58.6867 | 6.76 | 57.6635 |
| QC20U-pH 7.25 | 58.8011 | 7.03 | 57.7688 |
| QC20U-pH 7.5 | 58.7660 | 6.99 | 57.6467 |
| QC20U-pH 7.75 | 58.4453 | 7.49 | 57.4263 |
| QC20U-pH 8.0 | 58.9034 | 7.84 | 57.8862 |
| QC20U-pH 8.25 | 58.7673 | 8.01 | 57.7546 |
| QC20U-pH 8.5 | 59.1525 | 8.23 | 58.1378 |
| QC20U-pH 8.75 | 58.7750 | 8.48 | 57.7567 |
| QC20U-pH 9.0 | 59.0218 | 8.98 | 58.0056 |
| QC20U-pH 9.25 | 59.4197 | 9.26 | 58.4030 |
| QC20U-pH 9.5 | 60.1435 | 9.31 | 59.1210 |
| QC20U-IU | 57.2936 | | 56.2695 |

UNCAPPED

| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after transfer of qtz/clino, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer+acid added, g |
|---------------|-----------------------|--|---|--|---|
| QC20U-pH 3.0 | 13.7634 | 24.6253 | 24.6253 | 30.3292 | 52.7852 |
| QC20U-pH 3.5 | 13.6737 | 24.7739 | 24.7739 | 30.3774 | 52.5684 |
| QC20U-pH 4.0 | 13.7602 | 24.1501 | 24.1501 | 29.826 | 53.4697 |
| QC20U-pH 4.25 | 13.6685 | 22.92 | 22.92 | 28.5918 | 54.4274 |
| QC20U-pH 4.5 | 13.7018 | 24.7779 | 24.7779 | 30.3254 | 52.7219 |
| QC20U-pH 4.75 | 13.3834 | 24.8742 | 24.8742 | 30.9297 | 52.1173 |
| QC20U-pH 5.0 | 13.3304 | 24.6146 | 24.6146 | 30.5893 | 52.5634 |
| QC20U-pH 5.25 | 13.6852 | 25.4074 | 25.4074 | 30.9526 | 51.9997 |
| QC20U-pH 5.5 | 13.7934 | 27.8423 | 27.8423 | 33.4752 | 49.5728 |
| QC20U-pH 5.75 | 13.7957 | 27.0688 | 27.0688 | 32.8825 | 50.791 |
| QC20U-pH 6.0 | 13.3367 | 26.6817 | 26.6817 | 32.7158 | 50.6795 |
| QC20U-pH 6.25 | 13.6752 | 26.4121 | 26.4121 | 32.3821 | 51.5223 |
| QC20U-pH 6.5 | 13.4156 | 26.2204 | 26.2204 | 32.1623 | 50.9328 |
| QC20U-pH 6.75 | 13.6612 | 26.6875 | 26.6875 | 32.3207 | 50.6824 |
| QC20U-pH 7.0 | 13.6323 | 25.2362 | 25.2362 | 30.8409 | 52.0375 |
| QC20U-pH 7.25 | 13.4033 | 25.3688 | 25.3688 | 31.2114 | 51.8139 |
| QC20U-pH 7.5 | 13.6837 | 25.0925 | 25.0925 | 30.5772 | 52.2125 |
| QC20U-pH 7.75 | 13.6385 | 27.9584 | 27.9584 | 33.5829 | 49.1298 |
| QC20U-pH 8.0 | 13.6407 | 25.3139 | 25.3139 | 30.9843 | 52.1904 |
| QC20U-pH 8.25 | 13.7752 | 24.6581 | 24.6581 | 30.193 | 52.8849 |
| QC20U-pH 8.5 | 13.6962 | 23.7839 | 23.7839 | 29.6694 | 54.0111 |
| QC20U-pH 8.75 | 13.7015 | 24.8768 | 24.8768 | 30.7691 | 52.5476 |
| QC20U-pH 9.0 | 13.6762 | 25.8989 | 25.8989 | 31.5849 | 51.7199 |
| QC20U-pH 9.25 | 13.6963 | 24.7391 | 24.7391 | 30.239 | 53.3325 |
| QC20U-pH 9.5 | 13.6968 | 25.1003 | 25.1003 | 31.1298 | 53.4168 |

UNCAPPED

| | 3/1/99 | 3/2/99 | 3/1/99 | 3/2/99 |
|---------------|-------------------------------|------------------------------|---------------------------------|--------------------------------|
| Sample Name | Container wt. before sampling | Container wt. after sampling | wt. Of PP tubes before sampling | wt. Of PP tubes after sampling |
| QC20U-pH 3.0 | 52.6937 | 51.6748 | 24.6192 | 23.5883 |
| QC20U-pH 3.5 | 52.4735 | 51.4579 | 24.767 | 23.729 |
| QC20U-pH 4.0 | 53.3806 | 52.3639 | 23.5572 | 22.5403 |
| QC20U-pH 4.25 | 54.3468 | 53.329 | 22.4796 | 21.4619 |
| QC20U-pH 4.5 | 52.6166 | 51.5984 | 24.7347 | 23.7122 |
| QC20U-pH4.75 | 52.0065 | 50.9839 | 24.861 | 23.841 |
| QC20U-pH 5.0 | 52.4835 | 51.4599 | 24.5923 | 23.5753 |
| QC20U-pH 5.25 | 51.9148 | 50.8976 | 25.3983 | 24.3877 |
| QC20U-pH 5.5 | 49.4637 | 48.4433 | 27.8243 | 26.8049 |
| QC20U-pH 5.75 | 50.651 | 49.6286 | 27.0189 | 25.9981 |
| QC20U-pH 6.0 | 50.6022 | 49.5885 | 26.668 | 25.6252 |
| QC20U-pH 6.25 | 51.4435 | 50.4228 | 26.4013 | 25.3835 |
| QC20U-pH 6.5 | 50.8578 | 49.8398 | 26.1983 | 25.1729 |
| QC20U-pH 6.75 | 50.6035 | 49.5885 | 26.6839 | 25.6683 |
| QC20U-pH 7.0 | 51.9616 | 50.9438 | 25.2311 | 24.2155 |
| QC20U-pH 7.25 | 51.7397 | 50.7207 | 25.3534 | 24.3371 |
| QC20U-pH 7.5 | 52.1368 | 51.1128 | 25.0892 | 24.0746 |
| QC20U-pH 7.75 | 49.0291 | 47.9932 | 27.9496 | 26.9241 |
| QC20U-pH 8.0 | 52.1071 | 51.0871 | 25.3062 | 24.2834 |
| QC20U-pH 8.25 | 52.8048 | 51.7858 | 24.6455 | 23.6007 |
| QC20U-pH 8.5 | 53.9287 | 52.8905 | 23.7768 | 22.7532 |
| QC20U-pH 8.75 | 52.4636 | 51.4398 | 24.8715 | 23.8482 |
| QC20U-pH 9.0 | 51.6338 | 50.5203 | 25.8916 | 24.8839 |
| QC20U-pH 9.25 | 53.2293 | 52.1133 | 24.7205 | 23.6903 |
| QC20U-pH 9.5 | 53.2205 | 52.2033 | 25.076 | 24.029 |

| | PP tubes 3/1/99 | Desorption 3/2/99 |
|----------------|-----------------|----------------------|
| Sample Name | wt. of vial,g | wt. Of vial + sample |
| QC20U-pH 3a | 7.8931 | 8.3943 |
| QC20U-pH 3b | 7.8979 | 8.4044 |
| QC20U-pH 3.5a | 7.9974 | 8.4999 |
| QC20U-pH 3.5b | 7.8513 | 8.3483 |
| QC20U-pH 4a | 7.8731 | 8.3816 |
| QC20U-pH 4b | 7.8696 | 8.3733 |
| QC20U-pH 4.25a | 7.8566 | 8.3583 |
| QC20U-pH 4.25b | 7.8793 | 8.3822 |
| QC20U-pH 4.5a | 7.8156 | 8.3197 |
| QC20U-pH 4.5b | 7.823 | 8.3234 |
| QC20U-pH 4.75a | 7.892 | 8.398 |
| QC20U-pH4.75b | 7.884 | 8.389 |
| QC20U-pH 5a | 7.9411 | 8.4397 |
| QC20U-pH 5b | 7.8524 | 8.3438 |
| QC20U-pH 5.25a | 7.7969 | 8.2953 |
| QC20U-pH 5.25b | 7.8562 | 8.3515 |
| QC20U-pH 5.5a | 7.7862 | 8.2893 |
| QC20U-pH 5.5b | 7.8785 | 8.3785 |
| QC20U-pH 5.75a | 7.8626 | 8.3654 |
| QC20U-pH 5.75b | 7.838 | 8.3381 |
| QC20U-pH 6a | 7.7652 | 8.2696 |
| QC20U-pH 6b | 7.8274 | 8.3278 |
| QC20U-pH 6.25a | 7.8353 | 8.3382 |
| QC20U-pH 6.25a | 7.8454 | 8.3475 |
| QC20U-pH 6.5a | 7.8312 | 8.3306 |
| QC20U-pH 6.5b | 7.9057 | 8.4006 |
| QC20U-pH 6.75a | 7.8367 | 8.3397 |
| QC20U-pH 6.75b | 7.8533 | 8.354 |
| QC20U-pH 7a | 7.8728 | 8.3672 |
| QC20U-pH 7b | 7.8529 | 8.3536 |
| QC20U-pH 7.25a | 7.8147 | 8.3183 |
| QC20U-pH 7.25b | 7.9224 | 8.419 |
| QC20U-pH 7.5a | 7.849 | 8.3455 |
| QC20U-pH 7.5b | 7.871 | 8.3701 |
| QC20U-pH 7.75a | 7.7969 | 8.3011 |
| QC20U-pH 7.75b | 7.8938 | 8.3927 |
| QC20U-pH 8a | 7.9349 | 8.4335 |
| QC20U-pH 8b | 7.8895 | 8.3911 |
| QC20U-pH 8.25a | 7.9818 | 8.4876 |
| QC20U-pH 8.25b | 7.8163 | 8.3228 |
| QC20U-pH 8.5a | 7.8994 | 8.3966 |
| QC20U-pH 8.5b | 7.8785 | 8.3749 |
| QC20U-pH 8.75a | 7.8588 | 8.3614 |
| QC20U-pH 8.75b | 7.861 | 8.3558 |
| QC20U-pH 9a | 7.8927 | 8.3894 |
| QC20U-pH 9b | 7.8332 | 8.3327 |
| QC20U-pH 9.25a | 7.8825 | 8.3821 |
| QC20U-pH 9.25 | 7.8498 | 8.3537 |
| QC20U-pH 9.5a | 7.8533 | 8.3589 |
| QC20U-pH 9.5b | 7.8277 | 8.3328 |

| | PC containers 3/1/99 | Desorption 3/2/99 |
|--|----------------------|----------------------|
| | wt. of vial,g | wt. Of vial + sample |
| | 7.7653 | 8.2698 |
| | 7.7628 | 8.2634 |
| | 7.895 | 8.3932 |
| | 7.7658 | 8.2686 |
| | 7.8499 | 8.3556 |
| | 7.8351 | 8.339 |
| | 7.8783 | 8.3767 |
| | 7.8517 | 8.3562 |
| | 7.7482 | 8.2518 |
| | 7.7813 | 8.2846 |
| | 7.858 | 8.3613 |
| | 7.8378 | 8.342 |
| | 7.7436 | 8.2526 |
| | 7.7219 | 8.226 |
| | 7.8992 | 8.4042 |
| | 7.8721 | 8.3762 |
| | 7.8055 | 8.3127 |
| | 7.7832 | 8.2849 |
| | 7.7715 | 8.2745 |
| | 7.7638 | 8.2681 |
| | 7.8296 | 8.3246 |
| | 7.8362 | 8.3367 |
| | 7.7156 | 8.216 |
| | 7.9053 | 8.4029 |
| | 7.847 | 8.3478 |
| | 7.7847 | 8.2907 |
| | 7.7978 | 8.3014 |
| | 7.8476 | 8.3512 |
| | 7.7699 | 8.2751 |
| | 7.8688 | 8.3749 |
| | 7.8746 | 8.3791 |
| | 7.8619 | 8.3596 |
| | 7.8293 | 8.3272 |
| | 7.8379 | 8.3385 |
| | 7.8131 | 8.3225 |
| | 7.7209 | 8.2257 |
| | 7.7961 | 8.3001 |
| | 7.7789 | 8.2825 |
| | 7.7965 | 8.2996 |
| | 7.7387 | 8.2415 |
| | 7.8354 | 8.3361 |
| | 7.8373 | 8.3375 |
| | 7.8123 | 8.3184 |
| | 7.8237 | 8.3236 |
| | 7.844 | 8.4395 |
| | 7.7624 | 8.267 |
| | 7.7761 | 8.2811 |
| | 7.7973 | 8.394 |
| | 7.8094 | 8.3084 |
| | 7.7978 | 8.302 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 03-Mar-99 11:15

C14 Eff (0-156 keV) = 96.50 %

C14 CHI SQUARE IPA DATA PROCESSED - 03-Mar-99 11:25

C14 Chi Square = 31.30

H3 IPA DATA PROCESSED - 03-Mar-99 11:26

H3 Eff (0-18.6 keV) = 65.30 %

H3 CHI SQUARE IPA DATA PROCESSED - 03-Mar-99 11:37

H3 Chi Square = 23.72

BKG IPA DATA PROCESSED - 03-Mar-99 12:37

Bkg (0-18.6 keV) = 21.78 cpm

Bkg (0-156 keV) = 31.80 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 389.38H3 E²/B (1-18.6 keV) = 194.25

04 Mar 99 05:27

ALPHA/BETA - 1.02

Page

Protocol #:23

U-233 3% 2 sigma

User : A

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.13 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.50 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.52 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|-------|--------|------------|--------------|-------------|----------|
| P# 1 | 999.99 | 20.13 1.41 | 3.502 3.38 | 29.52 1.16 | 143.15 B |
| 23 2 | 8.14 | 5.30 66.93 | 542.567 3.02 | 549.10 3.07 | 609.79 |
| 23 3 | 8.41 | 1.03 308.0 | 525.273 3.02 | 528.15 3.08 | 600.03 |
| 23 4 | 8.08 | 2.52 133.5 | 546.993 3.02 | 551.17 3.08 | 606.06 |
| 23 5 | 8.38 | 0.75 421.6 | 526.928 3.02 | 527.64 3.09 | 611.08 |
| 23 6 | 8.28 | 4.02 85.20 | 533.455 3.02 | 537.26 3.08 | 607.07 |
| 23 7 | 8.14 | 2.60 129.2 | 543.058 3.02 | 546.15 3.08 | 605.29 |
| 23 8 | 12.19 | 3.49 80.09 | 361.059 3.03 | 365.39 3.12 | 602.76 |
| 23 9 | 11.85 | 0.29 908.3 | 371.688 3.03 | 372.67 3.13 | 604.02 |
| 23 10 | 15.65 | 0.00 0.00 | 280.524 3.04 | 280.00 3.18 | 614.17 |
| 23 11 | 15.19 | 1.99 122.2 | 289.059 3.04 | 292.46 3.15 | 603.39 |
| 23 12 | 41.54 | 0.00 0.00 | 103.479 3.10 | 102.54 3.49 | 615.51 |
| 23 13 | 43.27 | 0.44 321.9 | 99.202 3.11 | 100.20 3.47 | 607.71 |
| 23 14 | 47.81 | 0.53 251.9 | 89.533 3.12 | 90.54 3.52 | 606.85 |
| 23 15 | 50.39 | 0.19 683.9 | 84.690 3.13 | 86.37 3.53 | 605.03 |
| 23 16 | 50.89 | 0.00 0.00 | 83.843 3.13 | 83.76 3.59 | 607.05 |
| 23 17 | 50.85 | 0.14 898.1 | 83.932 3.13 | 84.58 3.57 | 607.83 |
| 23 18 | 48.71 | 0.48 277.1 | 87.876 3.12 | 88.52 3.54 | 604.87 |
| 23 19 | 49.13 | 0.06 2184 | 86.952 3.12 | 86.64 3.57 | 608.04 |
| 23 20 | 49.62 | 0.73 182.5 | 86.079 3.12 | 87.12 3.54 | 599.27 |
| 23 21 | 49.95 | 0.49 268.9 | 85.467 3.13 | 85.89 3.56 | 599.21 |
| 23 22 | 54.70 | 0.00 0.00 | 77.741 3.14 | 77.75 3.63 | 610.08 |
| 23 23 | 55.51 | 0.32 395.0 | 76.556 3.14 | 77.47 3.61 | 601.29 |
| 23 24 | 57.71 | 0.13 972.8 | 73.504 3.15 | 74.25 3.64 | 602.61 |
| 23 25 | 58.79 | 0.72 169.4 | 72.106 3.15 | 73.32 3.64 | 592.57 |
| 23 26 | 63.11 | 0.85 140.0 | 66.915 3.16 | 67.56 3.71 | 595.60 |
| 23 27 | 61.38 | 0.62 191.6 | 68.899 3.16 | 70.18 3.66 | 591.16 |
| 23 28 | 34.08 | 0.09 1823 | 126.926 3.08 | 126.70 3.39 | 598.49 |
| 23 29 | 32.55 | 0.24 676.9 | 133.119 3.08 | 133.36 3.36 | 603.98 |
| 23 30 | 56.60 | 0.00 0.00 | 75.014 3.14 | 74.82 3.66 | 600.62 |
| 23 31 | 57.14 | 0.06 1886 | 74.289 3.15 | 74.43 3.65 | 609.33 |
| 23 32 | 51.22 | 0.41 318.4 | 83.261 3.13 | 83.85 3.57 | 600.38 |
| 23 33 | 52.41 | 2.77 48.89 | 81.291 3.13 | 83.89 3.53 | 590.00 |
| 23 34 | 35.38 | 1.12 140.2 | 122.106 3.09 | 123.98 3.37 | 600.93 |
| 23 35 | 34.77 | 0.00 0.00 | 124.309 3.09 | 124.78 3.39 | 606.55 |
| 23 36 | 51.89 | 0.00 0.00 | 82.160 3.13 | 82.50 3.59 | 608.68 |
| 23 37 | 50.79 | 0.19 690.5 | 84.015 3.13 | 85.05 3.56 | 604.27 |
| 23 38 | 38.88 | 1.14 132.2 | 110.798 3.10 | 112.76 3.41 | 600.55 |
| 23 39 | 40.10 | 1.12 132.9 | 107.321 3.10 | 109.30 3.42 | 603.68 |
| 23 40 | 38.05 | 0.95 160.1 | 113.292 3.09 | 114.29 3.42 | 602.30 |
| 23 41 | 37.70 | 1.46 105.5 | 114.376 3.09 | 116.68 3.39 | 602.75 |

container
description
action
uncapped

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Protocol #:23 U-233 3% 2 sigma User : A

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 29.51 | 0.00 | 0.00 | 147.159 | 3.07 | 147.03 | 3.34 | 609.22 |
| 23 | 43 | 30.72 | 0.00 | 0.00 | 141.159 | 3.08 | 142.77 | 3.33 | 608.67 |
| 23 | 44 | 21.16 | 1.32 | 153.6 | 206.564 | 3.05 | 209.47 | 3.21 | 603.33 |
| 23 | 45 | 22.04 | 2.24 | 90.95 | 198.222 | 3.05 | 200.83 | 3.22 | 600.27 |
| 23 | 46 | 14.75 | 0.00 | 0.00 | 297.922 | 3.03 | 299.15 | 3.16 | 606.96 |
| 23 | 47 | 14.01 | 2.35 | 108.4 | 313.700 | 3.03 | 317.09 | 3.14 | 604.15 |
| 23 | 48 | 10.77 | 1.32 | 215.3 | 409.404 | 3.02 | 411.05 | 3.11 | 604.53 |
| 23 | 49 | 10.92 | 3.40 | 86.67 | 403.458 | 3.03 | 406.37 | 3.11 | 604.10 |
| 23 | 50 | 9.57 | 4.32 | 74.29 | 461.388 | 3.02 | 467.03 | 3.09 | 601.07 |
| 23 | 51 | 9.52 | 2.35 | 131.5 | 463.725 | 3.02 | 465.33 | 3.10 | 606.07 |
| 23 | 52 | 7.73 | 1.86 | 181.9 | 571.401 | 3.02 | 573.32 | 3.08 | 595.91 |
| 23 | 53 | 9.43 | 3.30 | 95.79 | 467.972 | 3.02 | 472.60 | 3.09 | 605.18 |
| 23 | 54 | 9.33 | 0.00 | 0.00 | 472.918 | 3.02 | 473.69 | 3.10 | 613.01 |
| 23 | 55 | 7.87 | 0.00 | 0.00 | 561.555 | 3.02 | 561.20 | 3.09 | 602.12 |
| 23 | 56 | 9.86 | 1.57 | 189.5 | 447.208 | 3.02 | 449.28 | 3.10 | 612.04 |
| 23 | 57 | 9.95 | 2.78 | 109.5 | 443.634 | 3.02 | 446.05 | 3.10 | 612.35 |

231 MISSING TUBE(S)

59 5.62 5.85 73.70 787.957 3.01 795.92 3.05 600.01

| | | | | | | | | | |
|----|----|------|-------|-------|----------|------|---------|------|--------|
| 23 | 60 | 5.65 | 3.41 | 120.1 | 784.109 | 3.01 | 788.53 | 3.05 | 597.64 |
| 23 | 61 | 4.34 | 8.67 | 59.51 | 1020.922 | 3.01 | 1031.31 | 3.03 | 598.76 |
| 23 | 62 | 4.11 | 2.25 | 207.5 | 1078.736 | 3.01 | 1081.91 | 3.04 | 601.61 |
| 23 | 63 | 2.39 | 2.46 | 249.9 | 1855.912 | 3.01 | 1856.25 | 3.03 | 596.91 |
| 23 | 64 | 2.37 | 2.23 | 275.6 | 1880.464 | 3.00 | 1885.24 | 3.02 | 602.91 |
| 23 | 65 | 1.94 | 5.13 | 140.9 | 2293.921 | 3.00 | 2298.83 | 3.01 | 601.61 |
| 23 | 66 | 2.00 | 11.87 | 67.45 | 2224.498 | 3.00 | 2237.98 | 3.01 | 597.64 |
| 23 | 67 | 2.63 | 11.05 | 62.39 | 1691.555 | 3.00 | 1703.94 | 3.01 | 597.37 |
| 23 | 68 | 2.52 | 5.66 | 113.1 | 1760.784 | 3.01 | 1770.08 | 3.02 | 600.71 |
| 23 | 69 | 2.64 | 6.76 | 94.48 | 1680.968 | 3.01 | 1687.90 | 3.02 | 600.89 |
| 23 | 70 | 2.66 | 1.67 | 342.6 | 1669.054 | 3.00 | 1668.22 | 3.03 | 603.83 |
| 23 | 71 | 2.61 | 2.09 | 279.4 | 1703.395 | 3.00 | 1708.41 | 3.02 | 602.69 |
| 23 | 72 | 2.57 | 6.33 | 101.5 | 1730.739 | 3.00 | 1737.01 | 3.02 | 602.28 |
| 23 | 73 | 2.74 | 11.62 | 58.64 | 1618.761 | 3.01 | 1633.25 | 3.02 | 598.47 |
| 23 | 74 | 2.72 | 11.49 | 59.42 | 1630.321 | 3.01 | 1642.54 | 3.02 | 602.40 |
| 23 | 75 | 3.34 | 6.52 | 86.81 | 1327.336 | 3.01 | 1336.34 | 3.03 | 601.09 |
| 23 | 76 | 3.32 | 0.95 | 529.6 | 1336.257 | 3.01 | 1337.04 | 3.03 | 602.22 |
| 23 | 77 | 3.03 | 7.26 | 82.91 | 1465.145 | 3.01 | 1476.42 | 3.02 | 604.28 |
| 23 | 78 | 3.10 | 8.90 | 68.84 | 1433.595 | 3.00 | 1444.03 | 3.02 | 601.20 |
| 23 | 79 | 3.33 | 7.80 | 74.38 | 1332.834 | 3.01 | 1339.25 | 3.03 | 596.21 |
| 23 | 80 | 3.35 | 2.56 | 204.0 | 1324.856 | 3.01 | 1325.40 | 3.03 | 600.79 |
| 23 | 81 | 2.92 | 8.98 | 70.40 | 1523.553 | 3.00 | 1532.12 | 3.02 | 602.71 |
| 23 | 82 | 2.97 | 4.45 | 129.5 | 1497.171 | 3.00 | 1504.15 | 3.02 | 597.21 |
| 23 | 83 | 2.92 | 4.18 | 138.1 | 1525.950 | 3.00 | 1530.75 | 3.02 | 601.71 |
| 23 | 84 | 2.94 | 4.70 | 123.8 | 1510.103 | 3.01 | 1516.05 | 3.02 | 604.20 |
| 23 | 85 | 3.28 | 6.09 | 92.99 | 1352.595 | 3.01 | 1360.42 | 3.03 | 599.44 |
| 23 | 86 | 3.30 | 8.05 | 72.68 | 1345.286 | 3.01 | 1353.51 | 3.03 | 601.54 |
| 23 | 87 | 2.65 | 5.91 | 106.2 | 1677.630 | 3.00 | 1681.80 | 3.02 | 599.66 |
| 23 | 88 | 2.63 | 1.16 | 490.5 | 1688.133 | 3.00 | 1689.49 | 3.03 | 604.02 |
| 23 | 89 | 2.90 | 3.32 | 171.7 | 1528.912 | 3.01 | 1532.20 | 3.03 | 600.36 |
| 23 | 90 | 3.00 | 1.20 | 444.3 | 1485.165 | 3.00 | 1486.14 | 3.02 | 605.94 |

| | | | | | | | | | |
|----|----|------|------|-------|----------|------|---------|------|--------|
| 23 | 91 | 2.74 | 7.61 | 83.75 | 1620.950 | 3.00 | 1629.24 | 3.02 | 603.07 |
| 23 | 92 | 2.84 | 6.28 | 97.26 | 1565.160 | 3.00 | 1571.18 | 3.02 | 600.68 |
| 23 | 93 | 3.71 | 1.70 | 285.6 | 1196.228 | 3.01 | 1198.78 | 3.04 | 604.32 |
| 23 | 94 | 3.66 | 1.45 | 334.7 | 1212.072 | 3.01 | 1213.92 | 3.04 | 604.45 |
| 23 | 95 | 3.34 | 3.52 | 151.4 | 1328.534 | 3.01 | 1331.55 | 3.03 | 606.93 |
| 23 | 96 | 3.44 | 0.00 | 0.00 | 1290.975 | 3.01 | 1292.28 | 3.03 | 602.60 |
| 23 | 97 | 4.28 | 6.50 | 76.83 | 1036.685 | 3.01 | 1043.84 | 3.03 | 601.42 |

98 4.42 4.76 99.97 1003.285 3.01 1010.07 3.04 598.13

05 Mar 99 13:57 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma Use

| | | | | | | | | | |
|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 99 | 6.56 | 1.82 | 201.6 | 674.547 | 3.01 | 675.51 | 3.07 | 602.87 |
| 23 | 100 | 6.48 | 1.78 | 207.0 | 682.609 | 3.01 | 685.91 | 3.06 | 601.34 |
| 23 | 101 | 9.54 | 4.08 | 78.35 | 462.431 | 3.02 | 466.07 | 3.09 | 596.39 |
| 23 | 102 | 9.45 | 2.94 | 106.8 | 466.763 | 3.02 | 471.64 | 3.09 | 599.54 |
| 23 | 103 | 10.60 | 4.30 | 70.88 | 415.838 | 3.03 | 419.34 | 3.10 | 601.60 |
| 23 | 104 | 10.80 | 4.22 | 71.47 | 407.979 | 3.03 | 412.98 | 3.10 | 597.46 |
| 23 | 105 | 11.82 | 4.49 | 64.62 | 372.471 | 3.03 | 376.31 | 3.12 | 599.25 |
| 23 | 106 | 11.62 | 2.07 | 134.1 | 378.942 | 3.03 | 382.27 | 3.12 | 598.52 |
| 23 | 107 | 12.23 | 0.72 | 365.3 | 360.112 | 3.03 | 360.66 | 3.13 | 602.51 |
| 23 | 108 | 11.87 | 3.63 | 78.42 | 371.140 | 3.03 | 375.19 | 3.11 | 596.16 |

23 SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 05-Mar-99 15:47

C14 Eff (0-156 keV) = 97.32 %

C14 CHI SQUARE IPA DATA PROCESSED - 05-Mar-99 15:58

C14 Chi Square = 14.94

H3 IPA DATA PROCESSED - 05-Mar-99 15:59

H3 Eff (0-18.6 keV) = 65.11 %

H3 CHI SQUARE IPA DATA PROCESSED - 05-Mar-99 16:10

H3 Chi Square = 20.41

BKG IPA DATA PROCESSED - 05-Mar-99 17:10

Bkg (0-18.6 keV) = 21.60 cpm

Bkg (0-156 keV) = 31.37 cpm

WARNING: Questionable C14 Background value - Please view historic

C14 E²/B (1-156 keV) = 396.11

H3 E²/B (1-18.6 keV) = 194.52

QC20U CAPPED EXPERIMENTS

2/18/99

Experimental Procedure for experiment - U sorption on mixed minerals (clinoptilolite and quartz) at 25° C.

Objective: To investigate the characteristics of U sorption on mixture of clinoptilolite and quartz as a function of pH.

Initial conditions: - $\Sigma U = 50 \text{ ppb}$ (2.14×10^{-7})

- capped polycarbonate containers
- pH range 3.5-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 700mg (0.70 g)
- M/V = 28.57 g/L

Equipment: Orion 920A pH meter and combination pH electrode
 Glove box
 CO₂ gas
 Gyrotory shaker
 Fisher Marathon 21K centrifuge
 Mettler PM4600 and AE240 balances
 Packard 2500 TR/AB LSC
 Eppendorf micropipettors and tips
 Oxford pipettes and tips
 Repipettor for transfer of scintillation cocktail

Supplies:

≤ 325 mesh Qtz & 100/200 mesh Clinoptilolite in NaF AT
 50 ppb ²³³U stock solution
 polycarbonate centrifuge tubes with caps (50 ml capacity)
 polypropylene centrifuge tubes with caps (50 ml capacity)
 pH buffer solutions
 Ultima-Gold liquid scintillation cocktail
 7 ml scintillation vials
 reagent grade NaOH (lot 976631)
 concentrated HNO₃ (lot 983546)
 HNO₃ and NaOH stock solutions at various concentrations prepared in degassed water.
 0.1M NaNO₃ matrix prepared with degassed water.
 weighing paper/boats
 deionized ultrapure water, DH₂O
 Teflon beaker

Note: Ultrapure water was degassed by boiling it in a flask and cooled with mouth covered with rubber stopper.

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using 0.1M degassed NaNO₃ stock solution.
 - Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ²³³U stock solution to experimental containers.
- Label 24 polycarbonate centrifuge tubes (50 ml capacity) QCU-pHi (where i is the approximate pH of each solution, see Table QCU).

- Label 1 polycarbonate centrifuge tube QC20U-IU(capped).
- Weigh and record the weight of each container.
- About 1L of ²³³U solution will be required for this experiment.
- Add ~35 g of the 50 ppb ²³³U stock solution to each container. Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.
- Record weight of each container.

C. Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the QC20U-IU(capped) container.
- Record weight of each sample container after addition of quartz.

D. Add clinoptilolite to sample containers.

- Add 0.700 g of clinoptilolite to each sample container. **Do not** add clinoptilolite to the QC20U-IU(capped) container.
- Record weight of each sample container after addition of clinoptilolite.

E. Adjust pH of each solution by adding HNO₃ or NaOH.

- Add the volume of HNO₃ or NaOH to each experimental container as listed in Table QC20U. Take care to add HNO₃ or NaOH in the concentrations specified in Table CQ20U.
- For sample QC20U-IU(capped) (as listed in Table QC20U) add 100 μ l of 50/50 V/V HNO₃ to the sample container.
- Weigh and record the weight of each sample container.

F. Sample QC20U-IU(capped) for initial [U].

- Label two 7 ml LSC vials as QC20U-IUa(capped) and QC20U-IUb(capped).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from QC20U-IU(capped) to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container QC20U-IU(capped).

G. Wait for equilibrium.

- Place caps on sample containers and do not tighten the caps.
- Place the containers on gyrotory shaker (~120 rpm) placed in carbon controlled atmosphere glove box for 2-3 days.
- Tighten the containers and equilibrate the samples for at least 10 days.

H. Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., QC20U-pH3a and QC20U-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- Measure pH of each experimental solution inside the glove box.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the

- appropriately labeled LSC vials inside the glove box.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container after pH measurement and sampling.

I. Transfer mixture of quartz and clinoptilolite for desorption.

- Record weight of PC sample containers. *Note: this step can be skipped if the mixture transfer is done immediately after the last step in section H.*
- For each PC sample container, label two corresponding polypropylene (PP) test tubes A and B(50 ml capacity).
- Record weight for the set of PP tubes A .
- Add 3.0 ml of 0.1M HNO₃ to 50 ml PP tubes labeled as **B-QC20Ud-pH(capped)**.
- Pour supernatant from PC containers into PP tubes with acid and labeled as **B-QC20Ud-pH(capped)** leaving behind about 7 to 8ml of solution in PC container with the solid mixture.
- Record the weight of PC containers containing the solid mixture and some solution.
- Stir the solid mixture in solution vigorously and immediately transfer all the solid and solution to the corresponding PP tubes marked as **A-QC20Ud-pH(capped)**.
- Record the weight of PP tubes **A-QC20Ud-pH(capped)**.
- Transfer supernatant with HNO₃ acid from the PP tubes **B-QC20Ud-pH(capped)** back to original PC containers.
- Record weights of PC sample containers.
- Add approximately 3 ml of 0.1 M HNO₃ to PP tubes containing solid mixture, **A-QC20Ud-pH(capped)**.
- Record weights of PP tubes **A-QC20Ud-pH(capped)** and PC sample containers.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

J. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QC20Ud-pH3a(capped) and QC20Ud-pH3b(capped)).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquot of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

CAPPED

2/18/99

| Sample Name | Original Container wt., g |
|---------------|---------------------------|
| QC20U-pH 3.0 | 22.1645 |
| QC20U-pH 3.5 | 22.1693 |
| QC20U-pH 4.0 | 22.348 |
| QC20U-pH 4.25 | 22.2912 |
| QC20U-pH 4.5 | 22.3223 |
| QC20U-pH4.75 | 22.0611 |
| QC20U-pH 5.0 | 22.2156 |
| QC20U-pH 5.25 | 22.5646 |
| QC20U-pH 5.5 | 22.2628 |
| QC20U-pH 5.75 | 22.3321 |
| QC20U-pH 6.0 | 22.295 |
| QC20U-pH 6.25 | 22.2479 |
| QC20U-pH 6.5 | 22.2783 |
| QC20U-pH 6.75 | 22.3205 |
| QC20U-pH 7.0 | 22.5938 |
| QC20U-pH 7.25 | 22.2407 |
| QC20U-pH 7.5 | 22.3038 |
| QC20U-pH 7.75 | 22.324 |
| QC20U-pH 8.0 | 22.2959 |
| QC20U-pH 8.25 | 22.0777 |
| QC20U-pH 8.5 | 22.2543 |
| QC20U-pH 8.75 | 22.2717 |
| QC20U-pH 9.0 | 22.5278 |
| QC20U-pH 9.25 | 22.1685 |
| | |
| QC20U-IU | 22.6264 |

CAPPED

2/18/99

| Sample Name | Container wt. + quartz, g |
|---------------|---------------------------|
| QC20U-pH 3.0 | 23.1662 |
| QC20U-pH 3.5 | 23.1719 |
| QC20U-pH 4.0 | 23.3516 |
| QC20U-pH 4.25 | 23.2952 |
| QC20U-pH 4.5 | 23.3256 |
| QC20U-pH4.75 | 23.0628 |
| QC20U-pH 5.0 | 23.2178 |
| QC20U-pH 5.25 | 23.5637 |
| QC20U-pH 5.5 | 23.2676 |
| QC20U-pH 5.75 | 23.3345 |
| QC20U-pH 6.0 | 23.2958 |
| QC20U-pH 6.25 | 23.2487 |
| QC20U-pH 6.5 | 23.2807 |
| QC20U-pH 6.75 | 23.3225 |
| QC20U-pH 7.0 | 23.5946 |
| QC20U-pH 7.25 | 23.2451 |
| QC20U-pH 7.5 | 23.3053 |
| QC20U-pH 7.75 | 23.3253 |
| QC20U-pH 8.0 | 23.2988 |
| QC20U-pH 8.25 | 23.0809 |
| QC20U-pH 8.5 | 23.2574 |
| QC20U-pH 8.75 | 23.2762 |
| QC20U-pH 9.0 | 23.5265 |
| QC20U-pH 9.25 | 23.1691 |

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2/18/99

| Sample Name | Container wt. + clinop + quartz, g |
|---------------|------------------------------------|
| QC20U-pH 3.0 | 23.867 |
| QC20U-pH 3.5 | 23.872 |
| QC20U-pH 4.0 | 24.0518 |
| QC20U-pH 4.25 | 23.9967 |
| QC20U-pH 4.5 | 24.0259 |
| QC20U-pH4.75 | 23.7629 |
| QC20U-pH 5.0 | 23.9181 |
| QC20U-pH 5.25 | 24.2636 |
| QC20U-pH 5.5 | 23.9682 |
| QC20U-pH 5.75 | 24.0348 |
| QC20U-pH 6.0 | 23.9967 |
| QC20U-pH 6.25 | 23.9495 |
| QC20U-pH 6.5 | 23.9807 |
| QC20U-pH 6.75 | 24.0229 |
| QC20U-pH 7.0 | 24.2954 |
| QC20U-pH 7.25 | 23.9456 |
| QC20U-pH 7.5 | 24.0054 |
| QC20U-pH 7.75 | 24.0258 |
| QC20U-pH 8.0 | 23.9988 |
| QC20U-pH 8.25 | 23.7808 |
| QC20U-pH 8.5 | 23.9578 |
| QC20U-pH 8.75 | 23.9759 |
| QC20U-pH 9.0 | 24.2278 |
| QC20U-pH 9.25 | 23.8703 |

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2/19/99

2/19/99

| Sample Name | Container wt. + U soln. + quartz + clinop, g | Container Wt. After additions of HNO ₃ and NaOH, g |
|---------------|--|---|
| QC20U-pH 3.0 | 58.8723 | 58.9335 |
| QC20U-pH 3.5 | 58.944 | 59.1436 |
| QC20U-pH 4.0 | 59.1211 | 59.4955 |
| QC20U-pH 4.25 | 58.9962 | 59.1891 |
| QC20U-pH 4.5 | 59.0256 | 59.0521 |
| QC20U-pH 4.75 | 58.7745 | 58.8245 |
| QC20U-pH 5.0 | 58.9222 | 58.9974 |
| QC20U-pH 5.25 | 59.2142 | 59.3136 |
| QC20U-pH 5.5 | 58.9623 | 59.0885 |
| QC20U-pH 5.75 | 59.0616 | 59.211 |
| QC20U-pH 6.0 | 59.0648 | 59.2393 |
| QC20U-pH 6.25 | 58.9734 | 59.1716 |
| QC20U-pH 6.5 | 58.9595 | 59.1806 |
| QC20U-pH 6.75 | 59.058 | 59.431 |
| QC20U-pH 7.0 | 59.2916 | 59.6948 |
| QC20U-pH 7.25 | 58.9948 | 59.0436 |
| QC20U-pH 7.5 | 59.0042 | 59.1128 |
| QC20U-pH 7.75 | 58.9948 | 59.1186 |
| QC20U-pH 8.0 | 59.024 | 59.1725 |
| QC20U-pH 8.25 | 58.7936 | 58.968 |
| QC20U-pH 8.5 | 58.9327 | 58.9988 |
| QC20U-pH 8.75 | 58.9559 | 59.0479 |
| QC20U-pH 9.0 | 59.2495 | 59.3745 |
| QC20U-pH 9.25 | 58.8202 | 58.9698 |
| | | |
| QC20U-IU | 57.6826 | 57.8071 |

CAPPED

3/16/99

| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|---------------|--|-------------|---|
| QC20U-pH 3.0 | 58.2421 | 3.39 | 57.2146 |
| QC20U-pH 3.5 | 58.7987 | 4.26 | 57.643 |
| QC20U-pH 4.0 | 59.2202 | 3.95 | 58.1919 |
| QC20U-pH 4.25 | 58.709 | 5.15 | 57.653 |
| QC20U-pH 4.5 | 58.6449 | 5.47 | 57.5839 |
| QC20U-pH 4.75 | 58.2196 | 5.36 | 56.8192 |
| QC20U-pH 5.0 | 58.1086 | 5.81 | 56.9773 |
| QC20U-pH 5.25 | 58.5654 | 5.96 | 57.5087 |
| QC20U-pH 5.5 | 58.3292 | 5.68 | 57.1823 |
| QC20U-pH 5.75 | 58.6344 | 6.05 | 57.5887 |
| QC20U-pH 6.0 | 58.4954 | 5.92 | 57.2897 |
| QC20U-pH 6.25 | 58.3248 | 6.67 | 57.1786 |
| QC20U-pH 6.5 | 58.5232 | 6.71 | 57.3612 |
| QC20U-pH 6.75 | 58.8635 | 6.82 | 57.7661 |
| QC20U-pH 7.0 | 59.1571 | 6.98 | 57.7568 |
| QC20U-pH 7.25 | 58.2569 | 7.24 | 57.0805 |
| QC20U-pH 7.5 | 58.6548 | 7.52 | 57.4606 |
| QC20U-pH 7.75 | 58.5561 | 8.17 | 57.2374 |
| QC20U-pH 8.0 | 58.5422 | 8.09 | 57.2802 |
| QC20U-pH 8.25 | 58.5276 | 8.49 | 57.3146 |
| QC20U-pH 8.5 | 58.1073 | 8.35 | 57.0688 |
| QC20U-pH 8.75 | 58.2252 | 8.82 | 57.176 |
| QC20U-pH 9.0 | 58.7419 | 9.06 | 57.7077 |
| QC20U-pH 9.25 | 57.9519 | 9.24 | 56.8521 |

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3/20/99
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CAPPED SORPTION

| Sample Name | wt. of vial,g | wt. Of vial + sample |
|----------------|---------------|----------------------|
| QC20U-pH 3a | 7.8899 | 8.3877 |
| QC20U-pH 3b | 7.8982 | 8.4014 |
| QC20U-pH 3.5a | 7.8704 | 8.366 |
| QC20U-pH 3.5b | 7.906 | 8.4029 |
| QC20U-pH 4a | 7.818 | 8.3172 |
| QC20U-pH 4b | 7.919 | 8.4258 |
| QC20U-pH 4.25a | 7.904 | 8.4009 |
| QC20U-pH 4.25b | 7.9179 | 8.4147 |
| QC20U-pH 4.5a | 7.859 | 8.3497 |
| QC20U-pH 4.5b | 7.8824 | 8.3823 |
| QC20U-pH 4.75a | 7.8325 | 8.3328 |
| QC20U-pH 4.75b | 7.8233 | 8.3288 |
| QC20U-pH 5a | 7.8197 | 8.3149 |
| QC20U-pH 5b | 7.8969 | 8.4009 |
| QC20U-pH 5.25a | 7.8788 | 8.3759 |
| QC20U-pH 5.25b | 7.9875 | 8.4821 |
| QC20U-pH 5.5a | 7.8879 | 8.3818 |
| QC20U-pH 5.5b | 7.8361 | 8.3391 |
| QC20U-pH 5.75a | 7.8536 | 8.3444 |
| QC20U-pH 5.75b | 7.8405 | 8.3441 |
| QC20U-pH 6a | 7.9153 | 8.4062 |
| QC20U-pH 6b | 7.8395 | 8.3373 |
| QC20U-pH 6.25a | 7.8539 | 8.3487 |
| QC20U-pH 6.25a | 7.8961 | 8.3791 |
| QC20U-pH 6.5a | 7.904 | 8.3978 |
| QC20U-pH 6.5b | 7.9156 | 8.4124 |
| QC20U-pH 6.75a | 7.8607 | 8.3604 |
| QC20U-pH 6.75b | 7.8669 | 8.3656 |
| QC20U-pH 7a | 7.8234 | 8.3188 |
| QC20U-pH 7b | 7.8597 | 8.3662 |
| QC20U-pH 7.25a | 7.8595 | 8.3625 |
| QC20U-pH 7.25b | 7.9953 | 8.4939 |
| QC20U-pH 7.5a | 7.8041 | 8.2973 |
| QC20U-pH 7.5b | 7.8532 | 8.3457 |
| QC20U-pH 7.75a | 7.8752 | 8.371 |
| QC20U-pH 7.75b | 7.8656 | 8.3708 |
| QC20U-pH 8a | 7.8655 | 8.3594 |
| QC20U-pH 8b | 7.8354 | 8.3394 |
| QC20U-pH 8.25a | 7.7891 | 8.2851 |
| QC20U-pH 8.25b | 7.8859 | 8.3855 |
| QC20U-pH 8.5a | 7.8922 | 8.3888 |
| QC20U-pH 8.5b | 7.8665 | 8.3654 |
| QC20U-pH 8.75a | 7.9086 | 8.4128 |
| QC20U-pH 8.75b | 7.8747 | 8.3758 |
| QC20U-pH 9a | 7.8579 | 8.3585 |
| QC20U-pH 9b | 7.8228 | 8.328 |
| QC20U-pH 9.25a | 7.9481 | 8.4543 |
| QC20U-pH 9.25 | 7.8739 | 8.3764 |
| QC20U-pH 9.5a | 7.8466 | 8.3219 |
| QC20U-pH 9.5b | 7.823 | 8.2963 |
| QC1.0U-Iua | 7.7876 | 8.2933 |
| QC1.0U-IUb | 7.812 | 8.3168 |
| QC1.0U-IUc | 7.812 | 8.3168 |
| QC1.0U-IUd | 7.7876 | 8.2933 |
| QC1.0U-IUe | 7.8399 | 8.3457 |
| QC1.0U-Iuf | 7.8136 | 8.3189 |
| QC1.0U-Iug | 7.7792 | 8.2779 |
| QC1.0U-Iuh | 7.7917 | 8.2897 |

234 MISSING TUBE(S) 2/21/99

109 8.31 5.49 63.91 531.313 3.02 539.23 3.07 608.72
 23 110 8.29 5.31 65.92 532.604 3.02 542.41 3.06 609.32
 23 111 8.13 3.84 89.39 543.891 3.02 551.32 3.07 606.17
 23 112 8.34 5.88 60.03 529.510 3.02 538.74 3.06 609.57
 23 113 8.04 3.85 89.53 549.894 3.02 558.32 3.06 607.46
 23 114 8.11 4.88 71.86 544.501 3.02 553.00 3.07 603.82

QC20U
-IU
capped

SYSTEM NORMALIZED

23 Mar 99 03:10 ALPHA/BETA - 1.02
 Protocol #:23 U-233 3% 2 sigma

Page

User : A

C14 IPA DATA PROCESSED - 23-Mar-99 03:10

C14 Eff (0-156 keV) = 95.99 %

C14 CHI SQUARE IPA DATA PROCESSED - 23-Mar-99 03:21

C14 Chi Square = 21.35

H3 IPA DATA PROCESSED - 23-Mar-99 03:22

H3 Eff (0-18.6 keV) = 64.59 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves
 & view historic data

H3 CHI SQUARE IPA DATA PROCESSED - 23-Mar-99 03:32

H3 Chi Square = 10.60

BKG IPA DATA PROCESSED - 23-Mar-99 04:33

Bkg (0-18.6 keV) = 20.23 cpm

Bkg (0-156 keV) = 29.92 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 395.58

H3 E²/B (1-18.6 keV) = 203.86

| | | wt. of vial | wt. of vial + sample |
|---------|--------------|-------------|----------------------|
| 2/19/99 | QC20U - I Va | 7.7884 | 8.2908 |
| | QC20U - I Vb | 7.8063 | 8.3083 |
| | QC20U - I Vc | 7.7888 | 8.2910 |
| | QC20U - I Vd | 7.8525 | 8.3558 |
| | QC20U - I Ve | 7.7662 | 8.2694 |
| | QC20U - I Vf | 7.7820 | 8.2859 |

3/21/99

AT
(Ext-2)
QC20U (2 set) Capped QC20U

| | | | | | | | | | |
|----|------|-------|------|---------|---------|--------|--------|--------|--------|
| 55 | 9.78 | 0.00 | 0.00 | 451.020 | 3.02 | 449.90 | 3.11 | 616.11 | |
| 23 | 56 | 9.69 | 1.34 | 222.3 | 455.756 | 3.02 | 457.03 | 3.10 | 614.52 |
| 23 | 57 | 15.95 | 0.61 | 377.7 | 275.369 | 3.04 | 275.69 | 3.17 | 617.74 |
| 23 | 58 | 15.77 | 0.33 | 683.7 | 278.424 | 3.04 | 279.23 | 3.17 | 617.12 |
| 23 | 59 | 13.05 | 1.28 | 200.5 | 337.159 | 3.03 | 338.22 | 3.14 | 608.75 |
| 23 | 60 | 12.72 | 1.76 | 149.8 | 345.994 | 3.03 | 347.91 | 3.13 | 612.85 |
| 23 | 61 | 29.10 | 0.50 | 343.8 | 149.372 | 3.07 | 150.28 | 3.31 | 614.67 |
| 23 | 62 | 30.10 | 1.04 | 162.7 | 144.297 | 3.07 | 145.28 | 3.32 | 617.02 |
| 23 | 63 | 54.15 | 1.09 | 117.7 | 78.691 | 3.13 | 80.30 | 3.57 | 613.64 |
| 23 | 64 | 49.98 | 0.75 | 176.4 | 85.539 | 3.12 | 86.95 | 3.53 | 615.45 |
| 23 | 65 | 33.28 | 0.56 | 284.0 | 130.187 | 3.08 | 131.41 | 3.35 | 612.24 |
| 23 | 66 | 32.34 | 0.48 | 336.4 | 134.038 | 3.08 | 135.39 | 3.34 | 611.49 |
| 23 | 67 | 73.00 | 0.25 | 430.0 | 57.513 | 3.18 | 58.45 | 3.79 | 615.02 |
| 23 | 68 | 71.95 | 0.44 | 251.7 | 58.402 | 3.18 | 58.87 | 3.80 | 613.72 |
| 23 | 69 | 57.83 | 0.00 | 0.00 | 73.607 | 3.14 | 73.71 | 3.65 | 619.54 |
| 23 | 70 | 58.01 | 0.84 | 147.0 | 73.248 | 3.14 | 74.84 | 3.61 | 617.02 |
| 23 | 71 | 44.37 | 0.60 | 231.4 | 96.781 | 3.11 | 97.57 | 3.48 | 612.81 |
| 23 | 72 | 42.07 | 0.92 | 156.2 | 102.280 | 3.10 | 104.21 | 3.43 | 608.28 |
| 23 | 73 | 54.45 | 1.52 | 84.69 | 78.239 | 3.13 | 80.58 | 3.55 | 610.22 |
| 23 | 74 | 51.07 | 0.54 | 240.9 | 83.641 | 3.12 | 84.31 | 3.56 | 613.30 |
| 23 | 75 | 56.46 | 0.00 | 0.00 | 75.334 | 3.14 | 75.52 | 3.63 | 619.68 |
| 23 | 76 | 56.55 | 0.00 | 0.00 | 75.208 | 3.14 | 74.91 | 3.65 | 619.58 |
| 23 | 77 | 82.19 | 1.47 | 72.35 | 50.693 | 3.21 | 52.29 | 3.86 | 602.88 |
| 23 | 78 | 83.43 | 1.35 | 77.80 | 49.901 | 3.21 | 51.92 | 3.85 | 610.95 |
| 23 | 79 | 84.12 | 0.00 | 0.00 | 49.464 | 3.21 | 49.23 | 3.98 | 624.07 |
| 23 | 80 | 85.32 | 0.00 | 0.00 | 48.709 | 3.22 | 49.08 | 3.96 | 621.14 |
| 23 | 81 | 77.63 | 0.62 | 173.5 | 53.908 | 3.19 | 55.08 | 3.83 | 613.25 |
| 23 | 82 | 77.45 | 0.08 | 1273 | 54.002 | 3.20 | 54.92 | 3.84 | 614.83 |
| 23 | 83 | 61.07 | 1.18 | 102.3 | 69.425 | 3.15 | 71.11 | 3.64 | 609.41 |
| 23 | 84 | 63.08 | 1.10 | 108.7 | 67.073 | 3.16 | 68.37 | 3.67 | 606.05 |
| 23 | 85 | 97.80 | 0.35 | 274.7 | 42.063 | 3.25 | 43.04 | 4.07 | 613.10 |
| 23 | 86 | 97.60 | 0.54 | 176.8 | 42.156 | 3.25 | 43.02 | 4.08 | 618.09 |
| 23 | 87 | 65.18 | 0.61 | 188.7 | 64.803 | 3.16 | 65.89 | 3.70 | 612.00 |
| 23 | 88 | 64.53 | 0.30 | 391.1 | 65.506 | 3.16 | 65.96 | 3.72 | 615.98 |
| 23 | 89 | 66.35 | 0.00 | 0.00 | 63.631 | 3.16 | 64.38 | 3.73 | 624.66 |
| 23 | 90 | 65.22 | 0.68 | 171.1 | 64.792 | 3.16 | 65.79 | 3.70 | 608.45 |
| 23 | 91 | 65.09 | 0.00 | 0.00 | 64.898 | 3.16 | 65.21 | 3.73 | 618.83 |
| 23 | 92 | 63.31 | 0.00 | 0.00 | 66.865 | 3.16 | 67.53 | 3.69 | 617.52 |
| 23 | 93 | 70.22 | 0.17 | 639.4 | 59.910 | 3.18 | 60.51 | 3.78 | 613.63 |
| 23 | 94 | 67.39 | 0.18 | 643.7 | 62.567 | 3.17 | 63.65 | 3.73 | 618.83 |
| 23 | 95 | 86.47 | 0.31 | 325.3 | 48.017 | 3.22 | 48.77 | 3.95 | 613.10 |
| 23 | 96 | 84.45 | 0.54 | 191.4 | 49.246 | 3.21 | 50.52 | 3.90 | 610.97 |
| 23 | 97 | 69.37 | 0.16 | 690.8 | 60.700 | 3.17 | 61.17 | 3.77 | 618.56 |
| 23 | 98 | 67.58 | 0.34 | 332.6 | 62.382 | 3.17 | 63.48 | 3.73 | 615.15 |
| 23 | 99 | 76.67 | 0.31 | 340.5 | 54.586 | 3.19 | 55.21 | 3.85 | 617.91 |
| 23 | 100 | 74.62 | 0.00 | 0.00 | 56.178 | 3.19 | 56.21 | 3.85 | 622.06 |
| 23 | 101 | 69.41 | 0.00 | 0.00 | 60.648 | 3.17 | 60.47 | 3.80 | 623.70 |
| 23 | 102 | 69.16 | 0.57 | 197.9 | 60.880 | 3.17 | 62.06 | 3.74 | 618.91 |

SYSTEM NORMALIZED

CAPPED

3/28/99

| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after transfer of qtz/clino, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer+acid added, g |
|---------------|--------------------|--|-----------------------------------|--|--|
| QC20U-pH 3.0 | 13.6579 | 21.1117 | 24.1493 | 29.9928 | 52.4711 |
| QC20U-pH 3.5 | 13.6836 | 23.5961 | 26.6179 | 32.2587 | 50.4958 |
| QC20U-pH 4.0 | 13.5571 | 23.4823 | 26.4956 | 32.4115 | 51.0262 |
| QC20U-pH 4.25 | 13.7629 | 23.2469 | 26.2554 | 31.9782 | 51.0244 |
| QC20U-pH 4.5 | 13.6793 | 23.8224 | 26.8193 | 32.6648 | 50.2929 |
| QC20U-pH 4.75 | 13.6855 | 21.9358 | 24.9351 | 30.5193 | 51.3388 |
| QC20U-pH 5.0 | 13.3224 | 22.034 | 25.0324 | 31.2063 | 50.9743 |
| QC20U-pH 5.25 | 13.6969 | 21.8724 | 24.868 | 30.9211 | 52.052 |
| QC20U-pH 5.5 | 13.6947 | 21.5299 | 24.5445 | 30.3507 | 52.024 |
| QC20U-pH 5.75 | 13.687 | 21.535 | 24.5322 | 30.3727 | 52.5638 |
| QC20U-pH 6.0 | 13.8746 | 23.8253 | 26.8229 | 32.4456 | 50.106 |
| QC20U-pH 6.25 | 13.589 | 24.0352 | 27.0399 | 32.8927 | 49.54 |
| QC20U-pH 6.5 | 13.7067 | 23.2027 | 26.1964 | 32.0138 | 50.5919 |
| QC20U-pH 6.75 | 13.6713 | 21.5748 | 24.5746 | 30.4269 | 52.6944 |
| QC20U-pH 7.0 | 13.6627 | 19.5214 | 22.5173 | 28.8202 | 54.64 |
| QC20U-pH 7.25 | 13.686 | 22.1571 | 25.1508 | 30.9484 | 51.2734 |
| QC20U-pH 7.5 | 13.7966 | 22.2487 | 25.2563 | 31.158 | 51.7667 |
| QC20U-pH 7.75 | 13.411 | 22.0282 | 25.0163 | 31.1785 | 51.3523 |
| QC20U-pH 8.0 | 13.6952 | 22.9669 | 25.9564 | 31.8205 | 50.7663 |
| QC20U-pH 8.25 | 13.7994 | 22.406 | 25.3898 | 31.0422 | 51.5076 |
| QC20U-pH 8.5 | 13.6714 | 21.9039 | 24.8946 | 30.7565 | 51.612 |
| QC20U-pH 8.75 | 13.3873 | 20.2339 | 23.2236 | 29.5296 | 53.0928 |
| QC20U-pH 9.0 | 13.6398 | 20.9319 | 23.9196 | 30.2069 | 53.1431 |
| QC20U-pH 9.25 | 13.6404 | 23.3224 | 26.2996 | 32.2381 | 49.9017 |
| QC20U-pH 9.5 | 13.6968 | 25.1003 | 25.1003 | 31.1298 | 53.4168 |

1

| 3/30/99 | | PP tubes | | Desorption | | PC containers | | Desorption | |
|----------------|---------------|----------------------|--|---------------|--|----------------------|--|------------|--|
| Sample Name | wt. of vial,g | wt. Of vial + sample | | wt. of vial,g | | wt. Of vial + sample | | | |
| QC20U-pH 3a | 7.9776 | 8.4774 | | 7.9153 | | 8.4181 | | | |
| QC20U-pH 3b | 7.9754 | 8.4764 | | 7.8851 | | 8.3886 | | | |
| QC20U-pH 3.5a | 7.8732 | 8.3705 | | 7.8636 | | 8.3639 | | | |
| QC20U-pH 3.5b | 7.8578 | 8.3582 | | 7.8532 | | 8.3528 | | | |
| QC20U-pH 4a | 7.9206 | 8.4213 | | 7.8877 | | 8.3893 | | | |
| QC20U-pH 4b | 7.8551 | 8.3584 | | 7.8693 | | 8.3727 | | | |
| QC20U-pH 4.25a | 7.8029 | 8.3027 | | 7.8835 | | 8.3841 | | | |
| QC20U-pH 4.25b | 7.8888 | 8.3905 | | 7.868 | | 8.3665 | | | |
| QC20U-pH 4.5a | 7.8256 | 8.3256 | | 7.8843 | | 8.3812 | | | |
| QC20U-pH 4.5b | 7.8675 | 8.3697 | | 7.8443 | | 8.341 | | | |
| QC20U-pH 4.75a | 7.9036 | 8.4043 | | 7.8888 | | 8.3919 | | | |
| QC20U-pH4.75b | 7.978 | 8.4808 | | 7.809 | | 8.3113 | | | |
| QC20U-pH 5a | 7.9166 | 8.4183 | | 7.7824 | | 8.2841 | | | |
| QC20U-pH 5b | 7.8648 | 8.3665 | | 7.8018 | | 8.3045 | | | |
| QC20U-pH 5.25a | 7.9196 | 8.4189 | | 7.8645 | | 8.366 | | | |
| QC20U-pH 5.25b | 7.8859 | 8.3875 | | 7.8339 | | 8.335 | | | |
| QC20U-pH 5.5a | 7.95 | 8.4452 | | 7.9096 | | 8.4123 | | | |
| QC20U-pH 5.5b | 7.89 | 8.3917 | | 7.8415 | | 8.3437 | | | |
| QC20U-pH 5.75a | 7.8895 | 8.3893 | | 7.8817 | | 8.385 | | | |
| QC20U-pH 5.75b | 7.879 | 8.3784 | | 7.8903 | | 8.3931 | | | |
| QC20U-pH 6a | 7.8322 | 8.3288 | | 7.8689 | | 8.3712 | | | |
| QC20U-pH 6b | 7.8904 | 8.3878 | | 7.8639 | | 8.3658 | | | |
| QC20U-pH 6.25a | 7.8364 | 8.3327 | | 7.8378 | | 8.3403 | | | |
| QC20U-pH 6.25a | 7.8422 | 8.3402 | | 7.7491 | | 8.2533 | | | |
| QC20U-pH 6.5a | 7.8737 | 8.3672 | | 7.8525 | | 8.3555 | | | |
| QC20U-pH 6.5b | 7.814 | 8.3087 | | 7.8601 | | 8.3612 | | | |
| QC20U-pH 6.75a | 7.9318 | 8.4219 | | 7.8387 | | 8.3414 | | | |
| QC20U-pH 6.75b | 7.9091 | 8.4015 | | 7.8851 | | 8.3867 | | | |
| QC20U-pH 7a | 7.9006 | 8.397 | | 7.9229 | | 8.422 | | | |
| QC20U-pH 7b | 7.859 | 8.3559 | | 7.8487 | | 8.35 | | | |
| QC20U-pH 7.25a | 7.8189 | 8.3141 | | 7.8491 | | 8.3485 | | | |
| QC20U-pH 7.25b | 7.8674 | 8.3631 | | 7.8975 | | 8.3986 | | | |
| QC20U-pH 7.5a | 7.8524 | 8.3449 | | 7.8796 | | 8.3787 | | | |
| QC20U-pH 7.5b | 7.8618 | 8.3557 | | 7.8869 | | 8.3886 | | | |
| QC20U-pH 7.75a | 7.8679 | 8.3637 | | 7.8694 | | 8.3702 | | | |
| QC20U-pH 7.75b | 7.9125 | 8.4077 | | 7.8326 | | 8.3343 | | | |
| QC20U-pH 8a | 7.9028 | 8.3987 | | 7.8867 | | 8.3866 | | | |
| QC20U-pH 8b | 7.8179 | 8.3148 | | 7.8311 | | 8.3325 | | | |
| QC20U-pH 8.25a | 7.8378 | 8.3317 | | 7.8895 | | 8.3887 | | | |
| QC20U-pH 8.25b | 7.8274 | 8.3232 | | 7.9532 | | 8.4535 | | | |
| QC20U-pH 8.5a | 7.8546 | 8.3485 | | 7.8664 | | 8.3656 | | | |
| QC20U-pH 8.5b | 7.91 | 8.4068 | | 7.8601 | | 8.3603 | | | |
| QC20U-pH 8.75a | 7.8967 | 8.3904 | | 7.886 | | 8.3862 | | | |
| QC20U-pH 8.75b | 7.8873 | 8.3837 | | 7.887 | | 8.3861 | | | |
| QC20U-pH 9a | 7.9069 | 8.404 | | 7.9045 | | 8.4025 | | | |
| QC20U-pH 9b | 7.8947 | 8.3929 | | 7.8399 | | 8.339 | | | |
| QC20U-pH 9.25a | 7.8643 | 8.3599 | | 7.8425 | | 8.3407 | | | |
| QC20U-pH 9.25 | 7.8827 | 8.3786 | | 7.8147 | | 8.3155 | | | |

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CAPPED

| 3/29/99 | | 3/30/99 | | 3/29/99 | | 3/30/99 | |
|---------------|-------------------------------|------------------------------|---------------------------------|--------------------------------|---------------|-------------------------------|------------------------------|
| Sample Name | Container wt. before sampling | Container wt. after sampling | wt. Of PP tubes before sampling | wt. Of PP tubes after sampling | Sample Name | Container wt. before sampling | Container wt. after sampling |
| QC20U-pH 3.0 | 52.3794 | 51.3561 | 23.9478 | 22.9421 | QC20U-pH 3.0 | 52.3794 | 51.3561 |
| QC20U-pH 3.5 | 50.4439 | 49.4305 | 26.5012 | 25.4813 | QC20U-pH 3.5 | 50.4439 | 49.4305 |
| QC20U-pH 4.0 | 50.9666 | 49.9518 | 26.3068 | 25.298 | QC20U-pH 4.0 | 50.9666 | 49.9518 |
| QC20U-pH 4.25 | 50.9113 | 49.8988 | 26.0634 | 25.0531 | QC20U-pH 4.25 | 50.9113 | 49.8988 |
| QC20U-pH 4.5 | 50.2167 | 49.2038 | 26.1856 | 25.1816 | QC20U-pH 4.5 | 50.2167 | 49.2038 |
| QC20U-pH4.75 | 51.2173 | 50.2003 | 24.8772 | 23.8645 | QC20U-pH4.75 | 51.2173 | 50.2003 |
| QC20U-pH 5.0 | 50.6345 | 49.6016 | 24.8633 | 23.8492 | QC20U-pH 5.0 | 50.6345 | 49.6016 |
| QC20U-pH 5.25 | 51.9581 | 50.9442 | 24.7697 | 23.7637 | QC20U-pH 5.25 | 51.9581 | 50.9442 |
| QC20U-pH 5.5 | 51.9588 | 50.9462 | 24.1737 | 23.1718 | QC20U-pH 5.5 | 51.9588 | 50.9462 |
| QC20U-pH 5.75 | 52.4837 | 51.4675 | 24.3296 | 23.3242 | QC20U-pH 5.75 | 52.4837 | 51.4675 |
| QC20U-pH 6.0 | 50.0028 | 48.9883 | 26.615 | 25.6064 | QC20U-pH 6.0 | 50.0028 | 48.9883 |
| QC20U-pH 6.25 | 49.4183 | 48.4002 | 26.9494 | 25.9439 | QC20U-pH 6.25 | 49.4183 | 48.4002 |
| QC20U-pH 6.5 | 50.4808 | 49.4655 | 26.0436 | 25.0448 | QC20U-pH 6.5 | 50.4808 | 49.4655 |
| QC20U-pH 6.75 | 52.5911 | 51.5758 | 24.4334 | 23.4442 | QC20U-pH 6.75 | 52.5911 | 51.5758 |
| QC20U-pH 7.0 | 54.505 | 53.4909 | 22.3806 | 21.3785 | QC20U-pH 7.0 | 54.505 | 53.4909 |
| QC20U-pH 7.25 | 51.1524 | 50.1426 | 25.0402 | 24.0417 | QC20U-pH 7.25 | 51.1524 | 50.1426 |
| QC20U-pH 7.5 | 51.6927 | 50.6829 | 25.0706 | 24.0744 | QC20U-pH 7.5 | 51.6927 | 50.6829 |
| QC20U-pH 7.75 | 51.1989 | 50.1828 | 24.8501 | 23.8518 | QC20U-pH 7.75 | 51.1989 | 50.1828 |
| QC20U-pH 8.0 | 50.6826 | 49.6729 | 25.7261 | 24.7265 | QC20U-pH 8.0 | 50.6826 | 49.6729 |
| QC20U-pH 8.25 | 51.4065 | 50.3965 | 25.0435 | 24.0478 | QC20U-pH 8.25 | 51.4065 | 50.3965 |
| QC20U-pH 8.5 | 51.515 | 50.5058 | 24.6534 | 23.6563 | QC20U-pH 8.5 | 51.515 | 50.5058 |
| QC20U-pH 8.75 | 52.9851 | 51.9742 | 22.8822 | 21.8864 | QC20U-pH 8.75 | 52.9851 | 51.9742 |
| QC20U-pH 9.0 | 53.0569 | 52.0497 | 23.8875 | 22.8878 | QC20U-pH 9.0 | 53.0569 | 52.0497 |
| QC20U-pH 9.25 | 49.8185 | 48.809 | 26.1015 | 25.1059 | QC20U-pH 9.25 | 49.8185 | 48.809 |

01 Apr 99 13:32

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

Pag
User :

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 2S% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 19.92 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.47 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.41 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|----------------------|--------|-------------|---------------|--------------|-------------|
| P# 1 | 999.99 | 19.92 1.42 | 3.465 3.40 | 29.41 1.17 | 145.25 |
| 23 2 | 8.00 | 3.33 102.7 | 552.035 3.02 | 556.84 3.08 | 607.54 Std. |
| 23 3 | 8.11 | 2.52 132.4 | 544.747 3.02 | 551.60 3.07 | 608.99 |
| 23 4 | 8.18 | 3.92 87.43 | 539.933 3.02 | 548.22 3.07 | 607.30 |
| 23 5 | 7.91 | 3.85 90.41 | 558.735 3.02 | 564.77 3.07 | 600.36 |
| 2314 MISSING TUBE(S) | | | | | |
| 20 | 6.70 | 1.42 251.6 | 660.863 3.01 | 661.63 3.07 | 596.53 |
| 23 21 | 6.72 | 4.19 90.72 | 657.845 3.02 | 664.63 3.06 | 595.64 |
| 23 22 | 4.68 | 26.02 24.11 | 946.321 3.01 | 973.15 3.01 | 563.96 |
| 23 23 | 4.50 | 6.75 72.29 | 984.535 3.01 | 991.03 3.04 | 581.98 |
| 23 24 | 5.29 | 5.41 81.06 | 838.123 3.01 | 843.74 3.05 | 580.53 |
| 23 25 | 5.41 | 3.19 130.1 | 819.456 3.01 | 823.27 3.05 | 586.04 |
| 23 26 | 3.30 | 4.63 118.1 | 1343.202 3.01 | 1348.47 3.03 | 586.00 |
| 23 27 | 3.23 | 1.13 451.6 | 1373.315 3.01 | 1374.92 3.03 | 592.15 |
| 23 28 | 3.13 | 3.72 147.9 | 1417.621 3.01 | 1421.70 3.03 | 586.05 |
| 23 29 | 3.07 | 3.86 144.4 | 1444.743 3.01 | 1449.41 3.03 | 588.64 |
| 23 30 | 2.89 | 4.65 125.6 | 1538.749 3.00 | 1543.25 3.02 | 588.21 |
| 23 31 | 2.83 | 8.70 73.17 | 1566.853 3.01 | 1579.42 3.02 | 589.92 |
| 23 32 | 2.70 | 8.97 73.01 | 1645.794 3.00 | 1655.03 3.02 | 589.55 |
| 23 33 | 2.63 | 7.08 90.64 | 1686.649 3.01 | 1693.40 3.02 | 589.29 |
| 23 34 | 2.59 | 1.32 435.9 | 1713.910 3.00 | 1717.69 3.02 | 592.58 |
| 23 35 | 2.67 | 7.05 90.30 | 1661.703 3.01 | 1669.84 3.02 | 589.67 |
| 23 36 | 2.65 | 8.38 78.05 | 1674.648 3.01 | 1681.91 3.02 | 592.16 |
| 23 37 | 2.69 | 8.70 75.02 | 1648.579 3.01 | 1657.95 3.02 | 588.48 |
| 23 38 | 2.57 | 10.82 63.99 | 1728.052 3.00 | 1737.90 3.02 | 590.12 |
| 23 39 | 2.59 | 10.58 64.92 | 1712.751 3.01 | 1728.12 3.01 | 548.54 |
| 23 40 | 3.13 | 0.00 0.00 | 1419.538 3.00 | 1421.39 3.03 | 591.21 |
| 23 41 | 3.09 | 10.50 59.82 | 1440.548 3.00 | 1455.05 3.01 | 588.01 |
| 23 42 | 3.13 | 3.08 176.1 | 1423.052 3.00 | 1427.14 3.02 | 594.96 |
| 23 43 | 3.17 | 7.52 78.30 | 1399.689 3.01 | 1408.44 3.02 | 590.21 |
| 23 44 | 2.85 | 9.55 67.39 | 1559.693 3.00 | 1571.64 3.02 | 590.48 |
| 23 45 | 2.80 | 4.01 146.0 | 1583.678 3.01 | 1588.09 3.03 | 589.37 |
| 23 46 | 2.44 | 2.21 272.7 | 1820.305 3.00 | 1823.46 3.02 | 594.13 |

| | | | | | |
|-------|------|-------------|---------------|--------------|--------|
| 23 47 | 2.43 | 5.59 116.0 | 1828.634 3.00 | 1835.20 3.02 | 591.60 |
| 23 48 | 1.95 | 11.36 70.55 | 2286.279 3.00 | 2299.82 3.01 | 588.10 |
| 23 49 | 2.02 | 3.35 203.0 | 2202.970 3.00 | 2205.24 3.02 | 590.75 |
| 23 50 | 2.54 | 4.10 150.3 | 1750.866 3.00 | 1755.63 3.02 | 592.18 |
| 23 51 | 2.60 | 3.54 169.8 | 1711.535 3.00 | 1713.66 3.02 | 590.98 |
| 23 52 | 2.68 | 9.18 71.83 | 1658.102 3.00 | 1667.23 3.02 | 589.18 |
| 23 53 | 2.66 | 4.14 145.5 | 1673.979 3.00 | 1679.23 3.02 | 595.08 |
| 23 54 | 2.63 | 8.98 73.92 | 1686.269 3.01 | 1697.96 3.02 | 590.20 |

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U-233 3% 2 sigma

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|-------|------|-------------|---------------|--------------|--------|
| 23 55 | 2.61 | 8.82 75.35 | 1705.347 3.00 | 1714.65 3.02 | 591.60 |
| 23 56 | 2.73 | 10.85 61.94 | 1628.769 3.00 | 1637.25 3.02 | 590.45 |
| 23 57 | 2.82 | 8.09 77.96 | 1574.195 3.01 | 1584.77 3.02 | 590.27 |
| 23 58 | 2.68 | 10.68 63.35 | 1659.968 3.00 | 1671.71 3.01 | 590.23 |
| 23 59 | 2.56 | 8.20 80.87 | 1736.769 3.00 | 1746.37 3.02 | 590.63 |
| 23 60 | 2.41 | 10.79 66.24 | 1845.083 3.00 | 1860.21 3.01 | 590.11 |
| 23 61 | 2.44 | 8.77 78.28 | 1821.125 3.00 | 1832.47 3.01 | 591.26 |
| 23 62 | 2.39 | 14.39 52.70 | 1860.970 3.00 | 1877.28 3.01 | 590.07 |
| 23 63 | 2.35 | 7.74 88.73 | 1896.961 3.00 | 1902.93 3.01 | 590.53 |
| 23 64 | 2.27 | 11.36 65.41 | 1966.579 3.00 | 1981.60 3.00 | 589.27 |
| 23 65 | 2.21 | 4.51 147.5 | 2008.300 3.00 | 2013.57 3.02 | 589.26 |
| 23 66 | 3.06 | 4.92 116.0 | 1448.823 3.01 | 1454.90 3.03 | 590.72 |
| 23 67 | 2.99 | 3.16 176.2 | 1486.501 3.00 | 1488.98 3.03 | 591.58 |

235. MISSING TUBE(S)

| | | | | | |
|-------|-------|------------|--------------|-------------|--------|
| 73 | 10.89 | 0.19 1438 | 404.799 3.03 | 407.68 3.11 | 597.79 |
| 23 74 | 10.57 | 3.07 96.54 | 417.349 3.02 | 420.63 3.10 | 590.37 |
| 23 75 | 17.37 | 1.73 130.4 | 252.378 3.04 | 254.35 3.18 | 593.60 |
| 23 76 | 17.42 | 0.00 0.00 | 251.644 3.04 | 249.00 3.21 | 600.63 |
| 23 77 | 14.64 | 1.05 229.6 | 300.223 3.03 | 301.39 3.16 | 594.20 |
| 23 78 | 14.39 | 1.97 126.0 | 305.639 3.03 | 308.88 3.14 | 590.33 |
| 23 79 | 32.69 | 0.24 668.0 | 132.509 3.08 | 133.39 3.36 | 595.78 |
| 23 80 | 33.60 | 0.94 169.8 | 128.827 3.08 | 130.44 3.35 | 590.31 |
| 23 81 | 54.77 | 0.73 172.7 | 77.729 3.14 | 78.86 3.59 | 591.79 |
| 23 82 | 53.34 | 1.10 117.4 | 79.906 3.13 | 81.57 3.56 | 589.08 |
| 23 83 | 35.78 | 0.51 301.3 | 120.766 3.09 | 121.54 3.39 | 595.17 |
| 23 84 | 36.43 | 1.98 79.42 | 118.550 3.09 | 120.90 3.37 | 586.38 |
| 23 85 | 66.40 | 1.15 101.1 | 63.493 3.17 | 65.09 3.70 | 584.94 |
| 23 86 | 65.60 | 0.96 120.7 | 64.309 3.17 | 65.16 3.72 | 587.25 |
| 23 87 | 57.38 | 1.29 96.82 | 74.018 3.14 | 75.97 3.60 | 585.40 |
| 23 88 | 55.90 | 0.94 133.6 | 76.034 3.14 | 77.21 3.61 | 592.75 |
| 23 89 | 45.23 | 1.46 96.18 | 94.788 3.11 | 96.48 3.48 | 590.48 |
| 23 90 | 46.60 | 0.00 0.00 | 91.943 3.12 | 91.21 3.55 | 603.06 |
| 23 91 | 46.81 | 0.29 464.8 | 91.472 3.12 | 91.50 3.53 | 597.16 |
| 23 92 | 48.32 | 0.57 234.5 | 88.526 3.12 | 89.36 3.53 | 593.72 |
| 23 93 | 52.10 | 0.00 0.00 | 81.852 3.13 | 81.87 3.59 | 594.98 |
| 23 94 | 49.68 | 0.15 878.4 | 86.008 3.12 | 87.23 3.54 | 591.34 |
| 23 95 | 66.31 | 0.00 0.00 | 63.554 3.17 | 63.02 3.79 | 598.89 |
| 23 96 | 64.56 | 0.56 208.5 | 65.370 3.16 | 66.61 3.70 | 590.84 |
| 23 97 | 74.59 | 0.00 0.00 | 56.127 3.19 | 55.79 3.88 | 602.65 |
| 23 98 | 74.11 | 0.00 0.00 | 56.500 3.19 | 56.46 3.86 | 599.29 |
| 23 99 | 83.37 | 0.72 143.1 | 49.840 3.22 | 50.45 3.94 | 587.77 |

| | | | | | | | | | |
|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 100 | 83.86 | 0.14 | 742.9 | 49.528 | 3.22 | 50.28 | 3.94 | 594.72 |
| 23 | 101 | 62.01 | 0.00 | 0.00 | 68.201 | 3.16 | 68.25 | 3.71 | 605.07 |
| 23 | 102 | 62.26 | 0.85 | 140.3 | 67.945 | 3.16 | 68.77 | 3.69 | 591.63 |
| 23 | 103 | 86.32 | 0.53 | 192.3 | 48.018 | 3.23 | 49.04 | 3.95 | 591.47 |
| 23 | 104 | 84.13 | 0.60 | 172.4 | 49.358 | 3.22 | 50.27 | 3.93 | 591.96 |
| 23 | 105 | 68.42 | 0.56 | 203.1 | 61.545 | 3.17 | 62.27 | 3.76 | 590.41 |
| 23 | 106 | 70.63 | 0.00 | 0.00 | 59.454 | 3.18 | 58.20 | 3.87 | 606.22 |
| 23 | 107 | 71.89 | 0.00 | 0.00 | 58.366 | 3.18 | 58.21 | 3.84 | 598.71 |
| 23 | 108 | 70.11 | 0.00 | 0.00 | 59.921 | 3.18 | 59.62 | 3.82 | 605.45 |
| 23 | 109 | 70.87 | 0.16 | 693.5 | 59.241 | 3.18 | 59.72 | 3.80 | 598.10 |
| 23 | 110 | 72.64 | 0.10 | 1129 | 57.713 | 3.19 | 57.85 | 3.84 | 599.95 |
| 23 | 111 | 74.17 | 0.74 | 148.6 | 56.451 | 3.19 | 58.41 | 3.77 | 591.34 |
| 23 | 112 | 73.93 | 0.29 | 375.9 | 56.646 | 3.19 | 57.07 | 3.84 | 598.85 |
| 23 | 113 | 92.62 | 0.59 | 165.5 | 44.538 | 3.24 | 45.72 | 4.01 | 593.29 |
| 23 | 114 | 91.99 | 0.10 | 939.9 | 44.845 | 3.24 | 45.76 | 4.02 | 597.58 |
| 23 | 115 | 41.88 | 1.26 | 115.1 | 102.648 | 3.10 | 104.28 | 3.44 | 591.62 |

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U-233 3% 2 sigma

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|----|-----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 116 | 42.04 | 0.00 | 0.00 | 102.315 | 3.10 | 101.60 | 3.49 | 606.49 |
| 23 | 117 | 88.34 | 0.66 | 152.5 | 46.841 | 3.23 | 48.28 | 3.95 | 590.59 |
| 23 | 118 | 86.32 | 0.27 | 370.3 | 48.064 | 3.22 | 48.87 | 3.96 | 600.34 |
| 23 | 119 | 53.30 | 1.11 | 115.8 | 79.912 | 3.13 | 81.64 | 3.56 | 593.97 |
| 23 | 120 | 53.79 | 0.98 | 131.0 | 79.153 | 3.14 | 80.29 | 3.58 | 592.55 |

200
capped
containers
desorp.Table CQ20U: Estimated solution pH and volumes of HNO₃ or NaOH solution needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with quartz and clinoptilolite (35ml).

| Estimated solution pH | Volume of HNO ₃ added, ml | Volume of NaOH added, ml | Molarity of HNO ₃ to use | Molarity of NaOH to use |
|-----------------------|--------------------------------------|--------------------------|-------------------------------------|-------------------------|
| 3 | 0.080 | 0.060 | 0.01 | 0.01 |
| 3.5 | 0.205 | 0.205 | 0.01 | 0.01 |
| 4 | 0.385 | 0.375 | 0.01 | 0.01 |
| 4.25 | 0.190 | 0.190 | 0.01 | 0.01 |
| 4.50 | 0.09 | 0.125 | 0.01 | 0.01 |
| 4.75 | 0.150 | 0.050 | 0.01 | 0.01 |
| 5 | 0.200 | 0.075 | 0.01 | 0.01 |
| 5.25 | 0.225 | 0.080 | 0.01 | 0.01 |
| 5.5 | 0.250 | 0.125 | 0.01 | 0.01 |
| 5.75 | 0.275 | 0.150 | 0.01 | 0.01 |
| 6 | 0.300 | 0.175 | 0.01 | 0.01 |
| 6.25 | 0.325 | 0.200 | 0.01 | 0.01 |
| 6.5 | 0.350 | 0.225 | 0.01 | 0.01 |
| 6.75 | 0.375 | 0.250 | 0.01 | 0.01 |
| 7 | 0.400 | 0.275 | 0.01 | 0.01 |
| 7.25 | 0.425 | 0.300 | 0.01 | 0.01 |
| 7.5 | 0.450 | 0.325 | 0.01 | 0.01 |
| 7.75 | 0.475 | 0.350 | 0.01 | 0.01 |
| 8 | 0.500 | 0.375 | 0.01 | 0.01 |
| 8.25 | 0.525 | 0.400 | 0.01 | 0.01 |
| 8.5 | 0.550 | 0.425 | 0.01 | 0.01 |
| 8.75 | 0.575 | 0.450 | 0.01 | 0.01 |
| 9 | 0.600 | 0.475 | 0.01 | 0.01 |
| 9.25 | 0.625 | 0.500 | 0.01 | 0.01 |
| 9.5 | 0.650 | 0.525 | 0.01 | 0.01 |
| 9.75 | 0.675 | 0.550 | 0.01 | 0.01 |
| 10 | 0.700 | 0.575 | 0.01 | 0.01 |
| QC100-U | 0.100 | | 50% HNO ₃ | |

Experimental Procedure for experiment - U sorption onto quartz at 25° C.

Objective: To investigate the characteristics of U sorption onto quartz as a function of pH.

Initial conditions:

- $\sum U = 50$ ppb (2.14×10^{-7})
- Capped containers
- pH range 3.0-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 0.000 g
- M/V = 28.57 g/L

Equipment:

- Orion 920A pH meter and combination pH electrode
- Gyratory shaker
- Glove box
- Fisher Marathon 21K centrifuge
- Mettler PM4600 and AE240 balances
- Packard 2500 TR/AB LSC
- Eppendorf micropipettors and tips
- Oxford pipettes and tips
- Repipettor for transfer of scintillation cocktail

Supplies:

- W510 * UC * RL * RFe * HL * ≤ 325 mesh AJ
- 50 ppb ²³³U stock solution
- polycarbonate centrifuge tubes with caps (50 ml capacity)
- polypropylene centrifuge tubes with caps (50 ml capacity)
- pH buffer solutions
- Ultima-Gold liquid scintillation cocktail
- 7 ml scintillation vials
- reagent grade NaOH (lot 976631)
- concentrated HNO₃ (lot 983546)
- HNO₃ and NaHCO₃ or NaOH degassed stock solutions at various concentrations
- weighing paper/boats
- deionized ultrapure water, DH₂O
- teflon beaker

Procedure:

- Prepare separate 50 ppb U solutions for sorption experiments.
 - On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using degassed 0.1m NaNO₃ stock solution.
 - Cap and label bottle accordingly.
- Transfer (2.14×10^{-7}) 50 ppb ²³³U stock solution to experimental containers.
 - Label 26 polycarbonate centrifuge tubes (50 ml capacity) CQU-pHi (where i is the approximate pH of each solution, see Table CQ0U).
 - Label 1 polycarbonate centrifuge tube CQ0U-IU.
 - Weigh and record the weight of each container.
 - About 1L of ²³³U solution will be required for this experiment.
 - Add ~35 g of the 50 ppb ²³³U stock solution to each container. *Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.*

- Record weight of each container.
- B. Add quartz to sample containers.
 - Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the CQ20-U-IU container.
 - Record weight of each sample container after addition of quartz .
- Adjust pH of each solution by adding HNO₃ or NaHCO₃.
 - Add the volume of HNO₃ or NaOH to each experimental container as listed in Table CQ0U. Take care to add HNO₃ or NaOH in the concentrations specified in Table CQ0U.
 - For sample CQ0U-IU (not listed in Table CQ0U) add 100 µl of 50/50 V/V HNO₃ to the sample container.
 - Weigh and record the weight of each sample container.
- Sample CQ0U-IU for initial [U].
 - Label two 7 ml LSC vials as CQ0U-IUa and CQ0U-IUb.
 - Add 0.5 ml of 0.02 M HNO₃ to each vial.
 - Record weight of each vial.
 - Transfer 0.5 ml of solution from CQ20-U-IU to each sample vial.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of container CQ0U-IU
- Wait for equilibrium.
 - Place caps on sample containers but do not tighten the caps.
 - Place containers inside the glove box. Tighten the caps after 2 days.
 - Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate for at least 10 days.
- Sample experimental solutions for [U] and pH.
 - For each sample solution label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ0U-pH3b).
 - Add 0.5 ml of 0.02 M HNO₃ to each vial.
 - Record weight of each vial.
 - Record weight of each sample container.
 - From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of each sample container.
 - Measure pH of each experimental solution.
 - Record weight of each sample container after pH measurement.
- Transfer quartz for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
 - Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
 - For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., CQ0dU-pH3 for sample container CQ0U-pH3).
 - Record weight of PP test tubes.
 - Using an Eppendorf or Oxford pipette, withdraw quartz from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
 - Record weights of sample containers and PP test tubes.
 - Add approximately 3 ml of 0.1 M HNO₃ to both experimental containers and PP test tubes and mix thoroughly.
 - Record weights of sample containers and PP test tubes.
 - Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.
- C. Sample solutions for [U].
- For each sample container and PP test tube label two 7 ml LSC vials (e.g., CQ0U-pH3a and CQ02U-pH3b).
 - Add 0.5 ml of 0.02 M HNO₃ to each vial.
 - Record weight of each vial.
 - Record weight of each sample container and PP test tube.
 - From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
 - Record weight of each vial.
 - Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
 - Record weight of each sample container and PP test tube.

Table CQ0U: Estimated solution pH and volumes of HNO3 or NaOH solutions needed for adjustment of pH in 0.1 m NaNO3 solutions with 50 ppb U in contact with qz/clino

| Estimated solution pH | Volume of HNO3 added, ml | Actual Vol. of HNO3 added, ml | | Molarity of HNO3 to use |
|-----------------------|--------------------------|-------------------------------|--------------------|-------------------------|
| 3 | 0.045 | 0.060 | | 1 |
| 3.5 | 0.143 | 0.205 | 0.020 1M NaOH | 0.1 |
| 4 | 0.140 | 0.0375 | | 0.01 |
| 4.25 | 0.014 | 0.190 | | 0.01 |
| Estimated solution pH | Volume of NaOH added, ml | Actual Volume of NaOH added | | Molarity of NaOH to use |
| 4.5 | 0.09 | 0.025 | | 0.01 |
| 4.75 | 0.150 | 0.050 | 0.050 [0.01M NaOH] | 0.01 |
| 5 | 0.200 | 0.075 | 0.075 [0.01M NaOH] | 0.01 |
| 5.25 | 0.225 | 0.100 | 0.100 [0.01M NaOH] | 0.01 |
| 5.5 | 0.250 | 0.125 | | 0.01 |
| 5.75 | 0.275 | 0.150 | 0.075 [0.01M NaOH] | 0.01 |
| 6 | 0.300 | 0.175 | | 0.01 |
| 6.25 | 0.325 | 0.200 | | 0.01 |
| 6.5 | 0.350 | 0.225 | | 0.01 |
| 6.75 | 0.375 | | 0.050 0.01M HNO3 | 0.01 |
| 7 | 0.400 | | 0.055 0.01M HNO3 | 0.01 |
| 7.25 | 0.050 | | | 0.05 |
| 7.5 | 0.075 | 0.110 | 0.015 0.05M HNO3 | 0.05 |
| 7.75 | 0.100 | 0.125 | 0.020 0.05M HNO3 | 0.05 |
| 8 | 0.125 | 0.150 | 0.025 0.05M HNO3 | 0.05 |
| 8.25 | 0.150 | 0.175 | 0.030 0.05M HNO3 | 0.05 |
| 8.5 | 0.065 | 0.090 | 0.005 0.1M HNO3 | 0.1 |
| 8.75 | 0.100 | 0.100 | 0.015 0.1M HNO3 | 0.1 |
| 9 | 0.125 | 0.125 | 0.010 0.1M HNO3 | 0.1 |
| 9.25 | 0.150 | 0.150 | 0.010 0.1M HNO3 | 0.1 |
| 9.5 | 0.175 | | | 0.1 |
| QCOU-IU | 0.100 | | | 50% HNO3 |

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2/18/99

| Sample Name | Wt. Of container g g |
|--------------|---------------------------------|
| QCOU-pH 3.5 | 22.2395 |
| QCOU-pH 4.0 | 22.1946 |
| QCOU-pH 4.25 | 22.1712 |
| QCOU-pH 4.5 | 22.2782 |
| QCOU-pH 4.75 | 22.1919 |
| QCOU-pH 5.0 | 22.3295 |
| QCOU-pH 5.25 | 22.1618 |
| QCOU-pH 5.5 | 22.4789 |
| QCOU-pH 5.75 | 22.0817 |
| QCOU-pH 6.0 | 22.1875 |
| QCOU-pH 6.25 | 22.09 |
| QCOU-pH 6.5 | 22.2926 |
| QCOU-pH 6.75 | 22.533 |
| QCOU-pH 7.0 | 22.1867 |
| QCOU-pH 7.25 | 22.4386 |
| QCOU-pH 7.5 | 22.1048 |
| QCOU-pH 7.75 | 22.2593 |
| QCOU-pH 8.0 | 22.2038 |
| QCOU-pH 8.25 | 22.1826 |
| QCOU-pH 8.5 | 22.2489 |
| QCOU-pH 8.75 | 22.934 |
| QCOU-pH 9.0 | 22.1506 |
| QCOU-pH 9.25 | 22.1944 |

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2/18/99

| Sample Name | Wt. Of container + quartz, g |
|--------------|------------------------------|
| | |
| QC0U-pH 3.5 | 23.2398 |
| QC0U-pH 4.0 | 23.1983 |
| QC0U-pH 4.25 | 23.1723 |
| QC0U-pH 4.5 | 23.2777 |
| QC0U-pH4.75 | 23.1931 |
| QC0U-pH 5.0 | 23.3331 |
| QC0U-pH 5.25 | 23.1644 |
| QC0U-pH 5.5 | 23.4796 |
| QC0U-pH 5.75 | 23.086 |
| QC0U-pH 6.0 | 23.1898 |
| QC0U-pH 6.25 | 23.0912 |
| QC0U-pH 6.5 | 23.2951 |
| QC0U-pH 6.75 | 23.5336 |
| QC0U-pH 7.0 | 23.1874 |
| QC0U-pH 7.25 | 23.4433 |
| QC0U-pH 7.5 | 23.1056 |
| QC0U-pH 7.75 | 23.2598 |
| QC0U-pH 8.0 | 23.2072 |
| QC0U-pH 8.25 | 23.1852 |
| QC0U-pH 8.5 | 23.2487 |
| QC0U-pH 8.75 | 22.8458 |
| QC0U-pH 9.0 | 23.1523 |
| QC0U-pH 9.25 | 23.1905 |

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2/18/99

| Sample Name | Wt. Of Container + quartz + U soln., g |
|--------------|--|
| | |
| QC0U-pH 3.5 | 58.3034 |
| QC0U-pH 4.0 | 58.2847 |
| QC0U-pH 4.25 | 58.1612 |
| QC0U-pH 4.5 | 58.295 |
| QC0U-pH4.75 | 58.2108 |
| QC0U-pH 5.0 | 58.3318 |
| QC0U-pH 5.25 | 58.1736 |
| QC0U-pH 5.5 | 58.5121 |
| QC0U-pH 5.75 | 58.0944 |
| QC0U-pH 6.0 | 58.2034 |
| QC0U-pH 6.25 | 58.1017 |
| QC0U-pH 6.5 | 58.2972 |
| QC0U-pH 6.75 | 58.544 |
| QC0U-pH 7.0 | 58.1963 |
| QC0U-pH 7.25 | 58.4628 |
| QC0U-pH 7.5 | 58.1138 |
| QC0U-pH 7.75 | 58.2504 |
| QC0U-pH 8.0 | 58.192 |
| QC0U-pH 8.25 | 58.1774 |
| QC0U-pH 8.5 | 58.2643 |
| QC0U-pH 8.75 | 57.8952 |
| QC0U-pH 9.0 | 58.1948 |
| QC0U-pH 9.25 | 58.2333 |

CAPPED

| Sample Name | Container Wt. After additions of HNO3 and NaOH., g |
|--------------|--|
| QC0U-pH 3.5 | 58.5032 |
| QC0U-pH 4.0 | 58.659 |
| QC0U-pH 4.25 | 58.3499 |
| QC0U-pH 4.5 | 58.3207 |
| QC0U-pH4.75 | 58.26 |
| QC0U-pH 5.0 | 58.4176 |
| QC0U-pH 5.25 | 58.2213 |
| QC0U-pH 5.5 | 58.6339 |
| QC0U-pH 5.75 | 58.2427 |
| QC0U-pH 6.0 | 58.3768 |
| QC0U-pH 6.25 | 58.3002 |
| QC0U-pH 6.5 | 58.5203 |
| QC0U-pH 6.75 | 58.9164 |
| QC0U-pH 7.0 | 58.5266 |
| QC0U-pH 7.25 | 58.5354 |
| QC0U-pH 7.5 | 58.2231 |
| QC0U-pH 7.75 | 58.3732 |
| QC0U-pH 8.0 | 58.3396 |
| QC0U-pH 8.25 | 58.3507 |
| QC0U-pH 8.5 | 58.3289 |
| QC0U-pH 8.75 | 57.9908 |
| QC0U-pH 9.0 | 58.317 |
| QC0U-pH 9.25 | 58.2781 |

CAPPED

| Sample Name | Container wt. before sampling & pH measurements, g | Measured pH | Container wt. after sampling & pH measurements, g |
|--------------|--|-------------|---|
| QC0U-pH 3.5 | 58.075 | 3.51 | 57.0306 |
| QC0U-pH 4.0 | 57.9595 | 3.15 | 56.9457 |
| QC0U-pH 4.25 | 57.9326 | 4.26 | 56.914 |
| QC0U-pH 4.5 | 57.7767 | 4.6 | 56.7784 |
| QC0U-pH4.75 | 57.8441 | 4.82 | 56.8248 |
| QC0U-pH 5.0 | 58.0761 | 5 | 57.048 |
| QC0U-pH 5.25 | 57.6051 | 4.68 | 56.6044 |
| QC0U-pH 5.5 | 58.1816 | 5.27 | 57.1639 |
| QC0U-pH 5.75 | 57.5609 | 5.46 | 56.4805 |
| QC0U-pH 6.0 | 56.6297 | 5.2 | 55.613 |
| QC0U-pH 6.25 | 57.6768 | 5.52 | 56.5839 |
| QC0U-pH 6.5 | 57.8916 | 5.84 | 56.8859 |
| QC0U-pH 6.75 | 58.4256 | 6.33 | 57.2239 |
| QC0U-pH 7.0 | 57.9073 | 6.29 | 56.7659 |
| QC0U-pH 7.25 | 57.8202 | 6.12 | 56.7209 |
| QC0U-pH 7.5 | 57.5984 | 6.88 | 56.5986 |
| QC0U-pH 7.75 | 57.9279 | 7.25 | 56.9169 |
| QC0U-pH 8.0 | 57.9922 | 7.66 | 57.0234 |
| QC0U-pH 8.25 | 57.8801 | 8.22 | 56.815 |
| QC0U-pH 8.5 | 57.6919 | 8.51 | 56.5514 |
| QC0U-pH 8.75 | 57.3205 | 8.75 | 56.2408 |
| QC0U-pH 9.0 | 57.5496 | 9.22 | 56.5143 |
| QC0U-pH 9.25 | 57.3943 | 9.47 | 56.3132 |

CAPPED

| Sample Name | wt. of vial | wt. Of vial + sample |
|----------------|-------------|----------------------|
| QC0U--pH 3.5a | 7.9047 | 8.3989 |
| QC0U-pH 3.5b | 7.8337 | 8.336 |
| QC0U--pH 4a | 7.8894 | 8.3894 |
| QC0U--pH 4b | 7.8698 | 8.3662 |
| QC0U--pH 4.25a | 7.9284 | 8.4209 |
| QC0U--pH 4.25b | 7.8604 | 8.3583 |
| QC0U-pH 4.5a | 7.8708 | 8.3678 |
| QC0U-pH 4.5b | 7.9443 | 8.4427 |
| QC0U-pH 4.75a | 7.8766 | 8.371 |
| QC0U-pH 4.75b | 7.9473 | 8.4489 |
| QC0U-pH 5a | 7.912 | 8.4069 |
| QC0U-pH 5b | 7.8722 | 8.3741 |
| QC0U-pH 5.25a | 7.8141 | 8.315 |
| QC0U-pH 5.25b | 7.908 | 8.4064 |
| QC0U-pH 5.5a | 7.839 | 8.3318 |
| QC0U-pH 5.5b | 7.8296 | 8.325 |
| QC0U-pH 5.75a | 7.7992 | 8.2945 |
| QC0U-pH 5.75b | 7.8397 | 8.3364 |
| QC0U-pH 6a | 7.8633 | 8.3582 |
| QC0U-pH 6b | 7.8851 | 8.3826 |
| QC0U-pH 6.25a | 7.8305 | 8.3308 |
| QC0U-pH 6.25b | 7.8993 | 8.4024 |
| QC0U-pH 6.5a | 7.855 | 8.3502 |
| QC0U-pH 6.5b | 7.9245 | 8.4146 |
| QC0U-pH 6.75a | 7.9003 | 8.3971 |
| QC0U-pH 6.75b | 7.9272 | 8.4299 |
| QC0U-pH 7a | 7.8597 | 8.3607 |
| QC0U-pH 7b | 7.8539 | 8.35 |
| QC0U-pH 7.25a | 7.8517 | 8.3483 |
| QC0U-pH 7.25b | 7.8686 | 8.3681 |
| QC0U-pH 7.5a | 7.8704 | 8.3703 |
| QC0U-pH 7.5b | 7.8491 | 8.3452 |
| QC0U-pH 7.75a | 7.842 | 8.3385 |
| QC0U-pH 7.75b | 7.8632 | 8.3572 |
| QC0U-pH 8a | 7.9015 | 8.3968 |
| QC0U-pH 8b | 7.8655 | 8.3627 |
| QC0U-pH 8.25a | 7.7939 | 8.2877 |
| QC0U-pH 8.25b | 7.9249 | 8.4192 |
| QC0U-pH 8.5a | 7.8491 | 8.3438 |
| QC0U-pH 8.5b | 7.9127 | 8.4092 |
| QC0U-pH 8.75a | 7.8663 | 8.3659 |
| QC0U-pH 8.75b | 7.8519 | 8.3482 |
| QC0U-pH 9a | 7.8034 | 8.2975 |
| QC0U-pH 9b | 7.8583 | 8.3592 |
| QC0U-pH 9.25a | 7.8371 | 8.3342 |
| QC0U-pH 9.25b | 7.8983 | 8.3943 |

QC0U-lua
QC0U-lub
QC0U-luc
QC0U-lud
QC0U-lue
QC0U-luf

20 Mar 99 09:28

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 2S% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 20.02 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.38 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.14 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

*Capped QC0U Sorption
Ext. 2nd out*

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|--------|------------|--------------|-------------|----------|
| P# 1 | 999.99 | 20.02 1.41 | 3.377 3.44 | 29.14 1.17 | 141.17 B |
| 23 2 | 8.33 | 2.19 149.8 | 530.236 3.02 | 534.61 3.08 | 614.57 |
| 23 3 | 8.29 | 3.26 103.1 | 532.691 3.02 | 537.69 3.08 | 611.92 |
| 23 4 | 8.52 | 5.21 66.23 | 518.454 3.02 | 526.03 3.07 | 610.42 |
| 23 5 | 8.37 | 5.55 63.22 | 527.806 3.02 | 534.30 3.07 | 607.91 |
| 23 6 | 8.56 | 2.41 134.9 | 516.132 3.02 | 518.76 3.09 | 617.34 |
| 23 7 | 8.47 | 3.12 106.3 | 522.007 3.02 | 525.05 3.08 | 613.55 |
| 23 8 | 9.30 | 1.59 192.3 | 475.440 3.02 | 477.96 3.09 | 615.63 |
| 23 9 | 9.11 | 0.00 0.00 | 484.548 3.02 | 485.35 3.10 | 622.40 |
| 23 10 | 9.85 | 2.92 104.8 | 447.790 3.02 | 452.38 3.09 | 612.53 |
| 23 11 | 10.07 | 2.22 134.3 | 438.430 3.02 | 440.67 3.10 | 617.49 |
| 23 12 | 11.34 | 2.38 118.8 | 388.598 3.03 | 393.26 3.11 | 617.22 |
| 23 13 | 11.43 | 1.15 237.5 | 385.424 3.03 | 387.83 3.12 | 615.72 |
| 23 14 | 10.45 | 1.42 203.4 | 421.886 3.02 | 424.07 3.11 | 611.96 |
| 23 15 | 10.45 | 1.32 217.7 | 422.078 3.02 | 424.64 3.10 | 617.09 |
| 23 16 | 16.50 | 2.65 89.22 | 265.956 3.04 | 270.38 3.15 | 611.24 |
| 23 17 | 15.68 | 2.30 104.4 | 280.105 3.04 | 285.47 3.14 | 614.63 |
| 23 18 | 18.19 | 0.76 283.6 | 241.098 3.04 | 243.10 3.19 | 613.81 |
| 23 19 | 17.76 | 2.78 82.04 | 247.017 3.04 | 251.66 3.16 | 612.91 |
| 23 20 | 16.56 | 1.66 139.0 | 265.101 3.04 | 268.39 3.16 | 615.16 |
| 23 21 | 15.99 | 2.74 87.57 | 274.609 3.04 | 279.43 3.15 | 612.11 |
| 23 22 | 22.03 | 2.18 93.14 | 198.348 3.05 | 202.18 3.21 | 612.55 |

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 23 | 21.73 | 2.25 | 90.74 | 201.133 | 3.05 | 205.47 | 3.20 | 612.41 |
| 23 | 24 | 30.89 | 1.12 | 150.0 | 140.521 | 3.07 | 142.96 | 3.31 | 612.91 |
| 23 | 25 | 29.34 | 2.03 | 86.48 | 148.123 | 3.07 | 152.76 | 3.27 | 617.41 |
| 23 | 26 | 27.27 | 1.87 | 96.91 | 159.586 | 3.06 | 162.65 | 3.27 | 613.84 |
| 23 | 27 | 26.91 | 1.31 | 137.6 | 161.803 | 3.06 | 164.43 | 3.27 | 613.36 |
| 23 | 28 | 29.85 | 0.75 | 225.5 | 145.568 | 3.07 | 148.35 | 3.30 | 611.59 |
| 23 | 29 | 29.40 | 2.84 | 62.96 | 147.779 | 3.07 | 151.95 | 3.27 | 607.02 |
| 23 | 30 | 37.76 | 0.98 | 154.8 | 114.314 | 3.09 | 116.78 | 3.38 | 614.90 |
| 23 | 31 | 38.11 | 0.26 | 564.7 | 113.233 | 3.09 | 114.81 | 3.40 | 624.79 |
| 23 | 32 | 37.25 | 1.30 | 118.8 | 115.979 | 3.09 | 118.54 | 3.37 | 612.69 |
| 23 | 33 | 38.01 | 1.42 | 107.5 | 113.566 | 3.09 | 116.03 | 3.38 | 612.57 |
| 23 | 34 | 39.25 | 0.97 | 153.0 | 109.846 | 3.09 | 111.70 | 3.41 | 610.94 |
| 23 | 35 | 40.09 | 1.16 | 128.0 | 107.548 | 3.10 | 109.60 | 3.41 | 615.05 |
| 23 | 36 | 38.47 | 1.50 | 101.3 | 112.168 | 3.09 | 115.08 | 3.38 | 612.39 |
| 23 | 37 | 39.16 | 0.61 | 241.2 | 110.106 | 3.09 | 112.15 | 3.40 | 618.06 |
| 23 | 38 | 36.09 | 0.21 | 736.1 | 119.760 | 3.09 | 120.46 | 3.39 | 616.61 |
| 23 | 39 | 36.02 | 0.55 | 278.7 | 120.054 | 3.09 | 121.86 | 3.37 | 619.24 |
| 23 | 40 | 38.53 | 0.46 | 324.8 | 111.988 | 3.09 | 113.30 | 3.41 | 614.28 |
| 23 | 41 | 38.20 | 1.24 | 122.8 | 112.984 | 3.09 | 115.31 | 3.39 | 615.99 |

21 Mar 99 00:56

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User

| | | | | | | | | | |
|-----|-----------------|-------|------|-------|---------|------|--------|------|--------|
| 23 | 42 | 31.51 | 1.37 | 122.1 | 137.721 | 3.07 | 140.62 | 3.31 | 608.12 |
| 23 | 43 | 31.18 | 1.21 | 138.2 | 139.182 | 3.07 | 140.62 | 3.33 | 613.46 |
| 23 | 44 | 22.57 | 0.00 | 0.00 | 193.566 | 3.05 | 193.37 | 3.25 | 616.36 |
| 23 | 45 | 22.56 | 1.61 | 122.8 | 193.742 | 3.05 | 196.13 | 3.23 | 612.47 |
| 23 | 46 | 21.75 | 0.00 | 0.00 | 201.083 | 3.05 | 202.08 | 3.23 | 617.00 |
| 23 | 47 | 21.70 | 0.81 | 244.6 | 201.462 | 3.05 | 203.40 | 3.22 | 616.85 |
| 237 | MISSING TUBE(S) | | | | | | | | |

CAPPED

| Sample Name | wt. Of PP tubes, g | wt. Of PP tubes after transfer of qtz/clino, g | wt. Of PP tubes after acid add, g | Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer & acid add, g |
|--------------|--------------------|--|-----------------------------------|--|--|
| QC0U-pH 3.5 | 13.4138 | 21.2852 | 24.3348 | 30.4239 | 51.9445 |
| QC0U-pH 4.0 | 13.7808 | 22.656 | 25.6893 | 31.1719 | 50.9359 |
| QC0U-pH 4.25 | 13.3195 | 22.6802 | 25.715 | 31.5926 | 50.322 |
| QC0U-pH 4.5 | 13.6938 | 21.3457 | 24.378 | 30.0287 | 51.8978 |
| QC0U-pH4.75 | 13.3174 | 21.713 | 24.7398 | 30.6473 | 51.2238 |
| QC0U-pH 5.0 | 13.8651 | 20.9052 | 23.9341 | 29.4276 | 52.8144 |
| QC0U-pH 5.25 | 13.7675 | 21.6206 | 24.6424 | 30.3035 | 51.5289 |
| QC0U-pH 5.5 | 13.6808 | 22.0589 | 25.0837 | 31.1244 | 51.5901 |
| QC0U-pH 5.75 | 13.6518 | 21.7224 | 24.7443 | 30.2698 | 51.2088 |
| QC0U-pH 6.0 | 13.7025 | 17.5002 | 20.5134 | 26.271 | 54.6003 |
| QC0U-pH 6.25 | 13.6753 | 21.9654 | 24.9816 | 30.5427 | 51.0169 |
| QC0U-pH 6.5 | 13.6601 | 21.3192 | 24.3389 | 30.0568 | 51.9747 |
| QC0U-pH 6.75 | 13.8008 | 20.9656 | 23.9796 | 30.0457 | 52.7965 |
| QC0U-pH 7.0 | 13.5995 | 20.8474 | 23.868 | 29.7701 | 52.266 |
| QC0U-pH 7.25 | 13.6988 | 21.8706 | 24.8945 | 30.9759 | 51.2961 |
| QC0U-pH 7.5 | 13.6369 | 21.3865 | 24.3995 | 30.2397 | 51.5801 |
| QC0U-pH 7.75 | 13.6932 | 23.3674 | 26.3822 | 32.279 | 49.9724 |
| QC0U-pH 8.0 | 13.7811 | 20.9377 | 23.9496 | 29.714 | 52.5862 |
| QC0U-pH 8.25 | 13.3388 | 23.016 | 26.0293 | 32.2348 | 49.8691 |
| QC0U-pH 8.5 | 13.7773 | 19.4989 | 22.5165 | 28.2794 | 53.4822 |
| QC0U-pH 8.75 | 13.6982 | 21.4451 | 24.4567 | 30.0947 | 51.2555 |
| QC0U-pH 9.0 | 13.8022 | 20.9199 | 23.9228 | 29.678 | 52.1024 |
| QC0U-pH 9.25 | 13.6409 | 21.3531 | 24.3618 | 30.2734 | 51.3205 |

DESORPTION (CAPPED)

| PP tubes | | Desorption | PC containers | | Desorption |
|---------------|---------------|----------------------|---------------|----------------------|------------|
| Sample Name | wt. of vial,g | wt. of vial+sample,g | wt. of vial,g | wt. of vial+sample,g | |
| QC0U-pH 3.5a | 7.8489 | 8.3512 | 7.8145 | 8.3179 | |
| QC0U-pH 3.5b | 7.8143 | 8.3181 | 7.8666 | 8.3681 | |
| QC0U-pH 4a | 7.8293 | 8.3304 | 7.9019 | 8.4052 | |
| QC0U-pH 4b | 7.8433 | 8.3431 | 7.9447 | 8.4499 | |
| QC0U-pH 4.25a | 7.8672 | 8.3645 | 7.9291 | 8.4309 | |
| QC0U-pH 4.25b | 7.8477 | 8.3492 | 7.84 | 8.3423 | |
| QC0U-pH 4.5a | 7.9254 | 8.4273 | 7.8093 | 8.3115 | |
| QC0U-pH 4.5b | 7.9131 | 8.4128 | 7.8852 | 8.3908 | |
| QC0U-pH 4.75a | 7.9206 | 8.4233 | 7.8253 | 8.327 | |
| QC0U-pH 4.75b | 7.8484 | 8.3478 | 7.8276 | 8.3304 | |
| QC0U-pH 5a | 7.8789 | 8.3806 | 7.9049 | 8.4061 | |
| QC0U-pH 5b | 7.9097 | 8.4095 | 7.8919 | 8.3952 | |
| QC0U-pH 5.25a | 7.8056 | 8.3049 | 7.9 | 8.4011 | |
| QC0U-pH 5.25b | 7.8152 | 8.3157 | 7.8769 | 8.3791 | |
| QC0U-pH 5.5a | 7.904 | 8.4031 | 7.8585 | 8.3594 | |
| QC0U-pH 5.5b | 7.8536 | 8.3556 | 7.855 | 8.3571 | |
| QC0U-pH 5.5c | 7.8471 | 8.3451 | 7.8799 | 8.381 | |
| QC0U-pH 5.5d | 7.8623 | 8.3626 | 7.8899 | 8.3939 | |
| QC0U-pH 5.75a | 7.8411 | 8.3453 | 7.8531 | 8.3548 | |
| QC0U-pH 5.75b | 7.8434 | 8.3479 | 7.9195 | 8.4208 | |
| QC0U-pH 6a | 7.9098 | 8.4062 | 7.8617 | 8.3636 | |
| QC0U-pH 6b | 7.914 | 8.411 | 7.8763 | 8.379 | |
| QC0U-pH 6.25a | 7.8479 | 8.345 | 7.8865 | 8.3855 | |
| QC0U-pH 6.25b | 7.9226 | 8.4256 | 7.9084 | 8.4089 | |
| QC0U-pH 6.5a | 7.8539 | 8.3554 | 7.7932 | 8.293 | |
| QC0U-pH 6.5b | 7.898 | 8.3973 | 7.7966 | 8.2989 | |
| QC0U-pH 6.75a | 7.8724 | 8.3724 | 7.9319 | 8.4314 | |
| QC0U-pH 6.75b | 7.8562 | 8.3562 | 7.8704 | 8.371 | |
| QC0U-pH 7a | 7.8955 | 8.3968 | 7.8648 | 8.3679 | |
| QC0U-pH 7b | 7.9111 | 8.4083 | 7.9293 | 8.4355 | |
| QC0U-pH 7.25a | 7.8905 | 8.3887 | 7.8801 | 8.3849 | |
| QC0U-pH 7.25b | 7.8733 | 8.3715 | 7.8104 | 8.3157 | |
| QC0U-pH 7.5a | 7.9554 | 8.4548 | 7.8845 | 8.3858 | |
| QC0U-pH 7.5b | 7.9265 | 8.4235 | 7.8443 | 8.3469 | |
| QC0U-pH 7.75a | 7.8335 | 8.3314 | 7.8201 | 8.3218 | |
| QC0U-pH 7.75b | 7.8745 | 8.3737 | 7.8104 | 8.314 | |
| QC0U-pH 8a | 7.86 | 8.3602 | 7.9448 | 8.4453 | |
| QC0U-pH 8b | 7.8988 | 8.3999 | 7.8302 | 8.3312 | |
| QC0U-pH 8.25a | 7.9614 | 8.3358 | 7.9176 | 8.4173 | |

| | | | | |
|---------------|--------|--------|--------|--------|
| QC0U-pH 8.25b | 7.9016 | 8.3975 | 7.8782 | 8.3796 |
| QC0U-pH 8.5a | 7.8782 | 8.3783 | 7.8562 | 8.3565 |
| QC0U-pH 8.5b | 7.879 | 8.377 | 7.8822 | 8.3845 |
| QC0U-pH 8.75a | 7.8181 | 8.3121 | 7.8084 | 8.3084 |
| QC0U-pH 8.75b | 7.8655 | 8.3641 | 7.8821 | 8.3839 |
| QC0U-pH 9a | 7.8633 | 8.866 | 7.8614 | 8.361 |
| QC0U-pH 9b | 7.8499 | 8.3499 | 7.8717 | 8.3724 |
| QC0U-pH 9.25a | 7.8788 | 8.3781 | | |
| QC0U-pH 9.25b | 7.8676 | 8.3685 | | |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 31-Mar-99 19:20

C14 Eff (0-156 keV) = 96.49 %

C14 CHI SQUARE IPA DATA PROCESSED - 31-Mar-99 19:30

C14 Chi Square = 28.11

H3 IPA DATA PROCESSED - 31-Mar-99 19:32

H3 Eff (0-18.6 keV) = 64.95 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves & view historic data

H3 CHI SQUARE IPA DATA PROCESSED - 31-Mar-99 19:42

H3 Chi Square = 23.96

BKG IPA DATA PROCESSED - 31-Mar-99 20:43

Bkg (0-18.6 keV) = 20.82 cpm

Bkg (0-156 keV) = 30.25 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 401.20H3 E²/B (1-18.6 keV) = 201.72

236 MISSING TUBE(S)

127 9.34 2.24 138.0 472.552 3.02 475.30 3.09 593.55

23 128 9.38 1.61 188.5 470.309 3.02 471.76 3.10 587.91

23 129 9.28 0.00 0.00 475.738 3.02 477.81 3.10 596.80

23 130 9.12 3.76 85.95 483.925 3.02 491.53 3.08 597.52

23 131 9.88 2.55 118.8 446.737 3.02 450.24 3.10 596.53

23 132 9.36 0.00 0.00 471.321 3.02 472.62 3.10 599.24

23 133 10.42 3.88 78.24 423.310 3.02 429.32 3.09 592.57

23 134 10.06 3.44 88.98 438.781 3.02 443.25 3.09 594.63

23 135 10.83 2.52 114.9 407.061 3.03 410.29 3.11 591.71

23 136 10.49 2.58 114.2 420.272 3.02 423.97 3.10 597.96

23 137 12.07 0.38 690.1 364.886 3.03 365.78 3.13 601.04

capped QC0U containers description

| | | | | | | | | |
|---------------------|-------|------|-------|---------|------|--------|------|--------|
| 23 138 | 11.84 | 3.64 | 77.82 | 371.957 | 3.03 | 374.30 | 3.12 | 595.96 |
| 23 139 | 10.30 | 1.73 | 168.4 | 428.380 | 3.02 | 431.27 | 3.10 | 595.96 |
| 23 140 | 10.42 | 1.67 | 172.9 | 423.598 | 3.02 | 426.06 | 3.10 | 597.32 |
| 23 141 | 13.07 | 2.96 | 90.00 | 336.856 | 3.03 | 341.21 | 3.12 | 596.55 |
| 23 142 | 13.18 | 1.02 | 248.5 | 333.788 | 3.03 | 335.23 | 3.14 | 598.35 |
| 23 143 | 17.10 | 1.95 | 116.8 | 256.418 | 3.04 | 259.59 | 3.17 | 596.15 |
| 23 144 | 16.97 | 0.70 | 315.6 | 258.409 | 3.04 | 260.33 | 3.18 | 597.97 |
| 23 145 | 13.97 | 2.56 | 99.84 | 314.788 | 3.03 | 318.19 | 3.14 | 596.55 |
| 23 146 | 14.50 | 2.01 | 123.1 | 303.087 | 3.03 | 306.86 | 3.14 | 597.15 |
| 23 147 | 19.55 | 1.87 | 113.9 | 224.003 | 3.05 | 227.11 | 3.19 | 594.15 |
| 23 148 | 19.98 | 2.40 | 88.79 | 218.957 | 3.05 | 222.49 | 3.20 | 597.07 |
| 23 149 | 28.82 | 1.45 | 120.1 | 150.733 | 3.07 | 153.13 | 3.29 | 597.00 |
| 23 150 | 27.98 | 2.10 | 85.73 | 155.363 | 3.07 | 158.18 | 3.28 | 591.87 |
| 23 151 | 28.11 | 0.00 | 0.00 | 154.735 | 3.07 | 155.50 | 3.31 | 601.08 |
| 23 152 | 27.86 | 0.93 | 187.7 | 156.119 | 3.07 | 157.84 | 3.29 | 594.15 |
| 23 153 | 30.36 | 1.09 | 154.2 | 142.912 | 3.07 | 145.92 | 3.30 | 602.42 |
| 23 154 | 31.28 | 2.17 | 78.52 | 138.607 | 3.08 | 140.82 | 3.32 | 596.46 |
| 23 155 | 21.45 | 1.94 | 104.8 | 203.761 | 3.05 | 205.55 | 3.22 | 597.70 |
| 23 156 | 21.57 | 0.80 | 246.6 | 202.655 | 3.05 | 203.13 | 3.24 | 599.45 |
| 23 157 | 26.78 | 0.99 | 180.6 | 162.480 | 3.06 | 163.68 | 3.29 | 595.45 |
| 23 158 | 26.60 | 0.91 | 197.6 | 163.640 | 3.06 | 165.44 | 3.28 | 594.95 |
| 23 159 | 31.14 | 0.76 | 217.5 | 139.245 | 3.08 | 140.14 | 3.34 | 597.41 |
| 23 160 | 30.63 | 1.20 | 140.1 | 141.719 | 3.07 | 143.95 | 3.31 | 599.20 |
| 23 161 | 31.49 | 0.50 | 327.7 | 137.691 | 3.08 | 138.26 | 3.35 | 596.69 |
| 23 162 | 33.82 | 1.28 | 125.6 | 127.937 | 3.08 | 129.13 | 3.36 | 596.65 |
| 23 163 | 35.48 | 1.87 | 85.31 | 121.817 | 3.09 | 124.19 | 3.36 | 596.56 |
| 23 164 | 34.86 | 0.33 | 466.6 | 124.045 | 3.09 | 124.75 | 3.38 | 599.87 |
| 23 165 | 40.77 | 0.61 | 237.4 | 105.586 | 3.10 | 106.64 | 3.44 | 606.57 |
| 23 166 | 40.77 | 0.14 | 996.8 | 105.586 | 3.10 | 106.74 | 3.44 | 604.53 |
| 23 167 | 19.68 | 0.00 | 0.00 | 222.500 | 3.05 | 223.58 | 3.21 | 601.60 |
| 23 168 | 20.60 | 1.39 | 147.7 | 212.312 | 3.05 | 214.62 | 3.21 | 598.25 |
| 23 169 | 24.10 | 0.87 | 216.4 | 180.933 | 3.06 | 183.41 | 3.25 | 600.92 |
| 23 170 | 24.47 | 0.88 | 211.8 | 178.145 | 3.06 | 180.31 | 3.25 | 601.79 |
| 23 171 | 23.49 | 1.32 | 145.3 | 185.722 | 3.06 | 186.68 | 3.25 | 596.76 |
| 23 172 | 22.59 | 2.17 | 92.10 | 193.348 | 3.05 | 196.35 | 3.22 | 596.49 |
| 238 MISSING TUBE(S) | | | | | | | | |
| 181 | 11.04 | 4.17 | 71.11 | 399.343 | 3.03 | 404.92 | 3.10 | 592.65 |
| 23 182 | 10.94 | 3.57 | 82.44 | 403.391 | 3.02 | 408.16 | 3.10 | 590.79 |
| 23 183 | 10.87 | 4.64 | 65.04 | 405.827 | 3.02 | 412.17 | 3.09 | 588.62 |
| 23 184 | 10.53 | 2.11 | 137.6 | 418.757 | 3.02 | 424.43 | 3.09 | 593.91 |

04 Apr 99 08:05

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

User :

| | | | | | | | | |
|--------|------|------|-------|---------|------|--------|------|--------|
| 23 185 | 9.71 | 1.50 | 198.8 | 454.413 | 3.02 | 458.13 | 3.09 | 596.05 |
| 23 186 | 9.76 | 5.08 | 63.26 | 452.068 | 3.02 | 457.88 | 3.09 | 587.40 |
| 23 187 | 8.27 | 4.38 | 78.46 | 534.141 | 3.02 | 541.20 | 3.07 | 593.66 |
| 23 188 | 8.19 | 5.97 | 59.80 | 539.514 | 3.02 | 546.90 | 3.07 | 588.80 |
| 23 189 | 7.02 | 8.28 | 48.51 | 629.868 | 3.02 | 640.24 | 3.05 | 586.92 |
| 23 190 | 7.25 | 7.39 | 52.66 | 609.500 | 3.02 | 620.52 | 3.05 | 584.64 |
| 23 191 | 7.98 | 4.01 | 86.56 | 553.678 | 3.02 | 559.31 | 3.07 | 588.40 |
| 23 192 | 8.10 | 1.81 | 181.8 | 545.300 | 3.02 | 549.48 | 3.08 | 589.73 |
| 23 193 | 5.31 | 2.11 | 193.2 | 835.895 | 3.01 | 839.89 | 3.05 | 590.46 |
| 23 194 | 5.29 | 5.22 | 83.68 | 836.800 | 3.01 | 845.26 | 3.04 | 587.61 |

| | | | | | | | | |
|--------|------|-------|-------|----------|------|---------|------|--------|
| 23 195 | 5.35 | 2.70 | 152.9 | 829.245 | 3.01 | 834.51 | 3.05 | 589.15 |
| 23 196 | 5.34 | 1.99 | 204.1 | 828.932 | 3.01 | 833.88 | 3.05 | 586.99 |
| 23 197 | 5.38 | 5.73 | 76.37 | 824.974 | 3.01 | 833.78 | 3.04 | 585.64 |
| 23 198 | 5.50 | 5.53 | 77.91 | 805.262 | 3.01 | 813.68 | 3.04 | 588.69 |
| 23 199 | 4.10 | 4.47 | 109.3 | 1082.389 | 3.01 | 1088.88 | 3.03 | 585.96 |
| 23 200 | 4.08 | 2.87 | 164.8 | 1088.447 | 3.01 | 1091.17 | 3.04 | 585.45 |
| 23 201 | 3.26 | 5.23 | 106.3 | 1364.633 | 3.00 | 1372.73 | 3.02 | 578.07 |
| 23 202 | 3.34 | 6.13 | 91.26 | 1327.373 | 3.01 | 1337.95 | 3.02 | 577.78 |
| 23 203 | 3.89 | 5.27 | 96.68 | 1140.494 | 3.01 | 1147.96 | 3.03 | 589.74 |
| 23 204 | 3.82 | 6.26 | 83.79 | 1161.980 | 3.01 | 1172.68 | 3.03 | 584.51 |
| 23 205 | 3.11 | 0.34 | 1517 | 1425.795 | 3.01 | 1425.89 | 3.03 | 585.23 |
| 23 206 | 3.10 | 5.56 | 103.2 | 1431.051 | 3.01 | 1436.39 | 3.03 | 585.21 |
| 23 207 | 2.97 | 6.01 | 98.50 | 1492.831 | 3.01 | 1500.89 | 3.02 | 583.66 |
| 23 208 | 2.93 | 6.02 | 98.99 | 1513.600 | 3.01 | 1522.46 | 3.02 | 582.45 |
| 23 209 | 2.94 | 9.33 | 67.67 | 1511.501 | 3.00 | 1521.27 | 3.02 | 582.24 |
| 23 210 | 2.92 | 9.53 | 66.70 | 1519.823 | 3.01 | 1531.89 | 3.02 | 585.00 |
| 23 211 | 3.76 | 5.88 | 89.26 | 1178.716 | 3.01 | 1184.42 | 3.03 | 584.52 |
| 23 212 | 3.73 | 6.09 | 86.91 | 1189.296 | 3.01 | 1198.74 | 3.03 | 584.18 |
| 23 213 | 3.38 | 9.37 | 62.91 | 1313.990 | 3.01 | 1324.14 | 3.02 | 586.04 |
| 23 214 | 3.29 | 4.70 | 116.6 | 1350.942 | 3.00 | 1357.82 | 3.02 | 586.64 |
| 23 215 | 3.63 | 5.98 | 89.53 | 1221.604 | 3.01 | 1231.19 | 3.03 | 584.33 |
| 23 216 | 3.65 | 5.56 | 95.19 | 1215.987 | 3.01 | 1223.19 | 3.03 | 589.16 |
| 23 217 | 2.85 | 3.59 | 160.3 | 1555.833 | 3.01 | 1563.57 | 3.02 | 581.99 |
| 23 218 | 2.85 | 4.64 | 126.6 | 1556.886 | 3.01 | 1564.97 | 3.02 | 585.01 |
| 23 219 | 4.74 | 4.55 | 100.0 | 934.721 | 3.01 | 938.52 | 3.05 | 601.82 |
| 23 220 | 3.57 | 4.17 | 124.8 | 1243.594 | 3.01 | 1250.14 | 3.03 | 591.53 |
| 23 221 | 2.17 | 0.00 | 0.00 | 2046.304 | 3.00 | 2049.39 | 3.02 | 584.65 |
| 23 222 | 2.16 | 8.32 | 86.98 | 2055.794 | 3.00 | 2065.96 | 3.02 | 583.39 |
| 23 223 | 3.82 | 2.07 | 232.3 | 1160.409 | 3.01 | 1163.52 | 3.04 | 585.14 |
| 23 224 | 3.84 | 3.52 | 140.7 | 1155.910 | 3.01 | 1159.39 | 3.04 | 586.91 |
| 23 225 | 1.61 | 14.86 | 62.58 | 2757.405 | 3.00 | 2777.42 | 3.01 | 545.85 |
| 23 226 | 3.20 | 3.52 | 154.1 | 1385.597 | 3.01 | 1389.96 | 3.03 | 585.30 |
| 23 227 | 3.61 | 6.12 | 87.91 | 1229.776 | 3.01 | 1239.28 | 3.03 | 586.40 |
| 23 228 | 3.52 | 6.22 | 87.79 | 1261.876 | 3.01 | 1268.88 | 3.03 | 585.28 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 04-Apr-99 11:50

C14 Eff (0-156 keV) = 96.35 %

C14 CHI SQUARE IPA DATA PROCESSED - 04-Apr-99 12:01

C14 Chi Square = 23.02

H3 IPA DATA PROCESSED - 04-Apr-99 12:02

H3 Eff (0-18.6 keV) = 64.86 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves & view historic data

3/2/99

AJ

Experimental Procedure for experiment C20 - U sorption on clinoptilolite at 25° C. *UNCAPPED*
(2nd Set)

Objective: To investigate the characteristics of U sorption on clinoptilolite as a function of pH.

Initial conditions: - $\Sigma U = 50$ ppb [2.1×10^{-7} molal]
 - equilibrium with atmospheric $CO_2(g)$; $pCO_2 = 10^{-3.5}$
 - pH range 3.0 - 9.5
 - solution volume = 35 ml
 - mass of solid = 0.700 g
 - M/V = 20 g/L

Equipment: Orion 920A pH meter and combination pH electrode
 Gyrotory shaker
 Fisher Marathon 21K centrifuge
 Mettler PM4600 and AE240 balances
 Packard 2500 TR/AB LSC
 Eppendorf micropipettors and tips
 Oxford pipettes and tips
 Repipettor for transfer of scintillation cocktail

Supplies: Na-clinoptilolite (CDV*100/200*UC*WA*CPT*HL*Naf)
 $2.1 \times 10^{-7} m$ ^{233}U stock solution (spike 28A)
 polycarbonate centrifuge tubes with caps (50 ml capacity)
 polypropylene centrifuge tubes with caps (50 ml capacity)
 pH buffer solutions
 Ultima-Gold liquid scintillation cocktail
 7 ml scintillation vials
 reagent grade $NaHCO_3$ (lot 936883)
 concentrated HNO_3 (lot 983546)
 HNO_3 and $NaHCO_3$ stock solutions at various concentrations
 weighing paper/boats
 deionized ultrapure water, DH_2O
 teflon beaker

Procedure:

A. Transfer $2.1 \times 10^{-7} m$ (50 ppb) ^{233}U stock solution to experimental containers.

- Label 29 polycarbonate centrifuge tubes (~40 ml capacity) C20U-pHi (where i is the approximate pH of each solution, see Table C20).
- Label 1 polycarbonate centrifuge tube C20U-IU.
- Weigh and record the weight of each container.
- The concentration of spike 28A is $2.1 \times 10^{-7} m$ ^{233}U and therefore this stock solution does not require dilution. About 1.2L of spike 28A will be required for this experiment.
- Add ~35 g of the $2.1 \times 10^{-7} m$ ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.*
- Record weight of each container.

B. Add clinoptilolite to sample containers.

- Add ~0.700 g of Na-clinoptilolite to each sample container. **Do not** add clinoptilolite to the C20U-IU container.
- Record weight of each sample container after addition of clinoptilolite.

Adjust pH of each solution by adding HNO_3 or $NaHCO_3$.

- Add the volume of HNO_3 or $NaHCO_3$ to each experimental container as listed in Table C20. Take care to add HNO_3 or $NaHCO_3$ in the concentrations specified in Table C20.
- For sample C20-U-IU (not listed in Table C20) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

Sample C20-U-IU for initial [U].

- Label two 7 ml LSC vials as C20-U-IUa and C20-U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from C20-U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container C20-U-IU

Wait for equilibrium.

- Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
- Place containers on gyrotory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.

Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., C20-U-pH3a and C20-U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

Transfer clinoptilolite for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity)

- (e.g., C20d-U-pH3 for sample container C20-U-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw clinoptilolite from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO₃ to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

C. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., C20-U-pH3a and C20-U-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table CQ0U: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with q/clinoz (35 ml)

| Estimated solution pH | Volume of HNO3 added, ml | Molarity of HNO3 to use |
|-----------------------|----------------------------|---------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO3 added, ml | Molarity of NaHCO3 to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |

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| SAMPLE NAME | Wt. of container, g |
|---------------------|---------------------|
| C20U-pH 3 | 22.2698 |
| AT C20U-pH 3.25 3.5 | 22.1503 |
| C20U-pH 3.5 4.0 | 22.4523 |
| C20U-pH 3.75 4.25 | 22.4432 |
| C20U-pH 4.0 4.5 | 22.3428 |
| C20U-pH 4.25 4.75 | 22.4005 |
| C20U-pH 4.5 5.0 | 22.3805 |
| C20U-pH 4.75 5.25 | 22.5997 |
| C20U-pH 5 5.5 | 22.2312 |
| C20U-pH 5.25 5.75 | 22.5663 |
| C20U-pH 5.5 6.0 | 22.1761 |
| C20U-pH 5.75 6.25 | 22.0155 |
| C20U-pH 6 6.5 | 22.1343 |
| C20U-pH 6.25 6.75 | 22.5258 |
| C20U-pH 6.5 7.0 | 22.0197 |
| C20U-pH 6.75 7.25 | 22.2984 |
| C20U-pH 7 7.5 | 22.4808 |
| C20U-pH 7.25 7.75 | 22.2353 |
| C20U-pH 7.5 8.0 | 22.2532 |
| C20U-pH 7.75 8.25 | 22.2597 |
| C20U-pH 8 8.5 | 22.2468 |
| C20U-pH 8.25 8.75 | 22.3424 |
| C20U-pH 8.5 9.0 | 22.2858 |
| C20U-pH 8.75 9.25 | 22.3918 |
| C20U-pH 9 9.5 | 22.0813 |
| C20U-pH 9.25 | |
| C20U-pH 9.5 | |
| C20U-IU | 22.5791 |

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4/2/99

| SAMPLE NAME | WT. OF CONTAINER + U SOLN., g |
|--------------|-------------------------------|
| C20U-pH 3 | 57.2817 |
| C20U-pH 3.5 | 57.1511 |
| C20U-pH 4 | 57.448 |
| C20U-pH 4.25 | 57.4492 |
| C20U-pH 4.5 | 57.3326 |
| C20U-pH 4.75 | 57.4015 |
| C20U-pH 5 | 57.3268 |
| C20U-pH 5.25 | 57.6058 |
| C20U-pH 5.5 | 57.2473 |
| C20U-pH 5.75 | 57.569 |
| C20U-pH 6 | 57.1929 |
| C20U-pH 6.25 | 57.0167 |
| C20U-pH 6.5 | 57.1464 |
| C20U-pH 6.75 | 57.5232 |
| C20U-pH 7 | 57.0089 |
| C20U-pH 7.25 | 57.296 |
| C20U-pH 7.5 | 57.4894 |
| C20U-pH 7.75 | 57.2464 |
| C20U-pH 8 | 57.2541 |
| C20U-pH 8.25 | 57.2613 |
| C20U-pH 8.5 | 57.2537 |
| C20U-pH 8.75 | 57.3512 |
| C20U-pH 9 | 57.282 |
| C20U-pH 9.25 | 57.4005 |
| C20U-pH 9.5 | 57.0837 |
| C20U-IU | 57.5866 |

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| SAMPLE NAME | WT. OF CONTAINER + U SOLN +CLINOP, g |
|--------------|---|
| C20U-pH 3 | 57.9897 |
| C20U-pH 3.5 | 57.8596 |
| C20U-pH 4 | 58.1559 |
| C20U-pH 4.25 | 58.1570 |
| C20U-pH 4.5 | 58.0338 |
| C20U-pH 4.75 | 58.1086 |
| C20U-pH 5 | 58.0341 |
| C20U-pH 5.25 | 58.3144 |
| C20U-pH 5.5 | 57.9559 |
| C20U-pH 5.75 | 58.2768 |
| C20U-pH 6 | 57.9008 |
| C20U-pH 6.25 | 57.7269 |
| C20U-pH 6.5 | 57.8559 |
| C20U-pH 6.75 | 58.2321 |
| C20U-pH 7 | 57.7187 |
| C20U-pH 7.25 | 58.0053 |
| C20U-pH 7.5 | 58.1991 |
| C20U-pH 7.75 | 57.9567 |
| C20U-pH 8 | 57.9644 |
| C20U-pH 8.25 | 57.9705 |
| C20U-pH 8.5 | 57.9631 |
| C20U-pH 8.75 | 58.0609 |
| C20U-pH 9 | 57.9918 |
| C20U-pH 9.25 | 58.1111 |
| C20U-pH 9.5 | 57.7916 |
| | |
| C20U-IU | |

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| SAMPLE NAME | WT. OF CONTAINER AFTER ADDITIONS OF HNO3 /NAHCO3, g |
|--------------|--|
| C20U-pH 3 | 58.3909 |
| C20U-pH 3.5 | 57.9706 |
| C20U-pH 4 | 58.3007 |
| C20U-pH 4.25 | 58.2422 |
| C20U-pH 4.5 | 58.3258 |
| C20U-pH 4.75 | 58.3185 |
| C20U-pH 5 | 58.2716 |
| C20U-pH 5.25 | 58.5717 |
| C20U-pH 5.5 | 58.2329 |
| C20U-pH 5.75 | 58.5671 |
| C20U-pH 6 | 58.2025 |
| C20U-pH 6.25 | 58.0461 |
| C20U-pH 6.5 | 58.9266 |
| C20U-pH 6.75 | 58.3172 |
| C20U-pH 7 | 57.8285 |
| C20U-pH 7.25 | 58.1439 |
| C20U-pH 7.5 | 58.4117 |
| C20U-pH 7.75 | 58.1270 |
| C20U-pH 8 | 58.2432 |
| C20U-pH 8.25 | 58.0685 |
| C20U-pH 8.5 | 58.1411 |
| C20U-pH 8.75 | 58.2293 |
| C20U-pH 9 | 58.3142 |
| C20U-pH 9.25 | 58.9848 |
| C20U-pH 9.5 | 59.1764 |
| | |
| C20U-IU | 57.6882 |

Table C20U: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb in contact with quartz (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 (0.110) | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 + 0.300 + 0.225 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 + 0.300 | 0.01 |
| 5.75 | 0.286 + 0.350 | 0.01 |
| 6 | 0.300 + 0.160 | 0.01 |
| 6.25 | 0.322 + 0.180 | 0.01 |
| 6.5 | 0.071 + 0.375 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |
| 9.25 | 0.825 | 1 |
| 9.5 | 1.300 | 1 |
| Q20U-IU | 0.100 | 50% HNO ₃ |

| Sample Name | wt. Of vial | wt. Of vial + sample |
|--------------|-------------|----------------------|
| C20-pH 3a | 7.7871 | 8.2838 |
| C20-pH 3b | 7.7956 | 8.2923 |
| C20-pH 4a | 7.7227 | 8.2176 |
| C20-pH 4b | 7.7522 | 8.2476 |
| C20-pH 4.25a | 7.8441 | 8.3390 |

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| | | |
|--------------|-------------------------|--------|
| C20-pH 4.25b | 7.7868 | 8.2824 |
| C20-pH 4.5a | 7.7846 | 8.2779 |
| C20-pH 4.5b | 7.8248 | 8.3204 |
| C20-pH 4.75a | 7.7426 | 8.2368 |
| C20-pH 4.75b | 7.7524 | 8.2490 |
| C20-pH 5a | 7.6984 | 8.1931 |
| C20-pH 5b | 7.7258 | 8.2226 |
| C20-pH 5.25a | 7.7989 8.224 AS 4/14/99 | 8.3297 |
| C20-pH 5.25b | 7.8747 8.463 | 8.3505 |
| C20-pH 5.5a | 7.8190 | 8.3134 |
| C20-pH 5.5b | 7.8046 | 8.3002 |
| C20-pH 5.75a | 7.8747 7.852 | 8.2858 |
| C20-pH 5.75b | 7.8967 8.273 | 8.3281 |
| C20-pH 6a | 7.9066 8.506 | 8.3493 |
| C20-pH 6b | 7.8936 8.626 | 8.3626 |
| C20-pH 6.25a | 7.9104 8.406 | 8.3384 |
| C20-pH 6.25b | 7.8796 7.936 | 8.2933 |
| C20-pH 6.5a | 7.8609 7.722 | 8.2706 |
| C20-pH 6.5b | 7.8645 8.303 | 8.3302 |
| C20-pH 6.75a | 7.9058 | 8.4008 |
| C20-pH 6.75b | 7.8939 | 8.3895 |
| C20-pH 7a | 7.9098 8.532 | 8.3506 |
| C20-pH 7b | 7.7850 8.090 | 8.3081 |
| C20-pH 7.25a | 7.8645 | 8.3602 |
| C20-pH 7.25b | 7.8594 | 8.3557 |
| C20-pH 7.5a | 7.7829 | 8.2763 |
| C20-pH 7.5b | 7.8392 | 8.3362 |
| C20-pH 7.75a | 7.8116 | 8.3066 |
| C20-pH 7.75b | 7.8558 | 8.3503 |
| C20-pH 8a | 7.8524 | 8.3463 |
| C20-pH 8b | 7.8752 | 8.3723 |
| C20-pH 8.25a | 7.8402 | 8.3353 |
| C20-pH 8.25b | 7.8287 | 8.4212 |
| C20-pH 8.5a | 7.8582 | 8.3515 |
| C20-pH 8.5b | 7.8189 | 8.3155 |
| C20-pH 8.75a | 7.8943 | 8.3884 |
| C20-pH 8.75b | 7.8799 | 8.3763 |
| C20-pH 9a | 7.7472 | 8.2429 |
| C20-pH 9b | 7.7325 | 8.2308 |
| C20-pH 9.25a | 7.7826 | 8.2910 |
| C20-pH 9.25b | 7.8173 | 8.3017 |
| C20-pH 9.5a | 7.8426 | 8.3341 |
| C20-pH 9.5b | 7.8862 | 8.3828 |
| C20U-lua | 7.8374 | 8.3418 |
| C20U-lub | 7.8725 | 8.3765 |
| C20U-luc | 7.8771 | 8.3809 |
| C20U-lud | 7.7977 | 8.302 |

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4/15/99

4/16/99

| Sample Name | Container wt. before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements |
|--------------|---|-------------|---|
| C20U-pH 3 | 58.0562 | 3 | 57.0276 |
| C20U-pH 3.5 | 57.6506 | 3.78 | 56.6621 |
| C20U-pH 4 | 57.9927 | 5.05 | 57.0092 |
| C20U-pH 4.25 | 57.9568 | 5.55 | 56.9271 |
| C20U-pH 4.5 | 57.9999 | 6.02 | 57 |
| C20U-pH 4.75 | 57.9555 | 6.31 | 56.9436 |
| C20U-pH 5 | 5/11/99 57.7029 | 5.07 | 56.687 |
| C20U-pH 5.25 | 58.2159 | 6.53 | 57.1841 |
| C20U-pH 5.5 | 57.7268 | 5.38 | 56.7231 |
| C20U-pH 5.75 | 58.0483 | 4.43 | 57.0468 |
| C20U-pH 6 | 57.1332 | 4.49 | 56.1333 |
| C20U-pH 6.25 | 57.3743 | 4.78 | 56.3744 |
| C20U-pH 6.5 | 57.6382 | 6.71 | 56.6114 |
| C20U-pH 6.75 | 57.598 | 5.89 | 56.5989 |
| C20U-pH 7 | 57.4906 | 7.08 | 56.4537 |
| C20U-pH 7.25 | 57.8134 | 7.1 | 56.7812 |
| C20U-pH 7.5 | 58.1286 | 7.39 | 57.0621 |
| C20U-pH 7.75 | 57.7801 | 7.55 | 56.7208 |
| C20U-pH 8 | 57.9075 | 7.74 | 56.7861 |
| C20U-pH 8.25 | 57.6564 | 8.03 | 56.6126 |
| C20U-pH 8.5 | 57.8568 | 8.29 | 56.8258 |
| C20U-pH 8.75 | 57.8957 | 8.66 | 56.8664 |
| C20U-pH 9 | 57.9185 | 8.89 | 56.8754 |
| C20U-pH 9.25 | 58.6502 | 9.15 | 57.6164 |
| C20U-pH 9.5 | 58.7672 | 9.1 | 57.7288 |

20 Apr 99 05:10 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma Page
User : f

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 2S% | BKG |
| Region A: | 0.0 - 100 | 0 | 0.3 | 19.49 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.39 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 28.64 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|-----------------|------------|--------------|-------------|----------|
| P# 1 | 999.99 | 19.49 1.43 | 3.388 3.44 | 28.64 1.18 | 143.82 B |
| 23 2 | 8.77 | 3.09 104.2 | 503.339 3.02 | 506.71 3.08 | 606.97 |
| 23 3 | 8.86 | 3.88 84.07 | 498.644 3.02 | 504.55 3.08 | 599.34 |
| 23 4 | 8.53 | 1.50 210.2 | 518.066 3.02 | 520.37 3.08 | 610.08 |
| 23 5 | 9.08 | 2.10 147.5 | 486.149 3.02 | 489.87 3.09 | 608.49 |
| 231 | MISSING TUBE(S) | | | | |
| 7 | 9.14 | 0.65 462.1 | 482.826 3.02 | 484.17 3.09 | 616.12 |
| 23 8 | 9.05 | 1.18 257.9 | 487.662 3.02 | 489.38 3.09 | 618.49 |
| 23 9 | 12.16 | 0.00 0.00 | 362.155 3.03 | 361.09 3.14 | 631.01 |
| 23 10 | 12.40 | 1.00 259.2 | 355.080 3.03 | 356.45 3.13 | 613.53 |
| 23 11 | 51.63 | 1.70 77.00 | 82.686 3.13 | 85.04 3.51 | 604.68 |
| 23 12 | 52.32 | 0.00 0.00 | 81.570 3.13 | 81.36 3.59 | 615.88 |
| 23 13 | 72.93 | 1.45 76.28 | 57.547 3.18 | 59.22 3.75 | 609.04 |
| 23 14 | 73.26 | 2.00 55.95 | 57.273 3.18 | 59.86 3.72 | 600.64 |
| 23 15 | 85.66 | 0.31 319.9 | 48.491 3.22 | 48.78 3.96 | 614.45 |
| 23 16 | 83.91 | 1.01 101.5 | 49.573 3.21 | 50.82 3.89 | 607.24 |
| 23 17 | 95.81 | 1.61 60.90 | 42.995 3.25 | 44.83 3.98 | 602.65 |
| 23 18 | 95.11 | 0.96 100.5 | 43.337 3.25 | 45.33 3.96 | 607.21 |
| 232 | MISSING TUBE(S) | | | | |
| 21 | 93.66 | 0.69 139.9 | 44.060 3.24 | 45.39 3.99 | 598.78 |
| 23 22 | 88.76 | 1.49 67.81 | 46.691 3.23 | 48.76 3.89 | 594.99 |
| 238 | MISSING TUBE(S) | | | | |
| 31 | 66.96 | 0.99 115.4 | 62.995 3.17 | 64.24 3.70 | 593.40 |
| 23 32 | 68.80 | 1.21 93.43 | 61.220 3.17 | 63.07 3.70 | 592.46 |

8/16/2001
Std.

5/25/99 C20U
50PPb
5/26/99 solution
uncapped

232 MISSING TUBE(S)

| | | | | | | | | | |
|----|-------|-------|-------|--------|---------|-------|--------|--------|--------|
| 35 | 65.45 | 0.47 | 243.4 | 64.526 | 3.16 | 65.16 | 3.71 | 603.47 | 725.9 |
| 23 | 36 | 66.11 | 0.72 | 157.8 | 63.848 | 3.16 | 65.00 | 3.70 | 600.18 |
| 23 | 37 | 74.07 | 1.45 | 75.64 | 56.636 | 3.19 | 58.63 | 3.75 | 602.08 |
| 23 | 38 | 72.73 | 1.10 | 100.3 | 57.770 | 3.18 | 59.35 | 3.75 | 600.73 |
| 23 | 39 | 37.98 | 1.47 | 102.7 | 113.621 | 3.09 | 115.81 | 3.38 | 601.82 |
| 23 | 40 | 35.98 | 1.30 | 118.6 | 120.125 | 3.09 | 121.39 | 3.38 | 606.69 |
| 23 | 41 | 27.99 | 0.91 | 189.3 | 155.383 | 3.07 | 156.75 | 3.29 | 607.56 |
| 23 | 42 | 27.15 | 1.40 | 127.1 | 160.369 | 3.06 | 163.15 | 3.26 | 605.96 |
| 23 | 43 | 17.13 | 2.35 | 96.94 | 256.040 | 3.04 | 259.34 | 3.16 | 605.54 |
| 23 | 44 | 14.61 | 0.71 | 335.6 | 300.856 | 3.03 | 303.33 | 3.14 | 602.30 |
| 23 | 45 | 11.16 | 4.08 | 71.56 | 394.909 | 3.03 | 399.05 | 3.10 | 605.63 |
| 23 | 46 | 10.82 | 1.77 | 159.1 | 407.333 | 3.03 | 409.26 | 3.11 | 607.33 |
| 23 | 47 | 9.01 | 1.71 | 179.9 | 489.842 | 3.02 | 493.12 | 3.09 | 610.84 |
| 23 | 48 | 9.12 | 0.91 | 330.6 | 484.002 | 3.02 | 484.96 | 3.10 | 610.15 |
| 23 | 49 | 8.24 | 2.97 | 111.7 | 535.932 | 3.02 | 539.45 | 3.08 | 606.95 |
| 23 | 50 | 8.56 | 0.00 | 0.00 | 516.121 | 3.02 | 517.16 | 3.09 | 607.31 |

C20V
Scorpion
uncapped

21 Apr 99 06:38

ALPHA/BETA - 1.02

Protocol #:23

U-233 3% 2 sigma

Page

User :

| | | | | | | | | | |
|----|----|------|------|-------|---------|------|--------|------|--------|
| 23 | 51 | 8.20 | 2.59 | 127.3 | 538.685 | 3.02 | 540.27 | 3.08 | 607.92 |
| 23 | 52 | 8.28 | 4.31 | 79.00 | 533.327 | 3.02 | 537.31 | 3.08 | 607.26 |
| 23 | 53 | 8.26 | 0.37 | 844.8 | 535.474 | 3.02 | 537.71 | 3.08 | 608.29 |
| 23 | 54 | 8.45 | 0.87 | 358.7 | 522.529 | 3.02 | 522.25 | 3.09 | 610.74 |
| 23 | 55 | 8.58 | 0.00 | 0.00 | 514.794 | 3.02 | 515.07 | 3.09 | 620.04 |
| 23 | 56 | 8.66 | 3.03 | 106.8 | 510.238 | 3.02 | 513.28 | 3.08 | 617.69 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 21-Apr-99 07:43

C14 Eff (0-156 keV) = 96.65 %

C14 CHI SQUARE IPA DATA PROCESSED - 21-Apr-99 07:54

C14 Chi Square = 21.22

H3 IPA DATA PROCESSED - 21-Apr-99 07:55

H3 Eff (0-18.6 keV) = 64.97 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves & view historic data

H3 CHI SQUARE IPA DATA PROCESSED - 21-Apr-99 08:05

H3 Chi Square = 10.68

BKG IPA DATA PROCESSED - 21-Apr-99 09:06

Bkg (0-18.6 keV) = 21.40 cpm

Bkg (0-156 keV) = 30.82 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 396.26H3 E²/B (1-18.6 keV) = 196.35

15 May 99 09:24

ALPHA/BETA - 1.02

Page

Protocol #:23

U-233 3% 2 sigma

User : A

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 2S% | BKG |
|-----------|------------|----|-----|-----|-------|
| Region A: | 0.0 - 100 | | 0 | 0.3 | 19.86 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.39 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 29.12 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|--------|------------|------------|------------|----------|
| P# 1 | 999.99 | 19.86 | 1.42 | 3.385 | 3.44 |
| 23 2 | 53.56 | 0.49 | 256.6 | 79.625 | 3.13 |
| 23 3 | 56.08 | 0.36 | 339.9 | 75.877 | 3.14 |
| 23 4 | 75.58 | 0.68 | 159.7 | 55.414 | 3.19 |
| 23 5 | 76.79 | 0.13 | 805.8 | 54.487 | 3.19 |
| 23 6 | 20.80 | 1.58 | 129.4 | 210.269 | 3.05 |
| 23 7 | 20.75 | 1.64 | 125.6 | 210.832 | 3.05 |
| 23 8 | 22.33 | 1.01 | 193.3 | 195.719 | 3.05 |
| 23 9 | 22.96 | 1.48 | 131.4 | 190.213 | 3.05 |
| 23 10 | 31.50 | 1.00 | 165.3 | 137.694 | 3.08 |
| 23 11 | 32.17 | 1.19 | 138.4 | 134.787 | 3.08 |
| 23 12 | 137.07 | 0.74 | 110.8 | 29.036 | 3.37 |
| 23 13 | 133.20 | 0.54 | 154.1 | 29.978 | 3.36 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 15-May-99 21:08

C14 Eff (0-156 keV) = 96.75 %

C14 CHI SQUARE IPA DATA PROCESSED - 15-May-99 21:19

C14 Chi Square = 26.99

H3 IPA DATA PROCESSED - 15-May-99 21:20

H3 Eff (0-18.6 keV) = 65.04 %

H3 CHI SQUARE IPA DATA PROCESSED - 15-May-99 21:31

H3 Chi Square = 22.88

BKG IPA DATA PROCESSED - 15-May-99 22:31

Bkg (0-18.6 keV) = 20.67 cpm

Bkg (0-156 keV) = 29.08 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 428.94H3 E²/B (1-18.6 keV) = 204.25

2/14/99 AJ
Experimental Procedure for experiment uncapped QFe20U sorption on clinoptilolite at 25° C.

Objective: To investigate the characteristics of U sorption onto quartz as a function of pH.

Initial conditions: - $\Sigma U = 50$ ppb [2.1×10^{-7} molal]
 - equilibrium with atmospheric $\text{CO}_2(\text{g})$; $\text{pCO}_2 = 10^{-3.5}$
 - pH range 3.0-9.5
 - solution volume = 35 ml
 - mass of solid = 0.700 g
 - $M/V = 20$

Equipment: Orion 920A pH meter and combination pH electrode
 Gyrotory shaker
 Fisher Marathon 21K centrifuge
 Mettler PM4600 and AE240 balances
 Packard 2500 TR/AB LSC
 Eppendorf micropipettors and tips
 Oxford pipettes and tips
 Repipettor for transfer of scintillation cocktail

Supplies: Quartz (W510*60/100*UC*WA*RC*HL)
 2.1×10^{-6} M ^{233}U stock solution (spike 28A)
 0.1M NaNO_3 matrix to dilute spike 28A
 polycarbonate centrifuge tubes with caps (50 ml capacity)
 polypropylene centrifuge tubes with caps (50 ml capacity)
 pH buffer solutions
 Ultima-Gold liquid scintillation cocktail
 7 ml scintillation vials
 reagent grade NaHCO_3 (lot 936883)
 concentrated HNO_3 (lot 983546)
 HNO_3 and NaHCO_3 stock solutions at various concentrations
 weighing paper/boats
 deionized ultrapure water, DH_2O
 teflon beaker

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
15. On the Metler balance, weigh 200g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 16. Dilute to a total of 2000g using 0.1M NaNO_3 stock solution.
 17. Cap and label bottle accordingly.
- B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.

- a. Label 24 polycarbonate centrifuge tubes (50 ml capacity) QFe20U-pHi (where i is the approximate pH of each solution, see Table QFe20U).
- b. Label 1 polycarbonate centrifuge tube QFe20U-IU.
- c. Weigh and record the weight of each container.
- d. About 900ml of ^{233}U solution will be required for this experiment.
- e. Add ~35 g of the 50 ppb ^{233}U stock solution to each container. *Note: the stock solution should be transferred to a Teflon beaker for easier dispensing into the sample containers.*
- f. Record weight of each container.

C. Add quartz to sample containers.

- Add ~0.700 g of quartz to each sample container. **Do not** add quartz to the QFe20U-IU container.
- Record weight of each sample container after addition of quartz.

D. Adjust pH of each solution by adding HNO_3 or NaHCO_3 .

- Add the volume of HNO_3 or NaHCO_3 to each experimental container as listed in Table QFe20U. Take care to add HNO_3 or NaHCO_3 in the concentrations specified in Table QFe20U.
- For sample QFe20U-IU (as listed in Table QFe20U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

E. Sample QFe20U-IU for initial [U].

- Label two 7 ml LSC vials as QFe20U-IUa and QFe20U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from QFe20U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container QFe20U-IU.

F. Wait for equilibrium.

- Place caps on sample containers but do not tighten. Leave caps loose so that solutions can equilibrate with air.
- Place containers on gyrotory shaker set at about 120 rpm and allow samples to equilibrate with air for at least 10 days.

G. Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., QFe20U-pH3a and

QFe20U-pH3b).

- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container after pH measurement and sampling for LSC counting.

H. Transfer quartz for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the quartz transfer is done immediately after the last step in section G..*
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., QFe20Ud-pH3 for sample container QFe20Ud-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw clinoptilolite from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO₃ to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

I. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., QFe20U-pH3a and QFe20U-pH3b).
- Add 0.5 ml of 0.02 M HNO₃ to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table QFe20U: Estimated solution pH and volumes of HNO₃ or NaHCO₃ solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with quartz (35 ml)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--|---------------------------------------|
| 2 | 0.421 | 1 |
| 2.25 | 0.235 | 1 |
| 2.5 | 0.131 | 1 |
| 2.75 | 0.072 | 1 |
| 3 | 0.394 | 0.1 |
| 3.25 | 0.209 | 0.1 |
| 3.5 | 0.106 | 0.1 |
| 3.75 | 0.047 | 0.1 |
| 4 | 0.071 | 0.02 |
| Estimated solution pH | Volume of NaHCO ₃ added, ml | Molarity of NaHCO ₃ to use |
| 4.25 | 0.084 | 0.005 |
| 4.5 | 0.292 | 0.005 |
| 4.75 | 0.205 | 0.01 |
| 5 | 0.239 | 0.01 |
| 5.25 | 0.260 | 0.01 |
| 5.5 | 0.274 | 0.01 |
| 5.75 | 0.286 | 0.01 |
| 6 | 0.300 | 0.01 |
| 6.25 | 0.322 | 0.01 |
| 6.5 | 0.071 | 0.05 |
| 6.75 | 0.084 | 0.05 |
| 7 | 0.106 | 0.05 |
| 7.25 | 0.145 | 0.05 |
| 7.5 | 0.214 | 0.05 |
| 7.75 | 0.169 | 0.1 |
| 8 | 0.282 | 0.1 |
| 8.25 | 0.097 | 0.5 |
| 8.5 | 0.174 | 0.5 |
| 8.75 | 0.160 | 1 |
| 9 | 0.305 | 1 |
| 9.25 | 0.825 | 1 |
| 9.5 | 1.300 | 1 |
| QFe20U-IU | 0.100 | 50/50 HNO ₃ |

UNCAPPED

4/15/99

| SAMPLE NAME | Wt. of container, g |
|----------------|---------------------|
| QFe20U-pH 3 | 22.3254 |
| QFe20U-pH 3.5 | 22.2556 |
| QFe20U-pH 4 | 22.2226 |
| QFe20U-pH 4.25 | 22.2616 |
| QFe20U-pH 4.5 | 22.224 |
| QFe20U-pH 4.75 | 22.1186 |
| QFe20U-pH 5 | 22.1164 |
| QFe20U-pH 5.25 | 22.1562 |
| QFe20U-pH 5.5 | 22.5396 |
| QFe20U-pH 5.75 | 22.2485 |
| QFe20U-pH 6 | 22.2868 |
| QFe20U-pH 6.25 | 22.2538 |
| QFe20U-pH 6.5 | 22.5423 |
| QFe20U-pH 6.75 | 22.2956 |
| QFe20U-pH 7 | 22.3039 |
| QFe20U-pH 7.25 | 22.2768 |
| QFe20U-pH 7.5 | 22.1931 |
| QFe20U-pH 7.75 | 22.2731 |
| QFe20U-pH 8 | 22.2255 |
| QFe20U-pH 8.25 | 22.2586 |
| QFe20U-pH 8.5 | 22.3135 |
| QFe20U-pH 8.75 | 22.2828 |
| QFe20U-pH 9 | 22.2818 |
| QFe20U-pH 9.25 | 22.3788 |
| QFe20U-pH 9.5 | 22.5644 |
| | |
| QFe20U-IU | |

UNCAPPED

4/16/99

| SAMPLE NAME | WT. OF CONTAINER + qtz, g |
|----------------|---------------------------|
| QFe20U-pH 3 | 23.0303 |
| QFe20U-pH 3.5 | 22.9600 |
| QFe20U-pH 4 | 22.926 |
| QFe20U-pH 4.25 | 22.9663 |
| QFe20U-pH 4.5 | 22.9286 |
| QFe20U-pH 4.75 | 22.8165 |
| QFe20U-pH 5 | 22.8134 |
| QFe20U-pH 5.25 | 22.8529 |
| QFe20U-pH 5.5 | 23.2373 |
| QFe20U-pH 5.75 | 22.9476 |
| QFe20U-pH 6 | 22.9831 |
| QFe20U-pH 6.25 | 22.9573 |
| QFe20U-pH 6.5 | 23.2444 |
| QFe20U-pH 6.75 | 22.9982 |
| QFe20U-pH 7 | 23.0062 |
| QFe20U-pH 7.25 | 22.9794 |
| QFe20U-pH 7.5 | 22.8949 |
| QFe20U-pH 7.75 | 22.9758 |
| QFe20U-pH 8 | 22.9282 |
| QFe20U-pH 8.25 | 22.9630 |
| QFe20U-pH 8.5 | 23.0180 |
| QFe20U-pH 8.75 | 22.9856 |
| QFe20U-pH 9 | 22.9851 |
| QFe20U-pH 9.25 | 23.0829 |
| QFe20U-pH 9.5 | 23.2688 |

UNCAPPED

4/15/99

| SAMPLE NAME | WT. OF CONTAINER + U SOLN +qtz , g |
|----------------|------------------------------------|
| QFe20U-pH 3 | 58.0234 |
| QFe20U-pH 3.5 | 57.9872 |
| QFe20U-pH 4 | 57.9255 |
| QFe20U-pH 4.25 | 58.0246 |
| QFe20U-pH 4.5 | 57.9297 |
| QFe20U-pH 4.75 | 57.8153 |
| QFe20U-pH 5 | 57.8102 |
| QFe20U-pH 5.25 | 57.9115 |
| QFe20U-pH 5.5 | 58.2304 |
| QFe20U-pH 5.75 | 57.9693 |
| QFe20U-pH 6 | 57.9927 |
| QFe20U-pH 6.25 | 57.9697 |
| QFe20U-pH 6.5 | 58.2763 |
| QFe20U-pH 6.75 | 57.9882 |
| QFe20U-pH 7 | 58.0289 |
| QFe20U-pH 7.25 | 57.9963 |
| QFe20U-pH 7.5 | 57.8870 |
| QFe20U-pH 7.75 | 57.9685 |
| QFe20U-pH 8 | 57.9398 |
| QFe20U-pH 8.25 | 57.9678 |
| QFe20U-pH 8.5 | 58.0327 |
| QFe20U-pH 8.75 | 58.0052 |
| QFe20U-pH 9 | 57.9934 |
| QFe20U-pH 9.25 | 58.0986 |
| QFe20U-pH 9.5 | 58.2761 |
| QFe20U-IU | |

UNCAPPED

4/19/99

| SAMPLE NAME | WT. OF CONTAINER AFTER ADDITIONS OF HNO3 /NAHCO3, g |
|----------------|--|
| QFe20U-pH 3 | 58.4180 |
| QFe20U-pH 3.5 | 58.0713 |
| QFe20U-pH 4 | 57.9936 |
| QFe20U-pH 4.25 | 58.1058 |
| QFe20U-pH 4.5 | 58.2223 |
| QFe20U-pH 4.75 | 58.0142 |
| QFe20U-pH 5 | 58.0472 |
| QFe20U-pH 5.25 | 58.1707 |
| QFe20U-pH 5.5 | 58.5005 |
| QFe20U-pH 5.75 | 58.2549 |
| QFe20U-pH 6 | 58.2943 |
| QFe20U-pH 6.25 | 58.296 |
| QFe20U-pH 6.5 | 58.3465 |
| QFe20U-pH 6.75 | 58.0669 |
| QFe20U-pH 7 | 58.1339 |
| QFe20U-pH 7.25 | 58.1407 |
| QFe20U-pH 7.5 | 58.1000 |
| QFe20U-pH 7.75 | 58.1363 |
| QFe20U-pH 8 | 58.2109 |
| QFe20U-pH 8.25 | 58.0696 |
| QFe20U-pH 8.5 | 58.2112 |
| QFe20U-pH 8.75 | 58.3317 |
| QFe20U-pH 9 | 58.3160 |
| QFe20U-pH 9.25 | 58.9711 |
| QFe20U-pH 9.5 | 59.6555 |
| QFe20U-IU | |

UN CAPPED

| Sample Name | Container wt./before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements |
|----------------|---|-------------|---|
| QFe20U-pH 3 | 57.9916 | 2.58 | 56.9802 |
| QFe20U-pH 3.5 | 57.7242 | 3.11 | 55.7131 |
| QFe20U-pH 4 | 57.4795 | 3.69 | 55.5008 |
| QFe20U-pH 4.25 | 57.5301 | 4.38 | 56.5315 |
| QFe20U-pH 4.5 | 57.9788 | 4.82 | 56.9742 |
| QFe20U-pH 4.75 | 57.6635 | 5.64 | 56.6899 |
| QFe20U-pH 5 | 57.794 | 6.1 | 56.796 |
| QFe20U-pH 5.25 | 57.9526 | 6.28 | 56.9184 |
| QFe20U-pH 5.5 | 58.2297 57.8253 | 4.49 | 56.8414 |
| QFe20U-pH 5.75 | 58.0249 | 6.59 | 57.024 |
| QFe20U-pH 6 | 57.9746 | 6.55 | 56.9547 |
| QFe20U-pH 6.25 | 57.895 | 6.62 | 56.8851 |
| QFe20U-pH 6.5 | 57.9848 | 6.69 | 56.9055 |
| QFe20U-pH 6.75 | 57.6138 | 6.95 | 56.6086 |
| QFe20U-pH 7 | 57.7188 | 7.1 | 56.7067 |
| QFe20U-pH 7.25 | 57.8859 | 7.23 | 56.8789 |
| QFe20U-pH 7.5 | 57.8066 | 7.26 | 56.7999 |
| QFe20U-pH 7.75 | 57.8525 | 7.62 | 56.8401 |
| QFe20U-pH 8 | 57.8136 | 7.87 | 56.7956 |
| QFe20U-pH 8.25 | 57.7349 | 8.14 | 56.7363 |
| QFe20U-pH 8.5 | 57.8668 | 8.39 | 56.8548 |
| QFe20U-pH 8.75 | 57.9782 | 8.63 | 56.9803 |
| QFe20U-pH 9 | 57.978 | 8.94 | 56.9724 |
| QFe20U-pH 9.25 | 58.7498 | 9.25 | 57.763 |
| QFe20U-pH 9.5 | 59.4224 | 9.26 | 58.4292 |

| Sample Name | wt. Of vial | wt. Of vial + sample |
|----------------|---------------------------|----------------------|
| QFe20U-pH 3a | 7.8516 | 8.3465 |
| QFe20U-pH 3b | 7.746 | 8.2362 |
| QFe20U-pH 3.5a | 7.8336 | 8.3227 |
| QFe20-pH 3.5b | 7.7932 | 8.3356 |
| QFe20-pH 3.5c | 7.8416 | 8.3471 |
| QFe20-pH 3.5d | 7.8946 | 8.3986 |
| QFe20-pH 4a | 7.7937 | 8.2512 |
| QFe20-pH 4b | 7.9327 | 8.455 |
| QFe20-pH 4c | 7.8543 | 8.3384 |
| QFe20-pH 4.25a | 7.9017 | 8.4006 |
| QFe20-pH 4.25b | 7.8532 | 8.3427 |
| QFe20-pH 4.5a | 7.9066 | 8.4016 |
| QFe20-pH 4.5b | 7.8441 | 8.3474 |
| QFe20-pH 4.75a | 7.802 | 8.2869 |
| QFe20-pH 4.75b | 7.8684 | 8.3678 |
| QFe20-pH 5a | 7.8399 | 8.3268 |
| QFe20-pH 5b | 7.8436 | 8.3402 |
| QFe20-pH 5.25a | 7.8486 | 8.3459 |
| QFe20-pH 5.25b | 7.7905 | 8.2883 |
| QFe20-pH 5.5a | 7.8669 7.8240 | 8.3254 AJ 5/18 |
| QFe20-pH 5.5b | 7.8383 7.76303 | 8.2638 AJ 5/18 |
| QFe20-pH 5.75a | 7.8236 | 8.3158 |
| QFe20-pH 5.75b | 7.8332 | 8.3225 |
| QFe20-pH 6a | 7.8522 | 8.3506 |
| QFe20-pH 6b | 7.9023 | 8.4022 |
| QFe20-pH 6.25a | 7.8251 | 8.3176 |
| QFe20-pH 6.25b | 7.8274 | 8.3198 |
| C20-pH 6.5a | 7.8462 | 8.3448 |
| C20-pH 6.5b | 7.8273 | 8.3307 |
| C20-pH 6.75a | 7.8483 | 8.3388 |
| C20-pH 6.75b | 7.8798 | 8.3697 |
| C20-pH 7a | 7.7554 | 8.25 |
| C20-pH 7b | 7.7872 | 8.277 |
| C20-pH 7.25a | 7.8246 | 8.3126 |
| C20-pH 7.25b | 7.8354 | 8.3261 |
| C20-pH 7.5a | 7.8146 | 8.304 |
| C20-pH 7.5b | 7.8402 | 8.3311 |

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| | | | |
|----|-----------------|--------|--------|
| Fe | C20-pH 7.75a | 7.7768 | 8.2674 |
| Fe | C20-pH 7.75b | 7.8629 | 8.3545 |
| Fe | C20-pH 8a | 7.8354 | 8.3239 |
| Fe | C20-pH 8b | 7.7451 | 8.242 |
| Fe | C20-pH 8.25a | 7.8527 | 8.31 |
| Fe | C20-pH 8.25b | 7.7654 | 8.2847 |
| Fe | C20-pH 8.5a | 7.8165 | 8.3 |
| Fe | C20-pH 8.5b | 7.835 | 8.334 |
| Fe | C20-pH 8.75a | 7.824 | 8.3147 |
| Fe | C20-pH 8.75b | 7.7836 | 8.2719 |
| Fe | C20-pH 9a | 7.8037 | 8.2915 |
| Fe | QFe20U-pH 9b | 7.7793 | 8.2776 |
| Fe | C20-pH 9.25a | 7.8792 | 8.369 |
| Fe | QFe20U-pH 9.25b | 7.8302 | 8.3221 |
| Fe | C20-pH 9.5a | 7.908 | 8.3976 |
| Fe | QFe20U-pH 9.5b | 7.8939 | 8.3815 |

04 May 99 14:29 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page #
User : Alk

C14 IPA DATA PROCESSED - 04-May-99 14:29
C14 Eff (0-156 keV) = 96.87 %
C14 CHI SQUARE IPA DATA PROCESSED - 04-May-99 14:39
C14 Chi Square = 21.07
H3 IPA DATA PROCESSED - 04-May-99 14:41
H3 Eff (0-18.6 keV) = 65.06 %
H3 CHI SQUARE IPA DATA PROCESSED - 04-May-99 14:51
H3 Chi Square = 15.94
BKG IPA DATA PROCESSED - 04-May-99 15:52
Bkg (0-18.6 keV) = 20.82 cpm
Bkg (0-156 keV) = 30.47 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 408.86
H3 E²/B (1-18.6 keV) = 202.48

05 May 99 08:41 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page
User :

Time: 999.99
Data Mode: CPM
Background Subtract: 1st Vial

Nuclide: MANUAL

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 2S% | BKG |
| Region A: | 0.0 - 100 | 0 | 0.3 | 20.30 | |
| Region B: | 100 - 350 | 0 | 3.0 | 3.40 | |
| Region C: | 0.0 - 2000 | 0 | 0.1 | 29.52 | |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

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5/4/99

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-------|--------|------------|--------------|-------------|----------|
| P# 1 | 999.99 | 20.30 1.40 | 3.404 3.43 | 29.52 1.16 | 141.54 B |
| 23 2 | 8.68 | 1.48 215.0 | 508.808 3.02 | 510.81 3.09 | 618.74 |
| 23 3 | 9.01 | 0.00 0.00 | 490.048 3.02 | 491.57 3.09 | 617.79 |
| 23 4 | 9.23 | 2.46 128.3 | 478.069 3.02 | 481.86 3.09 | 622.72 |
| 23 5 | 8.49 | 2.20 148.4 | 520.271 3.02 | 523.02 3.09 | 611.99 |
| 23 6 | 9.32 | 3.10 102.8 | 474.386 3.02 | 477.46 3.09 | 617.91 |
| 23 7 | 9.11 | 2.98 107.8 | 484.631 3.02 | 488.27 3.09 | 612.78 |
| 23 8 | 10.48 | 2.99 100.2 | 420.833 3.02 | 423.73 3.11 | 616.55 |
| 23 9 | 10.38 | 0.03 8696 | 425.112 3.02 | 425.40 3.11 | 621.82 |
| 23 10 | 12.69 | 1.77 149.9 | 346.793 3.03 | 350.07 3.13 | 614.44 |
| 23 11 | 12.68 | 2.42 111.3 | 347.306 3.03 | 350.21 3.13 | 617.47 |
| 23 12 | 19.43 | 0.34 606.7 | 225.314 3.05 | 226.94 3.21 | 618.95 |
| 23 13 | 18.78 | 1.06 203.5 | 233.284 3.04 | 234.38 3.20 | 615.26 |
| 23 14 | 22.15 | 0.00 0.00 | 197.318 3.05 | 197.71 3.24 | 620.88 |
| 23 15 | 21.03 | 1.39 147.7 | 207.913 3.05 | 209.90 3.22 | 616.62 |
| 23 16 | 23.57 | 0.49 384.6 | 185.141 3.06 | 186.27 3.25 | 617.84 |
| 23 17 | 22.23 | 0.49 400.9 | 196.596 3.05 | 197.74 3.24 | 617.74 |

232 MISSING TUBE(S)

20 21.44 1.35 150.7 203.919 3.05 205.93 3.22 618.39

| | | | | | |
|-------|-------|------------|--------------|-------------|--------|
| 23 21 | 21.53 | 2.05 100.6 | 203.238 3.05 | 204.90 3.23 | 613.85 |
| 23 22 | 20.79 | 0.77 263.2 | 210.449 3.05 | 212.04 3.22 | 620.52 |
| 23 23 | 20.27 | 0.00 0.00 | 215.984 3.05 | 215.72 3.23 | 616.82 |
| 23 24 | 20.74 | 2.41 87.48 | 210.916 3.05 | 214.50 3.20 | 613.20 |
| 23 25 | 20.28 | 1.01 205.6 | 215.728 3.05 | 217.33 3.21 | 617.45 |
| 23 26 | 20.79 | 0.87 234.6 | 210.353 3.05 | 212.23 3.22 | 614.82 |
| 23 27 | 21.00 | 1.61 128.1 | 208.215 3.05 | 210.63 3.22 | 613.67 |
| 23 28 | 20.66 | 2.50 84.72 | 211.698 3.05 | 215.06 3.20 | 615.43 |
| 23 29 | 20.61 | 0.47 430.1 | 212.219 3.05 | 213.08 3.22 | 617.07 |
| 23 30 | 17.43 | 0.00 0.00 | 251.616 3.04 | 252.70 3.19 | 617.25 |
| 23 31 | 18.45 | 1.11 195.1 | 237.517 3.04 | 239.10 3.19 | 617.28 |
| 23 32 | 16.27 | 2.08 113.7 | 269.737 3.04 | 271.71 3.17 | 616.48 |
| 23 33 | 16.25 | 1.12 206.5 | 270.196 3.04 | 272.08 3.17 | 616.64 |
| 23 34 | 16.36 | 0.55 415.0 | 268.356 3.04 | 269.14 3.18 | 615.44 |
| 23 35 | 16.78 | 2.83 83.64 | 261.435 3.04 | 264.17 3.17 | 613.04 |
| 23 36 | 12.21 | 0.10 2656 | 360.724 3.03 | 361.15 3.13 | 615.78 |
| 23 37 | 12.18 | 1.30 206.4 | 361.456 3.03 | 363.67 3.13 | 615.07 |
| 23 38 | 10.51 | 3.87 78.67 | 420.097 3.02 | 425.29 3.09 | 611.36 |
| 23 39 | 10.50 | 3.32 90.65 | 419.929 3.02 | 423.91 3.10 | 610.59 |
| 23 40 | 9.77 | 2.02 150.4 | 451.560 3.02 | 454.21 3.10 | 614.46 |
| 23 41 | 9.31 | 2.69 117.3 | 473.932 3.02 | 477.25 3.09 | 609.41 |
| 23 42 | 8.98 | 2.42 131.9 | 491.474 3.02 | 493.31 3.09 | 615.75 |

05 May 99 19:09 ALPHA/BETA - 1.02
Protocol #:23 U-233 3% 2 sigma

Page 1
User : Alk

| | | | | | |
|-------|------|------------|--------------|-------------|--------|
| 23 43 | 8.89 | 0.85 363.5 | 496.708 3.02 | 498.38 3.09 | 613.62 |
| 23 44 | 9.08 | 1.51 206.0 | 486.244 3.02 | 488.21 3.09 | 614.04 |
| 23 45 | 8.78 | 2.60 124.8 | 502.974 3.02 | 504.65 3.09 | 613.93 |
| 23 46 | 8.77 | 0.00 0.00 | 503.780 3.02 | 504.35 3.09 | 620.90 |
| 23 47 | 8.53 | 0.81 391.5 | 517.698 3.02 | 517.73 3.09 | 615.96 |
| 23 48 | 8.89 | 0.00 0.00 | 496.483 3.02 | 498.60 3.09 | 622.39 |
| 23 49 | 8.79 | 0.00 0.00 | 502.626 3.02 | 502.68 3.10 | 623.62 |
| 23 50 | 9.22 | 4.54 72.54 | 478.809 3.02 | 483.61 3.09 | 624.22 |
| 23 51 | 9.23 | 2.02 154.4 | 478.286 3.02 | 481.43 3.09 | 626.22 |

233 MISSING TUBE(S)

09 May 99 06:57 ALPHA/BETA - 1.02 Page #
Protocol #:23 U-233 3% 2 sigma User : Alk

109 8.87 2.14 149.2 498.062 3.02 502.16 3.08 620.70 3C
23 110 8.70 2.12 152.1 507.860 3.02 511.86 3.08 620.963 QFe20U
23 111 8.86 1.15 271.8 498.176 3.02 501.86 3.09 619.734 QFe20U
23
SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 09-May-99 07:23

C14 Eff (0-156 keV) = 96.67 %

C14 CHI SQUARE IPA DATA PROCESSED - 09-May-99 07:34

C14 Chi Square = 10.20

H3 IPA DATA PROCESSED - 09-May-99 07:35

H3 Eff (0-18.6 keV) = 64.82 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves
& view historic data

H3 CHI SQUARE IPA DATA PROCESSED - 09-May-99 07:45

H3 Chi Square = 23.03

BKG IPA DATA PROCESSED - 09-May-99 08:46

Bkg (0-18.6 keV) = 19.87 cpm

Bkg (0-156 keV) = 29.45 cpm

WARNING: Questionable C14 Background value - Please view historic data

C14 E²/B (1-156 keV) = 408.88

H3 E²/B (1-18.6 keV) = 209.96

21 May 99 01:09 ALPHA/BETA - 1.02 Page
Protocol #:23 U-233 3% 2 sigma User : Al

23 103 10.19 5.01 62.26 433.038 3.02 440.54 3.08 608.35
23 104 9.98 0.00 0.00 442.123 3.02 442.90 3.11 614.75
234 MISSING TUBE(S)
109 8.86 1.60 194.0 498.451 3.02 499.95 3.09 609.35
23 110 8.85 2.76 115.8 498.792 3.02 501.00 3.09 604.57
23 111 8.78 1.80 174.7 503.252 3.02 506.71 3.08 613.05
23 112 9.04 4.50 72.89 488.125 3.02 493.30 3.08 609.26
23 113 8.36 1.79 179.1 528.111 3.02 530.39 3.09 607.11
23 114 8.46 1.30 242.0 522.064 3.02 524.25 3.09 610.47
23 115 8.51 0.36 856.4 519.329 3.02 520.30 3.09 608.63
23 116 8.47 4.82 70.69 521.798 3.02 529.03 3.07 607.26
232 MISSING TUBE(S)
119 10.88 1.34 208.4 405.264 3.02 407.37 3.11 614.95
23 120 10.82 2.01 141.4 407.438 3.03 410.25 3.11 611.45
23
SYSTEM NORMALIZED

UNCAPPED

| PP TUBES 5/17 + 5/18 | | | CONTAINERS | | |
|----------------------|-------------|----------------------|----------------|-------------|----------------------|
| Sample Name | wt. Of vial | wt. Of vial + sample | Sample Name | wt. Of vial | wt. Of vial + sample |
| QFe20U-pH 3a | 7.8840 | 8.3768 | QFe20U-pH 3a | 7.8179 | 8.3166 |
| QFe20U-pH 3b | 7.8741 | 8.3810 | QFe20U-pH 3b | 7.8032 | 8.3036 |
| QFe20U-pH 3.5a | 7.8445 | 8.3441 | QFe20U-pH 3.5a | 7.8458 | 8.3502 |
| QFe20-pH 3.5b | 7.8878 | 8.3889 | QFe20-pH 3.5b | 7.8437 | 8.3439 |
| QFe20-pH 4a | 7.8344 | 8.3343 | QFe20-pH 4a | 7.8782 | 8.3801 |
| QFe20-pH 4b | 7.8358 | 8.3362 | QFe20-pH 4b | 7.813 | 8.3141 |
| QFe20-pH 4.25a | 7.8322 | 8.3343 | QFe20-pH 4.25a | 7.8258 | 8.3278 |
| QFe20-pH 4.25b | 7.7940 | 8.2979 | QFe20-pH 4.25b | 7.8858 | 8.3869 |
| QFe20-pH 4.5a | 7.8256 | 8.3277 | QFe20-pH 4.5a | 7.8432 | 8.3465 |
| QFe20-pH 4.5b | 7.8836 | 8.3881 | QFe20-pH 4.5b | 7.8166 | 8.3193 |
| QFe20-pH 4.75a | 7.8490 | 8.3563 | QFe20-pH 4.75a | 7.8034 | 8.3031 |
| QFe20-pH 4.75b | 7.8160 | 8.3286 | QFe20-pH 4.75b | 7.8543 | 8.3549 |
| QFe20-pH 5a | 7.8408 | 8.3428 | QFe20-pH 5a | 7.7526 | 8.2535 |
| QFe20-pH 5b | 7.9355 | 8.4432 | QFe20-pH 5b | 7.8231 | 8.3242 |
| QFe20-pH 5.25a | 7.8340 | 8.3358 | QFe20-pH 5.25a | 7.7415 | 8.2429 |
| QFe20-pH 5.25b | 7.8108 | 8.3132 | QFe20-pH 5.25b | 7.7546 | 8.253 |
| QFe20-pH 5.5a | 7.8325 | 8.3330 | QFe20-pH 5.5a | 7.6555 | 8.1562 |
| QFe20-pH 5.5b | 7.8834 | 8.3840 | QFe20-pH 5.5b | 7.7444 | 8.2534 |
| QFe20-pH 5.75a | 7.8843 | 8.3863 | QFe20-pH 5.75a | 7.7802 | 8.2803 |
| QFe20-pH 5.75b | 7.8361 | 8.3391 | QFe20-pH 5.75b | 7.782 | 8.2817 |
| QFe20-pH 6a | 7.8700 | 8.3731 | QFe20-pH 6a | 7.8632 | 8.3615 |
| QFe20-pH 6b | 7.8680 | 8.3743 | QFe20-pH 6b | 7.8223 | 8.3208 |
| QFe20-pH 6.25a | 7.8083 | 8.3084 | QFe20-pH 6.25a | 7.7507 | 8.2499 |
| QFe20-pH 6.25b | 7.8901 | 8.3932 | QFe20-pH 6.25b | 7.7828 | 8.2814 |
| QFe20-pH 6.5a | 7.8152 | 8.3162 | QFe20-pH 6.5a | 7.7704 | 8.2708 |
| QFe20-pH 6.5b | 7.7396 | 8.2439 | QFe20-pH 6.5b | 7.7984 | 8.2982 |
| QFe20-pH 6.75a | 7.8918 | 8.3929 | QFe20-pH 6.75a | 7.7992 | 8.2981 |
| QFe20-pH 6.75b | 7.8256 | 8.3278 | QFe20-pH 6.75b | 7.8559 | 8.3552 |
| QFe20-pH 7a | 7.8045 | 8.3061 | QFe20-pH 7a | 7.7856 | 8.2858 |
| QFe20-pH 7b | 7.8360 | 8.3377 | QFe20-pH 7b | 7.8133 | 8.313 |
| QFe20-pH 7.25a | 7.8266 | 8.3299 | QFe20-pH 7.25a | 7.8213 | 8.3215 |
| QFe20-pH 7.25b | 7.7907 | 8.2935 | QFe20-pH 7.25b | 7.8088 | 8.3077 |
| QFe20-pH 7.5a | 7.8125 | 8.3147 | QFe20-pH 7.5a | 7.8167 | 8.3133 |
| QFe20-pH 7.5b | 7.8504 | 8.3538 | QFe20-pH 7.5b | 7.7663 | 8.2658 |
| QFe20-pH 7.75a | 7.7803 | 8.2831 | QFe20-pH 7.75a | 7.7547 | 8.2548 |
| QFe20-pH 7.75b | 7.7849 | 8.2861 | QFe20-pH 7.75b | 7.8218 | 8.3214 |
| QFe20-pH 8a | 7.8702 | 8.3727 | QFe20-pH 8a | 7.7723 | 8.2729 |
| QFe20-pH 8b | 7.7938 | 8.2975 | QFe20-pH 8b | 7.7661 | 8.2664 |
| QFe20-pH 8.25a | 7.8703 | 8.3775 | QFe20-pH 8.25a | 7.8193 | 8.3202 |
| QFe20-pH 8.25b | 7.8508 | 8.3543 | QFe20-pH 8.25b | 7.7473 | 8.2474 |
| QFe20-pH 8.5a | 7.8492 | 8.3518 | QFe20-pH 8.5a | 7.7888 | 8.2888 |
| QFe20-pH 8.5b | 7.8275 | 8.3320 | QFe20-pH 8.5b | 7.7384 | 8.2383 |
| QFe20-pH 8.75a | 7.8469 | 8.3530 | QFe20-pH 8.75a | 7.8013 | 8.3012 |
| QFe20-pH 8.75b | 7.8641 | 8.3678 | QFe20-pH 8.75b | 7.8061 | 8.3055 |

4/19/99

AJ

4/20/99

BET surface area analysis on Quartz crushed to 60/100 mesh size & with iron impurities (QFeU)

Equipment used:- SA 3100 COUTLER analyzer
tube assembly.

Mettler AE240 balance

Liquid N_2 .

- (1) wt. of tube assembly #1 = 33.6475 g
amount of gt2 added = 1.6510 g
wt. of tube assembly + outgassed sample = 35.2956 g
wt. of outgassed sample (QFeU) = 35.2956
- 33.6475
1.6481 g

BET surface area = 0.072 m^2/g

- (2) wt. of tube assembly #4 = 33.4884 g
wt. of gt2 added = 2.2533 g
wt. of tube assembly + outgassed QFeU = 35.7407 g
wt. of outgassed QFeU = 35.7407
33.4884
2.2523 g
BET surface area = 0.052 m^2/g

- (3) wt. of tube assembly #5 = 33.3359 g
wt. of gt2 added = 2.8946 g
wt. of tube assembly + outgassed QFeU = 36.2295 g
wt. of outgassed sample = 36.2295
- 33.3359
2.8936 g
BET surface area = 0.048 m^2/g

4/20/99

AJ

4/21/99

BET surface area analysis on QFeU similarly as above.

- (4) wt. of tube assembly #7 = 33.6457 g
wt. of gt2 added = 2.4969 g
wt. of outgassed sample + assembly = 36.1409 g
wt. of outgassed QFeU sample = 36.1409
- 33.6457
2.4952 g

BET surface area = 0.074 m^2/g

- (5) wt. of tube assembly #9 = 33.2214 g
wt. of gt2 added = 3.5060 g
wt. of outgassed sample + assembly = 36.7252 g
wt. of outgassed QFeU sample = 36.7252
- 33.2214
3.5038 g

BET surface area = 0.056 m^2/g

5/3/99

Prepare 0.1M HNO_3 stock solution using method as on Page 10-11.
(conc. HNO_3 (lot # 983546))

5/4/99

Preparation of reducing agent using same method as on page (19) for SiO_2 detection by Milton Roy Spectronic 1201 spectrophotometer.

| | | |
|-----------------|--------|--------|
| QFe20-pH 9a | 7.8326 | 8.3395 |
| QFe20U-pH 9b | 7.8170 | 8.3203 |
| QFe20-pH 9.25a | 7.8018 | 8.3090 |
| QFe20U-pH 9.25b | 7.8281 | 8.3309 |
| QFe20-pH 9.5a | 7.7857 | 8.2897 |
| QFe20U-pH 9.5b | 7.8686 | 8.3733 |

| | | |
|-----------------|--------|--------|
| QFe20-pH 9a | 7.7181 | 8.2181 |
| QFe20U-pH 9b | 7.8173 | 8.3173 |
| QFe20-pH 9.25a | 7.8439 | 8.3432 |
| QFe20U-pH 9.25b | 7.8254 | 8.3237 |
| QFe20-pH 9.5a | 7.8108 | 8.3083 |
| QFe20U-pH 9.5b | 7.7186 | 8.217 |

UNCAPPED

| Sample Name | 5/3/99 wt. Of PP tubes, g | 5/4/99 wt. Of PP tubes after transfer of qtz/clino, g | 5/4/99 wt. Of PP tubes after acid add, g | 5/4/99 Container wt. after transfer of supernatant, g | Container wt. after supernatant retransfer & acid add, g |
|----------------|---------------------------------|--|--|--|---|
| QFe20U-pH 3 | 13.7806 | 19.0495 | 22.0107 | 27.5662 | 55.0600 |
| QFe20U-pH 3.5 | 13.7510 | 18.5800 | 21.6008 | 27.1504 | 53.2416 |
| QFe20U-pH 4 | 13.6555 | 18.3568 | 21.3674 | 26.9168 | 53.7496 |
| QFe20U-pH 4.25 | 13.7143 | 17.4115 | 20.4142 | 25.9421 | 55.8206 |
| QFe20U-pH 4.5 | 13.8564 | 15.5912 | 18.5647 | 23.9747 | 58.2397 |
| QFe20U-pH 4.75 | 13.6599 | 15.5193 | 18.5320 | 23.9502 | 57.8269 |
| QFe20U-pH 5 | 13.8047 | 15.4623 | 18.4745 | 23.8525 | 55.1063 |
| QFe20U-pH 5.25 | 13.6033 | 15.2454 | 18.2637 | 23.8850 | 58.2803 |
| QFe20U-pH 5.5 | 13.7340 | 17.0582 | 20.0862 | 25.8752 | 56.5647 |
| QFe20U-pH 5.75 | 13.8019 | 15.4662 | 18.4836 | 23.9298 | 58.3461 |
| QFe20U-pH 6 | 13.4095 | 15.8362 | 18.8277 | 24.8650 | 57.4439 |
| QFe20U-pH 6.25 | 13.4005 | 16.2767 | 19.2772 | 25.1433 | 56.9914 |
| QFe20U-pH 6.5 | 13.6896 | 16.6880 | 19.6848 | 25.4738 | 56.9145 |
| QFe20U-pH 6.75 | 13.7788 | 16.4953 | 19.4938 | 25.0007 | 56.8861 |
| QFe20U-pH 7 | 13.7034 | 15.9592 | 18.9580 | 24.5177 | 57.4336 |
| QFe20U-pH 7.25 | 13.7748 | 15.9509 | 18.9509 | 24.3095 | 56.8102 |
| QFe20U-pH 7.5 | 13.7912 | 16.5443 | 19.5722 | 24.8504 | 57.0431 |
| QFe20U-pH 7.75 | 13.7911 | 16.2636 | 19.2834 | 24.7337 | 57.3800 |
| QFe20U-pH 8 | 13.6345 | 16.0852 | 19.0963 | 24.5487 | 57.2900 |
| QFe20U-pH 8.25 | 13.6861 | 16.7580 | 19.7796 | 25.2827 | 56.6660 |
| QFe20U-pH 8.5 | 13.7871 | 16.7388 | 19.7715 | 25.1835 | 56.9078 |
| QFe20U-pH 8.75 | 13.0848 | 15.8161 | 18.8481 | 24.9479 | 57.2540 |
| QFe20U-pH 9 | 13.5024 | 16.9376 | 19.9639 | 25.6387 | 56.5258 |
| QFe20U-pH 9.25 | 13.3253 | 17.1912 | 20.2221 | 26.2375 | 56.8720 |
| QFe20U-pH 9.5 | 13.6433 | 16.1082 | 19.1361 | 24.9548 | 58.9642 |

UNCAPPED DESORPTION

| Sample Name | 5/17/99 Container wt. before sampling | 5/17 Container wt. after sampling | 5/18/99 wt. of PP tubes before sampling | 5/18 wt. Of PP tubes after sampling |
|----------------|---|---|---|---|
| QFe20U-pH 3 | 54.8728 | 53.8627 | 22.0074 | 20.9947 |
| QFe20U-pH 3.5 | 53.1438 | 52.1263 | 21.5963 | 20.5867 |
| QFe20U-pH 4 | 53.6912 | 52.6767 | 21.3632 | 20.3536 |
| QFe20U-pH 4.25 | 55.7619 | 54.7551 | 20.4099 | 19.4092 |
| QFe20U-pH 4.5 | 58.1779 | 57.1685 | 18.5605 | 17.5509 |
| QFe20U-pH 4.75 | 57.7680 | 56.7624 | 18.5281 | 17.4462 |
| QFe20U-pH 5 | 55.0478 | 54.0434 | 18.4707 | 17.4542 |
| QFe20U-pH 5.25 | 58.2219 | 57.2176 | 18.2604 | 17.2191 |
| QFe20U-pH 5.5 | 57.6044 | 56.5234 | 18.3564 | 17.3112 |
| QFe20U-pH 5.75 | 58.2625 | 57.2557 | 18.4794 | 17.4494 |
| QFe20U-pH 6 | 57.3828 | 56.3788 | 18.8184 | 17.7977 |
| QFe20U-pH 6.25 | 56.9319 | 55.9243 | 19.2481 | 18.2391 |
| QFe20U-pH 6.5 | 56.8522 | 55.8469 | 19.6797 | 18.6709 |
| QFe20U-pH 6.75 | 56.8259 | 55.8212 | 19.4884 | 18.4468 |
| QFe20U-pH 7 | 57.3760 | 56.3716 | 18.9476 | 17.9272 |
| QFe20U-pH 7.25 | 56.6801 | 55.6756 | 18.9442 | 17.8862 |
| QFe20U-pH 7.5 | 56.9786 | 55.9672 | 19.5628 | 18.5519 |
| QFe20U-pH 7.75 | 57.3169 | 56.3104 | 19.2696 | 18.2683 |
| QFe20U-pH 8 | 57.1849 | 56.1738 | 19.0856 | 18.0669 |
| QFe20U-pH 8.25 | 56.6110 | 55.6046 | 19.7701 | 18.7125 |
| QFe20U-pH 8.5 | 56.8464 | 55.8401 | 19.7637 | 18.7353 |
| QFe20U-pH 8.75 | 57.1758 | 56.1659 | 18.8392 | 17.7488 |
| QFe20U-pH 9 | 56.4513 | 55.4398 | 19.9563 | 18.9410 |
| QFe20U-pH 9.25 | 56.7327 | 55.724 | 20.2170 | 19.1905 |
| QFe20U-pH 9.5 | 58.8962 | 57.8922 | 19.1286 | 18.1142 |

5/19/99

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 2S% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 19.62 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.47 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 28.94 |

Quench Indicator: SIS
alpha cpm U-233 1st vial bkgnd
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:2S% | CPMB B:2S% | CPMC C:2S% | SIS FLAG |
|-----|-----------------|------------|---------------|---------------|---------------------|
| P# | 1 | 999.99 | 19.62 1.43 | 3.468 3.40 | 28.94 1.18 147.55 |
| 23 | 2 | 13.27 | 0.43 578.0 | 331.875 3.03 | 332.63 3.14 605.40 |
| 23 | 3 | 13.46 | 1.56 162.1 | 326.844 3.03 | 328.42 3.14 602.90 |
| 23 | 4 | 13.08 | 1.33 191.3 | 336.364 3.03 | 337.65 3.14 598.91 |
| 23 | 5 | 13.61 | 3.23 80.61 | 323.057 3.03 | 327.71 3.13 600.97 |
| 23 | 6 | 10.89 | 0.49 554.2 | 405.164 3.02 | 406.41 3.11 601.57 |
| 23 | 7 | 10.97 | 3.72 78.79 | 401.910 3.03 | 406.89 3.10 594.02 |
| 23 | 8 | 6.01 | 5.34 76.49 | 736.798 3.01 | 743.11 3.05 583.84 |
| 23 | 9 | 6.00 | 5.55 73.98 | 737.199 3.01 | 745.56 3.05 584.59 |
| 23 | 10 | 6.22 | 0.64 565.6 | 711.001 3.01 | 714.63 3.06 587.24 |
| 23 | 11 | 6.17 | 2.42 156.3 | 718.088 3.01 | 721.14 3.06 583.71 |
| 23 | 12 | 3.14 | 2.36 224.8 | 1414.366 3.00 | 1418.51 3.03 584.74 |
| 23 | 13 | 3.09 | 0.00 0.00 | 1438.603 3.00 | 1437.08 3.03 586.33 |
| 23 | 14 | 2.12 | 2.55 253.6 | 2099.362 3.00 | 2108.80 3.01 582.36 |
| 23 | 15 | 2.17 | 14.48 54.77 | 2052.753 3.00 | 2066.91 3.01 580.25 |
| 23 | 16 | 2.27 | 2.85 221.0 | 1963.052 3.00 | 1969.74 3.01 580.69 |
| 23 | 17 | 2.24 | 9.40 76.63 | 1989.836 3.00 | 2002.76 3.01 580.65 |
| 232 | MISSING TUBE(S) | | | | |
| 20 | 2.10 | 6.10 114.9 | 2112.723 3.01 | 2118.20 3.02 | 582.66 |
| 23 | 21 | 2.10 | 7.53 95.62 | 2124.151 3.00 | 2131.54 3.01 585.34 |
| 23 | 22 | 2.35 | 7.19 94.02 | 1895.255 3.00 | 1905.10 3.01 587.82 |
| 23 | 23 | 2.34 | 3.46 181.7 | 1897.387 3.00 | 1901.40 3.02 583.62 |
| 23 | 24 | 2.58 | 6.74 94.94 | 1719.013 3.01 | 1727.26 3.02 585.55 |
| 23 | 25 | 2.50 | 5.18 121.7 | 1775.732 3.00 | 1783.06 3.02 587.68 |
| 23 | 26 | 2.67 | 5.48 112.1 | 1666.195 3.00 | 1674.06 3.02 587.16 |
| 23 | 27 | 2.56 | 8.12 81.17 | 1734.032 3.00 | 1742.15 3.02 583.73 |
| 23 | 28 | 2.58 | 9.45 71.08 | 1719.788 3.01 | 1729.98 3.02 586.70 |
| 23 | 29 | 2.53 | 1.33 432.8 | 1755.821 3.00 | 1762.36 3.02 587.38 |
| 23 | 30 | 2.29 | 14.44 53.44 | 1941.510 3.00 | 1954.90 3.01 581.92 |
| 23 | 31 | 2.25 | 7.94 88.24 | 1972.976 3.00 | 1984.39 3.01 582.01 |
| 23 | 32 | 2.37 | 6.54 101.6 | 1879.654 3.00 | 1887.09 3.01 585.03 |
| 23 | 33 | 2.50 | 10.38 66.78 | 1778.132 3.00 | 1788.66 3.02 583.22 |
| 23 | 34 | 3.33 | 8.91 65.77 | 1333.469 3.01 | 1343.43 3.02 580.91 |
| 23 | 35 | 3.27 | 6.99 81.73 | 1360.141 3.00 | 1365.56 3.02 579.44 |
| 23 | 36 | 2.08 | 12.11 64.53 | 2145.571 3.00 | 2159.52 3.00 581.88 |
| 23 | 37 | 2.09 | 13.40 59.37 | 2126.676 3.00 | 2139.00 3.01 582.19 |
| 23 | 38 | 5.73 | 2.55 154.8 | 772.274 3.01 | 777.52 3.05 581.24 |
| 23 | 39 | 5.47 | 0.13 3003 | 809.878 3.01 | 811.28 3.06 582.21 |
| 23 | 40 | 9.68 | 0.63 460.7 | 455.623 3.02 | 457.11 3.10 586.85 |
| 23 | 41 | 9.90 | 5.43 58.78 | 445.623 3.02 | 453.59 3.08 580.04 |
| 23 | 42 | 14.26 | 0.23 1040 | 308.173 3.03 | 310.96 3.14 589.10 |

20 May 99 12:16
Protocol #:23

ALPHA/BETA - 1.02
U-233 3% 2 sigma

Page
User : A

| | | | | | |
|-----|-----------------|------------|--------------|--------------|--------------------|
| 23 | 43 | 14.48 | 0.13 1753 | 303.438 3.03 | 304.07 3.16 589.33 |
| 23 | 44 | 16.83 | 3.50 67.51 | 260.584 3.04 | 265.12 3.16 580.97 |
| 23 | 45 | 16.85 | 1.39 161.7 | 260.330 3.04 | 262.75 3.17 584.70 |
| 23 | 46 | 15.48 | 3.12 78.16 | 283.612 3.04 | 288.11 3.14 580.38 |
| 23 | 47 | 15.59 | 1.04 223.6 | 281.586 3.04 | 284.53 3.15 586.17 |
| 23 | 48 | 15.62 | 3.43 71.29 | 281.295 3.04 | 285.15 3.15 583.60 |
| 23 | 49 | 15.33 | 1.45 162.6 | 286.812 3.03 | 289.59 3.15 588.15 |
| 23 | 50 | 21.99 | 1.85 108.0 | 198.624 3.05 | 202.76 3.21 585.24 |
| 23 | 51 | 22.03 | 2.85 71.50 | 198.257 3.05 | 201.79 3.21 580.43 |
| 233 | MISSING TUBE(S) | | | | |
| 55 | 9.58 | 1.16 256.1 | 460.833 3.02 | 463.02 3.10 | 609.75 |
| 23 | 56 | 9.70 | 1.72 172.9 | 454.676 3.02 | 456.83 3.10 611.23 |
| 23 | 57 | 9.58 | 2.93 105.2 | 460.624 3.02 | 464.07 3.09 607.09 |
| 23 | 58 | 9.68 | 1.46 203.4 | 455.933 3.02 | 456.39 3.10 603.29 |
| 23 | 59 | 9.88 | 2.85 106.2 | 446.330 3.02 | 449.40 3.10 609.08 |
| 23 | 60 | 9.62 | 3.46 89.90 | 458.694 3.02 | 463.47 3.09 604.55 |
| 23 | 61 | 10.02 | 0.44 642.4 | 440.045 3.02 | 441.02 3.11 608.80 |
| 23 | 62 | 9.93 | 0.02 13948 | 444.568 3.02 | 445.68 3.10 613.10 |
| 23 | 63 | 10.76 | 1.57 179.4 | 409.636 3.03 | 412.23 3.11 610.10 |
| 23 | 64 | 10.80 | 0.11 2587 | 408.106 3.03 | 408.38 3.12 609.13 |
| 23 | 65 | 12.66 | 0.60 421.1 | 347.559 3.03 | 348.55 3.13 612.50 |
| 23 | 66 | 12.77 | 0.66 381.5 | 344.535 3.03 | 346.47 3.13 607.44 |
| 23 | 67 | 26.33 | 1.92 95.47 | 165.313 3.06 | 167.07 3.27 607.84 |
| 23 | 68 | 26.38 | 1.54 118.1 | 165.069 3.06 | 167.54 3.26 609.69 |
| 23 | 69 | 15.23 | 3.23 76.28 | 288.324 3.04 | 293.71 3.14 601.86 |
| 23 | 70 | 15.13 | 2.66 91.96 | 290.319 3.04 | 293.47 3.15 605.67 |
| 232 | MISSING TUBE(S) | | | | |
| 73 | 15.64 | 0.59 389.9 | 280.739 3.04 | 283.21 3.16 | 608.75 |
| 23 | 74 | 16.40 | 0.00 0.00 | 267.569 3.04 | 268.32 3.18 608.65 |
| 23 | 75 | 16.15 | 1.81 128.4 | 271.764 3.04 | 274.09 3.16 605.45 |
| 23 | 76 | 16.11 | 2.42 97.40 | 272.385 3.04 | 275.03 3.16 601.90 |
| 23 | 77 | 15.87 | 3.89 63.04 | 276.557 3.04 | 281.39 3.15 601.11 |
| 23 | 78 | 15.93 | 2.54 93.44 | 275.628 3.04 | 278.59 3.16 606.15 |
| 23 | 79 | 14.89 | 1.34 178.8 | 295.054 3.04 | 297.05 3.15 605.29 |
| 23 | 80 | 15.47 | 0.74 310.4 | 284.056 3.04 | 286.57 3.15 608.72 |
| 23 | 81 | 15.51 | 0.24 946.4 | 283.186 3.04 | 284.54 3.16 608.07 |
| 23 | 82 | 15.28 | 0.08 2799 | 287.435 3.04 | 287.81 3.17 608.58 |
| 23 | 83 | 15.85 | 2.91 82.59 | 276.910 3.04 | 280.27 3.15 603.16 |
| 23 | 84 | 15.84 | 0.65 351.8 | 277.151 3.04 | 278.13 3.17 608.58 |
| 23 | 85 | 14.64 | 1.97 124.2 | 300.220 3.03 | 302.48 3.15 606.06 |
| 23 | 86 | 14.71 | 2.88 86.30 | 298.775 3.03 | 302.54 3.14 601.99 |
| 23 | 87 | 13.72 | 3.49 74.85 | 320.512 3.03 | 325.43 3.13 600.40 |
| 23 | 88 | 13.79 | 1.12 219.9 | 318.794 3.03 | 320.95 3.14 605.03 |
| 23 | 89 | 12.56 | 1.80 145.9 | 350.433 3.03 | 352.51 3.13 604.78 |
| 23 | 90 | 12.48 | 4.50 62.08 | 353.022 3.03 | 357.84 3.11 606.74 |
| 23 | 91 | 11.10 | 2.27 124.1 | 397.343 3.02 | 399.62 3.11 603.65 |
| 23 | 92 | 10.64 | 2.19 131.5 | 414.577 3.02 | 417.68 3.10 607.18 |

| | | | | | | | | | |
|----|-----|------|------|-------|---------|------|--------|------|--------|
| 23 | 93 | 9.90 | 0.89 | 325.7 | 445.724 | 3.02 | 446.41 | 3.11 | 604.94 |
| 23 | 94 | 9.63 | 0.42 | 683.1 | 458.318 | 3.02 | 459.33 | 3.10 | 605.75 |
| 23 | 95 | 9.64 | 0.00 | 0.00 | 457.735 | 3.02 | 457.89 | 3.10 | 606.10 |
| 23 | 96 | 9.83 | 2.36 | 127.5 | 448.923 | 3.02 | 452.55 | 3.09 | 604.09 |
| 23 | 97 | 9.29 | 1.59 | 191.0 | 474.896 | 3.02 | 478.06 | 3.09 | 605.75 |
| 23 | 98 | 9.41 | 2.91 | 106.7 | 468.796 | 3.02 | 473.40 | 3.09 | 606.52 |
| 23 | 99 | 9.45 | 1.02 | 291.7 | 467.114 | 3.02 | 467.36 | 3.10 | 604.90 |
| 23 | 100 | 9.55 | 0.70 | 420.5 | 462.396 | 3.02 | 463.31 | 3.10 | 608.66 |
| 23 | 101 | 9.54 | 0.30 | 971.2 | 462.465 | 3.02 | 462.36 | 3.11 | 614.56 |
| 23 | 102 | 9.48 | 0.85 | 348.5 | 465.308 | 3.02 | 467.26 | 3.10 | 610.56 |

231 MISSING TUBE(S)

9 10.03 1.75 166.9 439.804 3.02 441.98 3.10 608.00

| | | | | | | | | | |
|----|----|-------|------|-------|---------|------|--------|------|--------|
| 23 | 10 | 10.31 | 3.02 | 98.18 | 428.060 | 3.02 | 431.44 | 3.10 | 608.01 |
| 23 | 11 | 26.30 | 1.94 | 93.87 | 165.607 | 3.06 | 168.32 | 3.26 | 607.83 |
| 23 | 12 | 25.55 | 2.37 | 78.69 | 170.607 | 3.06 | 173.75 | 3.25 | 608.78 |
| 23 | 13 | 12.53 | 1.20 | 214.7 | 351.383 | 3.03 | 352.72 | 3.13 | 603.28 |
| 23 | 14 | 12.76 | 2.32 | 113.1 | 345.067 | 3.03 | 348.43 | 3.12 | 605.88 |
| 23 | 15 | 10.00 | 1.11 | 259.0 | 441.634 | 3.02 | 441.30 | 3.11 | 611.93 |
| 23 | 16 | 9.93 | 4.28 | 72.49 | 444.368 | 3.02 | 449.44 | 3.09 | 605.99 |

232 MISSING TUBE(S)

19 6.12 2.34 161.3 722.778 3.01 725.32 3.06 585.24

| | | | | | | | | | |
|----|----|-------|-------|-------|----------|------|---------|------|--------|
| 23 | 20 | 6.03 | 0.00 | 0.00 | 733.616 | 3.01 | 733.59 | 3.07 | 586.31 |
| 23 | 21 | 2.20 | 0.61 | 988.3 | 2026.179 | 3.00 | 2027.76 | 3.02 | 579.57 |
| 23 | 22 | 2.10 | 6.80 | 103.9 | 2118.539 | 3.00 | 2125.68 | 3.01 | 578.91 |
| 23 | 23 | 2.07 | 11.05 | 69.47 | 2151.223 | 3.00 | 2161.73 | 3.01 | 578.39 |
| 23 | 24 | 2.09 | 6.93 | 102.5 | 2128.213 | 3.00 | 2138.38 | 3.01 | 581.27 |
| 23 | 25 | 21.55 | 2.88 | 71.15 | 202.898 | 3.05 | 206.90 | 3.20 | 580.61 |
| 23 | 26 | 21.90 | 1.80 | 110.5 | 199.602 | 3.05 | 202.08 | 3.22 | 582.94 |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 26-May-99 19:02

C14 Eff (0-156 keV) = 96.45 %

C14 CHI SQUARE IPA DATA PROCESSED - 26-May-99 19:13

C14 Chi Square = 15.65

H3 IPA DATA PROCESSED - 26-May-99 19:14

H3 Eff (0-18.6 keV) = 64.74 %

WARNING: Questionable H3 Efficiency value - Please rerun quench curves
& view historic data

Experimental Procedure for experiment - U sorption onto clinoptilolite at 25° C.

CAPPED C20U

Objective: To investigate the characteristics of U sorption onto quartz as a function of pH.

Initial conditions:

- $\sum U = 50$ ppb (2.14×10^{-7})
- Capped containers
- pH range 3.0-9.5
- solution volume = 35 ml
- mass of solid 1, quartz = 1.000 g
- mass of solid 2, clinoptilolite = 0.000 g
- M/V = 28.57 g/L

Equipment:

- Orion 920A pH meter and combination pH electrode
- Gyratory shaker
- Glove box
- Fisher Marathon 21K centrifuge
- Mettler PM4600 and AE240 balances
- Packard 2500 TR/AB LSC
- Eppendorf micropipettors and tips
- Oxford pipettes and tips
- Repipettor for transfer of scintillation cocktail

Supplies:

- W510 *UC* RFe *RC* HL * ≤ 325 mesh AJ
- 50 ppb ^{233}U stock solution
- polycarbonate centrifuge tubes with caps (50 ml capacity)
- polypropylene centrifuge tubes with caps (50 ml capacity)
- pH buffer solutions
- Ultima-Gold liquid scintillation cocktail
- 7 ml scintillation vials
- reagent grade NaOH (lot 980698)
- concentrated HNO_3 (lot 983546)
- HNO_3 and NaOHdegassed stock solutions at various concentrations
- weighing paper/boats
- deionized ultrapure water, DH_2O
- teflon beaker

Note: Ultrapure water was degassed by boiling it in a flask and cooled with mouth covered with rubber stopper.

Procedure:

- A. Prepare separate 50 ppb U solutions for sorption experiments.
- On the Metler balance, weigh 200 g of 500 ppb U stock solution into a tared 2000 ml FEP bottle.
 - Dilute to a total of 2000 g using degassed 0.1M NaNO_3 stock solution.
 - Cap and label bottle accordingly.

B. Transfer (2.14×10^{-7}) 50 ppb ^{233}U stock solution to experimental containers.

- Label 26 polycarbonate centrifuge tubes (50 ml capacity) C20U-pHi (where i is the approximate pH of each solution, see Table C20U).
- Label 1 polycarbonate centrifuge tube C20U-IU.
- Weigh and record the weight of each container.
- About 1L of ^{233}U solution will be required for this experiment.
- Add ~35 g of the 50 ppb ^{233}U stock solution to each container.

Note: the stock solution should be transferred to a teflon beaker for easier dispensing into the sample containers.

- Record weight of each container.

C. Add quartz to sample containers.

- Add ~1.000 g of quartz to each sample container. **Do not** add quartz to the C20U-IU container.
- Record weight of each sample container after addition of quartz.

D. Adjust pH of each solution by adding HNO_3 or NaOH .

- Add the volume of HNO_3 or NaOH to each experimental container as listed in Table C20U. Take care to add HNO_3 or NaOH in the concentrations specified in Table C20U.
- For sample C20U-IU (not listed in Table C20U) add 100 μl of 50/50 V/V HNO_3 to the sample container.
- Weigh and record the weight of each sample container.

E. Sample CQ0U-IU for initial [U].

- Label two 7 ml LSC vials as C20U-IUa and C20U-IUb.
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Transfer 0.5 ml of solution from C20U-IU to each sample vial.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of container C20U-IU

F. Wait for equilibrium.

- Place caps on sample containers but do not tighten the caps.
- Place containers inside the glove box. Tighten the caps after 2 days.
- Place containers on gyratory shaker set at about 120 rpm and allow samples to equilibrate for at least 10 days.

G. Sample experimental solutions for [U] and pH.

- For each sample solution label two 7 ml LSC vials (e.g., C20U-pH3a and C20U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container.
- From each sample container withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container.
- Measure pH of each experimental solution.
- Record weight of each sample container after pH measurement.

H. Transfer quartz for desorption.

- Record weight of sample containers. *Note: this step can be skipped if the clinoptilolite transfer is done immediately after the last step in section F.*
- Centrifuge polycarbonate tubes containing experimental solutions at about 5,000 rpm for 5 min to remove fine particles from suspension.
- For each sample container, label a corresponding polypropylene (PP) test tube (50 ml capacity) (e.g., C20dU-pH3 for sample container C20U-pH3).
- Record weight of PP test tubes.
- Using an Eppendorf or Oxford pipette, withdraw quartz from the polycarbonate sample containers and transfer to the corresponding PP test tubes.
- Record weights of sample containers and PP test tubes.
- Add approximately 3 ml of 0.1 M HNO_3 to both experimental containers and PP test tubes and mix thoroughly.
- Record weights of sample containers and PP test tubes.
- Place sample containers and PP test tubes on gyratory shaker (at about 120 rpm) and allow to desorb for at least 10 days.

I. Sample solutions for [U].

- For each sample container and PP test tube label two 7 ml LSC vials (e.g., C20U-pH3a and C20U-pH3b).
- Add 0.5 ml of 0.02 M HNO_3 to each vial.
- Record weight of each vial.
- Record weight of each sample container and PP test tube.
- From each sample container and PP test tube withdraw two 0.5 ml aliquots of solution and transfer to the appropriately labeled LSC vials.
- Record weight of each vial.
- Add 5 ml of Ultima-Gold cocktail to each vial, mix, and set aside for counting.
- Record weight of each sample container and PP test tube.

Table CQ0U: Estimated solution pH and volumes of HNO₃ or NaOH solutions needed for adjustment of pH in 0.1 m NaNO₃ solutions with 50 ppb U in contact with qz/clino (35 ml and capped)

| Estimated solution pH | Volume of HNO ₃ added, ml | Molarity of HNO ₃ to use |
|-----------------------|--------------------------------------|-------------------------------------|
| 3 | 0.045 | 1 |
| 3.25 | 0.254 | 1 |
| 3.5 | 0.143 | 0.1 |
| 3.75 | 0.080 | 0.1 |
| 4 | 0.452 | 0.01 |
| 4.25 | 0.254 | 0.01 |
| 4.5 | 0.143 | 0.01 |
| 4.75 | 0.081 | 0.01 |
| 5 | 0.045 | 0.01 |
| 5.25 | 0.025 | 0.01 |
| 5.5 | 0.140 | 0.001 |
| 5.75 | 0.073 | 0.001 |
| 6 | 0.035 | 0.001 |
| 6.25 | 0.140 | 0.001 |
| 6.5 | 0.010 | 0.001 |
| Estimated solution pH | Volume of NaOH added, ml | Molarity of NaOH to use |
| 6.75 | 0.007 | 0.001 |
| 7 | 0.012 | 0.001 |
| 7.25 | 0.018 | 0.001 |
| 7.5 | 0.026 | 0.001 |
| 7.75 | 0.038 | 0.001 |
| 8 | 0.059 | 0.001 |
| 8.25 | 0.095 | 0.001 |
| 8.5 | 0.160 | 0.001 |
| 8.75 | 0.027 | 0.01 |
| 9 | 0.047 | 0.01 |
| 9.25 | 0.082 | 0.01 |
| 9.5 | 0.145 | 0.01 |

CAPPED

3/31/99

| SAMPLE NAME | Wt. of container, g |
|--------------|---------------------|
| C20U-pH 3 | 22.1267 |
| C20U-pH 3.5 | 21.9274 |
| C20U-pH 4 | 22.1862 |
| C20U-pH 4.25 | 22.5285 |
| C20U-pH 4.5 | 22.212 |
| C20U-pH 4.75 | 22.1244 |
| C20U-pH 5 | 22.2846 |
| C20U-pH 5.25 | 22.3162 |
| C20U-pH 5.5 | 22.5059 |
| C20U-pH 5.75 | 21.9452 |
| C20U-pH 6 | 22.2562 |
| C20U-pH 6.25 | 22.2638 |
| C20U-pH 6.5 | 22.4589 |
| C20U-pH 6.75 | 22.1417 |
| C20U-pH 7 | 22.3031 |
| C20U-pH 7.25 | 22.1862 |
| C20U-pH 7.5 | 22.0453 |
| C20U-pH 7.75 | 22.1308 |
| C20U-pH 8 | 22.1459 |
| C20U-pH 8.25 | 22.0033 |
| C20U-pH 8.5 | 22.2792 |
| C20U-pH 8.75 | 22.1029 |
| C20U-pH 9 | 22.3603 |
| C20U-pH 9.25 | 21.9743 |
| C20U-pH 9.5 | 22.3189 |
| C20U-IU | 22.4011 |

CAPPED

4/5/99

| SAMPLE NAME | WT. OF CONTAINER +CLINOP , g |
|--------------|------------------------------|
| C20U-pH 3 | 22.8383 |
| C20U-pH 3.5 | 22.6617 |
| C20U-pH 4 | 22.8951 |
| C20U-pH 4.25 | 23.2367 |
| C20U-pH 4.5 | 22.9218 |
| C20U-pH 4.75 | 22.8343 |
| C20U-pH 5 | 23.0304 |
| C20U-pH 5.25 | 23.0262 |
| C20U-pH 5.5 | 23.2803 |
| C20U-pH 5.75 | 22.8420 |
| C20U-pH 6 | 22.9668 |
| C20U-pH 6.25 | 22.7041 |
| C20U-pH 6.5 | 23.1895 |
| C20U-pH 6.75 | 22.8522 |
| C20U-pH 7 | 23.0145 |
| C20U-pH 7.25 | 22.8969 |
| C20U-pH 7.5 | 22.9751 |
| C20U-pH 7.75 | 22.7293 |
| C20U-pH 8 | 22.7536 |
| C20U-pH 8.25 | 22.8192 |
| C20U-pH 8.5 | 22.9898 |
| C20U-pH 8.75 | 22.8148 |
| C20U-pH 9 | 22.8562 |
| C20U-pH 9.25 | 22.7913 |
| C20U-pH 9.5 | 23.0304 |
| | |
| C20U-IU | 48.5970 |

CAPPED

4/5/99

| SAMPLE NAME | WT. OF CONTAINER + U SOLN., g |
|--------------|-------------------------------|
| C20U-pH 3 | 57.8638 |
| C20U-pH 3.5 | 57.6675 |
| C20U-pH 4 | 57.9024 |
| C20U-pH 4.25 | 58.2645 |
| C20U-pH 4.5 | 57.9079 |
| C20U-pH 4.75 | 57.7982 |
| C20U-pH 5 | 57.9889 |
| C20U-pH 5.25 | 58.0234 |
| C20U-pH 5.5 | 58.3124 |
| C20U-pH 5.75 | 57.9029 |
| C20U-pH 6 | 57.9604 |
| C20U-pH 6.25 | 57.7163 |
| C20U-pH 6.5 | 58.2092 |
| C20U-pH 6.75 | 57.8425 |
| C20U-pH 7 | 57.8228 |
| C20U-pH 7.25 | 57.9521 |
| C20U-pH 7.5 | 57.9877 |
| C20U-pH 7.75 | 57.7654 |
| C20U-pH 8 | 57.7739 |
| C20U-pH 8.25 | 57.8679 |
| C20U-pH 8.5 | 58.0319 |
| C20U-pH 8.75 | 57.9461 |
| C20U-pH 9 | 57.8662 |
| C20U-pH 9.25 | 58.1328 |
| C20U-pH 9.5 | 58.0208 |

~~UNCAPPED~~

AJ

4/5/99

| SAMPLE NAME | WT. OF CONTAINER AFTER ADDITIONS OF HNO ₃ /NAHCO ₃ , g |
|--------------|---|
| C20U-pH 3 | 57.9315 |
| C20U-pH 3.5 | 57.8690 |
| C20U-pH 4 | 58.3035 |
| C20U-pH 4.25 | 58.4661 |
| C20U-pH 4.5 | 57.9672 |
| C20U-pH 4.75 | 57.8727 |
| C20U-pH 5 | 58.0872 |
| C20U-pH 5.25 | 58.1712 |
| C20U-pH 5.5 | 58.4853 |
| C20U-pH 5.75 | 58.0993 |
| C20U-pH 6 | 58.2080 |
| C20U-pH 6.25 | 57.9882 |
| C20U-pH 6.5 | 58.5058 |
| C20U-pH 6.75 | 58.1879 |
| C20U-pH 7 | 58.2167 |
| C20U-pH 7.25 | 58.0504 |
| C20U-pH 7.5 | 58.1105 |
| C20U-pH 7.75 | 57.9119 |
| C20U-pH 8 | 57.9454 |
| C20U-pH 8.25 | 58.0904 |
| C20U-pH 8.5 | 58.1055 |
| C20U-pH 8.75 | 58.0436 |
| C20U-pH 9 | 58.0155 |
| C20U-pH 9.25 | 58.3291 |
| C20U-pH 9.5 | 58.3033 |

CAPPED

4/29, 4/30, 5/10/99, 5/24/99

| Sample Name | Container wt. before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements |
|--------------|---|------------------------|---|
| C20U-pH 3 | 57.4055 | 2.45 | 56.3848 |
| C20U-pH 3.5 | 57.3785 | 3.37 | 56.3686 |
| C20U-pH 4 | 57.5555 | 2.92 | 56.5303 |
| C20U-pH 4.25 | 58.0125 | 4.66 | 57.9991 |
| C20U-pH 4.5 | 57.5241 | 5.10 | 56.5088 |
| C20U-pH 4.75 | 57.2056 | 5.24 | 56.1803 |
| C20U-pH 5 | 57.4179 | 5.40 | 56.4122 |
| C20U-pH 5.25 | 57.7551 | 4.08 | 56.7656 |
| C20U-pH 5.5 | 57.9162 | 4.19 | 56.9019 |
| C20U-pH 5.75 | 57.6004 | 5.68 | 56.5901 |
| C20U-pH 6 | 57.5813 | 5.82 | 56.5510 |
| C20U-pH 6.25 | 57.4328 | 6.75 | 56.3976 |
| C20U-pH 6.5 | 58.0057 | 4.6 ^{5/24} AJ | 56.8038 |
| C20U-pH 6.75 | 57.7474 | 4.42 | 55.9628 |
| C20U-pH 7 | 57.6769 | 6.39 | 56.7289 |
| C20U-pH 7.25 | 57.6765 | 4.66 | 56.6336 |
| C20U-pH 7.5 | 57.3905 | 7.46 | 56.6638 |
| C20U-pH 7.75 | 57.3888 | 7.23 | 56.3688 |
| C20U-pH 8 | 57.3055 | 6.92 | 56.3769 |
| C20U-pH 8.25 | 57.6680 | 7.62 | 56.2698 |
| C20U-pH 8.5 | 57.4445 | 7.91 | 56.6303 |
| C20U-pH 8.75 | 57.4760 | 8.21 | 56.4082 |
| C20U-pH 9 | 57.8340 | 8.84 | 56.4487 |
| C20U-pH 9.25 | 57.5060 | 9.04 | 56.6275 |
| C20U-pH 9.5 | 58.6553 | 9.53 | 56.5122 |

| Sample Name | wt. Of vial ^{4/26/99} (CAPPED) | wt. Of vial + sample ^{4/30/99} |
|--------------|---|---|
| C20-pH 3a | 7.8064 | 8.2961 |
| C20-pH 3b | 7.8985 | 8.3429 |
| C20-pH 4a | 7.9167 | 8.3995 |
| C20-pH 4b | 7.9096 | 8.4004 |
| C20-pH 4.25a | 7.8719 | 8.3517 |
| C20-pH 4.25b | 7.8038 | 8.2748 |
| C20-pH 4.5a | 7.9112 | 8.3965 |
| C20-pH 4.5b | 7.9250 | 8.4091 |
| C20-pH 4.75a | 7.8973 | 8.3789 |
| C20-pH 4.75b | 7.9356 | 8.4094 |
| C20-pH 5a | 7.8141 | 8.2858 |
| C20-pH 5b | 7.8917 | 8.3782 |
| C20-pH 5.25a | 7.7967 | 8.2556 |
| C20-pH 5.25b | 7.9470 | 8.4249 |
| C20-pH 5.5a | 7.8722 | 8.3534 |
| C20-pH 5.5b | 7.9220 | 8.4088 |
| C20-pH 5.75a | 7.8930 | 8.3767 |
| C20-pH 5.75b | 7.8440 | 8.3305 |
| C20-pH 6a | 7.8118 | 8.3070 |
| C20-pH 6b | 7.8936 | 8.3787 |
| C20-pH 6.25a | 7.8408 | 8.3243 |
| C20-pH 6.25b | 7.8418 | 8.3293 |
| C20-pH 6.5a | ^{AT} 7.7532 | 8.2284 |
| C20-pH 6.5b | ^{5/24} 7.8576 | 8.3536 |
| C20-pH 6.75a | 7.8507 | 8.3378 |
| C20-pH 6.75b | 7.8356 | 8.3291 |
| C20-pH 7a | 7.8289 | 8.3130 |
| C20-pH 7b | 7.8475 | 8.3356 |
| C20-pH 7.25a | 7.7343 | 8.2200 |
| C20-pH 7.25b | 7.9190 | 8.4074 |
| C20-pH 7.5a | 7.8098 | 8.2923 |
| C20-pH 7.5b | 7.8210 | 8.3078 |
| C20-pH 7.75a | 7.8465 | 8.3284 |
| C20-pH 7.75b | 7.8347 | 8.3195 |
| C20-pH 8a | 7.8639 | 8.3511 |
| C20-pH 8b | 7.7894 | 8.2725 |
| C20-pH 8.25a | 7.8828 | 8.3644 |
| C20-pH 8.25b | 7.8332 | 8.3193 |
| C20-pH 8.5a | 7.8903 | 8.3771 |
| C20-pH 8.5b | 7.8167 | 8.2995 |

| | | |
|--------------|--------|--------|
| C20-pH 8.75a | 7.8593 | 8.3267 |
| C20-pH 8.75b | 7.8264 | 8.3145 |
| C20-pH 9a | 7.8342 | 8.3191 |
| C20-pH 9b | 7.8046 | 8.3088 |
| C20-pH 9.25a | 7.8351 | 8.3173 |
| C20-pH 9.25b | 7.8792 | 8.3674 |
| C20-pH 9.5a | 7.8232 | 8.3082 |
| C20-pH 9.5b | 7.7822 | 8.2673 |
| | | |
| | | |
| | | |

| | | | | | | | | | |
|-----|-----------------|--------|-------|---------|---------|--------|--------|--------|--------|
| 55 | 8.65 | 0.00 | 0.00 | 510.584 | 3.02 | 511.06 | 3.09 | 614.26 | |
| 23 | 56 | 8.50 | 2.76 | 119.6 | 519.655 | 3.02 | 522.60 | 3.09 | 612.57 |
| 23 | 57 | 10.43 | 0.51 | 556.5 | 422.675 | 3.02 | 422.93 | 3.12 | 614.15 |
| 23 | 58 | 10.89 | 0.00 | 0.00 | 404.860 | 3.02 | 405.10 | 3.12 | 621.23 |
| 23 | 59 | 9.15 | 0.00 | 0.00 | 482.498 | 3.02 | 482.61 | 3.10 | 614.85 |
| 23 | 60 | 8.95 | 1.27 | 245.7 | 493.691 | 3.02 | 494.62 | 3.10 | 611.34 |
| 23 | 61 | 36.02 | 0.00 | 0.00 | 119.972 | 3.09 | 119.29 | 3.42 | 616.05 |
| 23 | 62 | 36.11 | 1.28 | 123.0 | 119.747 | 3.09 | 121.49 | 3.38 | 610.88 |
| 23 | 63 | 69.56 | 0.00 | 0.00 | 60.483 | 3.17 | 60.84 | 3.79 | 621.65 |
| 23 | 64 | 70.26 | 0.00 | 0.00 | 59.847 | 3.18 | 60.14 | 3.80 | 614.38 |
| 23 | 65 | 70.25 | 0.00 | 0.00 | 59.856 | 3.18 | 59.74 | 3.82 | 616.02 |
| 23 | 66 | 69.22 | 0.00 | 0.00 | 60.797 | 3.17 | 60.92 | 3.79 | 619.81 |
| 23 | 67 | 109.18 | 0.00 | 0.00 | 37.309 | 3.29 | 37.27 | 4.30 | 635.80 |
| 23 | 68 | 105.88 | 0.26 | 361.2 | 38.568 | 3.28 | 39.48 | 4.18 | 613.59 |
| 23 | 69 | 51.54 | 0.00 | 0.00 | 82.840 | 3.13 | 83.42 | 3.57 | 621.30 |
| 23 | 70 | 49.48 | 0.00 | 0.00 | 86.410 | 3.12 | 85.92 | 3.58 | 620.96 |
| 23 | 71 | 57.04 | 0.90 | 139.0 | 74.506 | 3.14 | 75.53 | 3.62 | 609.05 |
| 23 | 72 | 58.06 | 0.00 | 0.00 | 73.137 | 3.14 | 73.29 | 3.66 | 617.24 |
| 23 | 73 | 106.67 | 0.00 | 0.00 | 38.257 | 3.28 | 38.25 | 4.26 | 623.71 |
| 23 | 74 | 109.03 | 0.03 | 3085 | 37.355 | 3.29 | 38.06 | 4.23 | 619.62 |
| 23 | 75 | 118.18 | 1.14 | 78.91 | 34.200 | 3.32 | 35.81 | 4.26 | 602.74 |
| 23 | 76 | 127.38 | 1.15 | 75.38 | 31.499 | 3.34 | 33.33 | 4.34 | 603.38 |
| 23 | 77 | 154.58 | 0.22 | 358.1 | 25.351 | 3.43 | 25.92 | 4.81 | 609.39 |
| 23 | 78 | 156.80 | 0.00 | 0.00 | 24.951 | 3.44 | 25.48 | 4.84 | 620.51 |
| 234 | MISSING TUBE(S) | | | | | | | | |
| 83 | 127.08 | 0.22 | 388.5 | 31.566 | 3.34 | 32.33 | 4.44 | 618.78 | 79 |
| 23 | 84 | 125.75 | 0.08 | 1087 | 31.936 | 3.34 | 32.27 | 4.47 | 619.15 |
| 232 | MISSING TUBE(S) | | | | | | | | |

5/5/99

C20
Capped
Position

6.25b

| | | | | | | | | | |
|-----|-----------------|--------|------|--------|--------|-------|-------|--------|--------|
| 87 | 112.54 | 0.00 | 0.00 | 36.084 | 3.30 | 36.09 | 4.34 | 623.86 | 7.59 |
| 23 | 88 | 108.33 | 0.00 | 0.00 | 37.628 | 3.29 | 37.58 | 4.29 | 627.32 |
| 23 | 89 | 120.38 | 0.36 | 246.0 | 33.512 | 3.32 | 34.15 | 4.38 | 616.91 |
| 23 | 90 | 121.36 | 0.00 | 0.00 | 33.214 | 3.33 | 33.21 | 4.45 | 636.29 |
| 23 | 91 | 111.21 | 0.12 | 774.2 | 36.556 | 3.30 | 37.14 | 4.27 | 616.43 |
| 23 | 92 | 115.42 | 0.20 | 455.8 | 35.099 | 3.31 | 35.64 | 4.33 | 616.41 |
| 23 | 93 | 93.34 | 0.00 | 0.00 | 44.218 | 3.24 | 44.57 | 4.07 | 625.71 |
| 23 | 94 | 91.64 | 0.00 | 0.00 | 45.101 | 3.24 | 45.78 | 4.03 | 623.63 |
| 23 | 95 | 106.94 | 0.41 | 226.7 | 38.152 | 3.28 | 39.11 | 4.19 | 614.93 |
| 23 | 96 | 110.01 | 0.00 | 0.00 | 37.010 | 3.29 | 37.43 | 4.27 | 618.02 |
| 23 | 97 | 142.51 | 0.00 | 0.00 | 27.794 | 3.39 | 28.37 | 4.65 | 622.71 |
| 23 | 98 | 142.82 | 0.00 | 0.00 | 27.740 | 3.39 | 28.01 | 4.69 | 619.80 |
| 23 | 99 | 243.52 | 0.11 | 611.1 | 14.845 | 3.77 | 15.46 | 5.99 | 618.10 |
| 23 | 100 | 244.98 | 0.00 | 0.00 | 14.740 | 3.78 | 15.01 | 6.12 | 635.18 |
| 23 | 101 | 329.73 | 0.16 | 356.6 | 10.074 | 4.18 | 10.75 | 7.25 | 627.70 |
| 23 | 102 | 316.57 | 0.37 | 158.1 | 10.634 | 4.11 | 11.56 | 6.90 | 615.37 |
| 23 | 103 | 245.27 | 0.17 | 374.4 | 14.715 | 3.78 | 15.32 | 6.01 | 630.78 |
| 23 | 104 | 254.62 | 0.06 | 1117 | 14.049 | 3.82 | 14.65 | 6.15 | 638.16 |
| 234 | MISSING TUBE(S) | | | | | | | | |

26 May 99 10:40

ALPHA/BETA - 1.02

Page #

Protocol #:23

U-233 3% 2 sigma

User : Alk

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-----|-------|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 100 | | 0 | 0.3 | 19.39 |
| Region B: | 100 - 350 | | 0 | 3.0 | 3.37 |
| Region C: | 0.0 - 2000 | | 0 | 0.1 | 28.60 |

Quench Indicator: SIS

alpha cpm U-233 1st vial bkgnd

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|------|------------|------------|--------------|---------------------|
| P# | 1 | 999.99 | 19.39 1.44 | 3.366 3.45 | 28.60 1.18 144.39 B |
| 23 | 2 | 55.30 | 0.70 176.4 | 77.014 3.13 | 77.78 3.59 613.08 |
| 23 | 3 | 53.56 | 1.58 81.24 | 79.606 3.13 | 81.80 3.53 606.82 |
| 23 | 4 | 38.45 | 2.72 56.74 | 112.239 3.09 | 115.19 3.37 604.91 |
| 23 | 5 | 37.61 | 0.53 281.9 | 114.821 3.09 | 115.72 3.40 613.08 |
| 23 | 6 | 58.82 | 0.66 183.2 | 72.187 3.14 | 73.49 3.61 616.10 |
| 23 | 7 | 56.31 | 0.94 130.6 | 75.554 3.14 | 76.78 3.59 613.13 |

5/26/99

5/28/99

Experimental Procedure for SiO₂ analysis using SpectrophotometryObjective: To determine the concentrations of SiO₂ in experimental solutions of U-sorption experiments.

Initial Conditions: Operating wavelength = 815 nm

Equipment and Supplies:

Milton Roy Spectronic 1201
1 cm cuvettes (FS)
Analytical balance (mettler PM4600)
Hot Plate and magnetic stirrer (Corning model PC220)
Orion 920A PH meter
Nessler tubes matched, 50 ml, EXAX
Glass beakers, flasks, magnetic stirrers
Polyethylene bottles
Pipetors and tips
Weighing paper/boats
deionized ultrapure water, DH₂O
HCl (lot# 956110)
NaOH (lot# 980698)
Ammonium molybdate (lot # 901533)
Oxalic acid (lot# 906135B)
1-amino-2-naphthol-4-sulfonic acid (lot# 1807KEKS)
Sodium sulfite (lot# 904672)
Sodium bisulfite (lot# 915268)
Filter paper
SiO₂ reference solution (lot# 9944097-24)

Reagents: SiO₂ reference solution diluted to 10 ppm (prepared on 3/22/99 by JP)
6M NaOH soln.(prepared on 7/24/98 by AJ)
Ammonium molybdate solution (prepared On 5/3/99 by RC)
Oxalic Acid solution (prepared On 3/22/99 by JP)
Reducing Agent (prepared on 5/4/99 by AJ)
1+1 HCl solution (prepared On 7/24/98 by AJ)

Procedure:

Heteropoly Blue method as described in TOP-014

Experimental Procedure for Al analysis using Spectrophotometry

Objective: To determine the concentrations of Al in experimental solutions of U-sorption experiments.

Initial Conditions: Operating wavelength = 535 nm

Equipment and Supplies:

Milton Roy Spectronic 1201
1 cm cuvettes (FS)
Analytical balance (mettler PM4600)
Hot Plate and magnetic stirrer (Corning model PC220)
Orion 920A PH meter
Nessler tubes matched, 50 ml, EXAX
Glass beakers, flasks, magnetic stirrers
Polyethylene bottles
Pipettors and tips
Weighing paper/boats
deionized ultrapure water, DH₂O
H₂SO₄ (lot# 913495)
Na acetate trihydrate (lot# 937077)
Acetic acid (lot # 971798)
Disodium ethylenediamine tetra acetate, EDTA (lot# 905518)
Eriochrome cyanine R (lot# 249482 588, Fluka Chemie AG)
Al reference solution (lot# 981033-18)

Reagents:

Al reference solution diluted to 5 ppm
0.01M EDTA (prepared on 7/22/98 by AJ)
0.02N H₂SO₄ (prepared on 7/22/98 by AJ)
Stock dye solution (prepared on 7/22/98 by AJ)
Working dye solution (prepared on 7/22/98 by AJ)
Buffer reagent (prepared On 7/22/98 by AJ)

Procedure:

As described in TOP-013

For Al³⁺ calibration Set wavelength = 535 nm.

calibration

Standard solutions were prepared by additions of different volumes of 5 ppm Al std. solution per 150 ml sample as follows:-

| (Al Conc.) | H ₂ O | Al. Std. | 0.02N H ₂ SO ₄ | ascorbic Acid | Buffer Reagent | Dye soln. |
|-------------|------------------|-------------|---|------------------|-------------------|--------------|
| Blank 0 PPb | 33 ml | 0 ml | 1 ml | 1 ml | 10 ml | 5 ml |
| 10 PPb | 32.9 ml | 0.1 | 1 | 1 | 10 | 5 |
| 50 PPb | 32.5 ml | 0.5 | 1 | 1 | 10 | 5 |
| 100 PPb | 32 ml | 1.0 | 1 | 1 | 10 | 5 |
| 250 PPb | 30.5 | 2.5 | 1 | 1 | 10 | 5 |

Note:- Samples

Samples were prepared by the additions of 5 ml experimental solution from desired PH as follows:

| Expt. Soln. | H ₂ O | 0.02N H ₂ SO ₄ | Ascorbic Acid | Buffer Reagent | Dye soln. |
|-------------|------------------|---|------------------|-------------------|--------------|
| 5 ml | 28 ml | 1 ml | 1 ml | 10 ml | 5 ml |

Results:-

| PH | CQ0V | CQ10V | CQ20V |
|-----|-------------|-------|-------|
| 3.5 | AJ 48.8 6.9 | 49.9 | 48.8 |
| 4.5 | 44.3 4.3 | 46.9 | 44.3 |
| 5.0 | 16.0 4.7 | 21.4 | 16.0 |
| 6.0 | 0 4.5 | 29.4 | 0.8 |
| 6.5 | 4.6 | 60.4 | 46.9 |
| 7.0 | 4.38 | 34.3 | 2.3 |
| 8.0 | 4.39 | 33.4 | 0.6 |
| 9.0 | 3.7 | 7.2 | 19.5 |

For SiO₂ Spectrophotometer wavelength set to 815 nm.

Calibration

Standard ^{AT} #4 solutions were prepared by additions of different volumes of 10 ppm SiO₂ solution per 57 ml sample as follows:-

| SiO ₂ Conc. | H ₂ O | SiO ₂ Std. | HCl | Ammon. molybdate | Oxalic Acid | Reducing Agent |
|------------------------|------------------|-----------------------|------|---------------------|----------------|-------------------|
| 0 PPb | 50 ml | 0 ml | 1 ml | 2 ml | 2 ml | 2 ml |
| 88 PPb | 49.5 | 0.5 | 1 | 2 | 2 | 2 |
| 351 PPb | 48 | 2 | 1 | 2 | 2 | 2 |
| 877 PPb | 45 | 5 | 1 | 2 | 2 | 2 |
| 1754 PPb | 40 | 10 | 1 | 2 | 2 | 2 |

*

Results:-

| PH | (QC0U) conc. | (QC10U) conc. | (QC20U) conc. |
|-----|-----------------|------------------|------------------|
| 3.5 | 45 | 1094 | 1416 |
| 4.5 | 75 | 1063 | 1333 |
| 5.0 | 358 | 942 | 892 |
| 6.0 | 508 | 1088 | 716 |
| 6.5 | 622 | 1645 | 1400 |
| 7.0 | 766 | 1362 | 962 |
| 8.0 | 1149 | 1687 | 1231 |
| 9.0 | 1516 | 1718 | 1934 |

*

Note:- Samples were prepared by the addition of 5 ml aliquots from each PH & therefore additions are as follows:-

Samples:-

| Expt. Soln | H ₂ O | HCl | Ammon. molybdate | Oxalic Acid | Reducing Agent |
|------------|------------------|------|---------------------|----------------|-------------------|
| 5 ml | 45 ml | 1 ml | 2 ml | 2 ml | 2 ml |

SiO₂ and Al solution concentrations
Measurement of solutions from desorption containers of mixed mineral (qtz/clinop) experiments

| Exp soln. | QC0U | | | QC10U | | | QC20U | | |
|-----------|---------------|-------------------------|-----------|---------------|-------------------------|-----------|---------------|-------------------------|-----------|
| | equilib pH | SiO ₂ ppm | Al ppb | equilib pH | SiO ₂ ppm | Al ppb | equilib pH | SiO ₂ ppm | Al ppb |
| 3.5 | 3.42 | 0.42 | 69 | 4.09 | 12.38 | 499 | 4.06 | 16.05 | 488 |
| 4.5 | 4.6 | 0.76 | 43 | 5.94 | 12.03 | 469 | 6.02 | 15.11 | 443 |
| 5.0 | 5.9 | 3.99 | 47 | 6.38 | 10.65 | 214 | 5.47 | 10.08 | 160 |
| 6.0 | 6.47 | 5.70 | 45 | 6.58 | 12.31 | 294 | 5.69 | 8.07 | 8 |
| 6.5 | 6.67 | 7.00 | 46 | 6.67 | 18.66 | 604 | 5.87 | 15.87 | 469 |
| 7.0 | 7.02 | 8.64 | 43.8 | 7.07 | 15.44 | 343 | 6.76 | 10.88 | 23 |
| 8.0 | 7.93 | 13.01 | 43.9 | 7.94 | 19.14 | 334 | 7.84 | 13.94 | 6 |
| 9.0 | 8.94 | 17.19 | 37 | 8.96 | 19.49 | 72 | 8.98 | 21.96 | 195 |

6/22/99 AT

6/23/99 Objective:- BET surface area analysis on Y-Alumina using COULTER SA 3100.Equipment & Supplies:-

COULTER SA 3100

Y-Alumina (lot # 39812)
C16F19

Sample holders (tube assembly)

liquid nitrogen

He & N₂ gas tanks

Mettler AE240 weighing balance

Weighing boats

Procedure:- Followed as per COULTER SA 3100 manual

- ① wt. of tube assembly #5 = 33.3692 g
 wt. of Y-alumina added = 0.4130 g
 wt. of tube assembly + outgassed sample = 33.7551 g
 wt. of outgassed sample = 33.7551
 - 33.3692
 0.3859 g

$$\text{BET SA} = \boxed{93.062} \text{ m}^2/\text{g}$$

- ② wt. of tube assembly #7 = 33.6226 g
 0.6788
 - 0.1732
 wt. of sample added = $\boxed{0.5056}$ g

wt. of tube assembly + outgassed sample = 34.0935 g

$$\begin{array}{r} \text{wt. of outgassed sample} = 34.0935 \\ - 33.6226 \\ \hline 0.4709 \text{ g} \end{array}$$

$$\text{BET SA} = \boxed{92.560} \text{ m}^2/\text{g}$$

- ③ wt. of tube assembly #1 = 33.6262 g
 wt. of Y-Al₂O₃ added = 0.3064 g
 wt. of tube assembly + outgassed sample = 33.9120 g
 wt. of outgassed sample = 33.9120
 - 33.6262
 0.2858 g

$$\text{BET SA} = 91.407 \text{ m}^2/\text{g}$$

- ④ Preparation of 0.1M NaCl stock solution for Tc-99 spike. NaCl (lot # 914193)

5.9 g NaCl diluted to 1L with DI H₂O.

2 batches were prepared, total of 2L of 0.1M NaCl.

- ⑤ Calculations for dilution of Tc-99 spike.
 Volume = 5 ml.
 Activity = 10 μCi . Specific activity = 17.08 mCi/g
 Conc. = 2 $\mu\text{Ci}/\text{gml}$

Total

$$\begin{aligned} \text{Activity} &= 10 \mu\text{Ci} \\ &= 10 \mu\text{Ci} \times 2.220 \times 10^6 \frac{\text{dpm}}{\mu\text{Ci}} \\ &= 2.220 \times 10^7 \text{ dpm} \end{aligned}$$

a) Dilution to 2L

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$$\text{Activity} = \frac{2.220 \times 10^7 \text{ dpm}}{2 \text{ L}}$$

$$= \frac{2.220 \times 10^7 \text{ dpm}}{2000 \text{ ml}}$$

$$= 11,100 \text{ dpm/ml}$$

The sample spike was diluted to 2L
using 0.1 M NaCl.

6/24/99
AJ

| Sample Name | wt. of vial | wt. Of vial + sample | sample wt. |
|-------------|-------------|----------------------|------------|
| Tc-1 | 7.2862 | | ~ 0.5000 |
| Tc-2 | 7.2829 | | ~ 0.5000 |
| Tc-3 | 7.2404 | 7.3400 | 0.0996 |
| Tc-4 | 7.3122 | 7.5124 | 0.2002 |
| Tc-5 | 7.2734 | 7.5737 | 0.3003 |
| Tc-6 | 7.3186 | 7.7194 | 0.4008 |
| Tc-7 | 7.268 | 7.7687 | 0.5007 |
| Tc-8 | 7.3149 | 7.9148 | 0.5999 |
| Tc-9 | 7.2322 | 7.9339 | 0.7017 |
| Tc-10 | 7.2501 | 8.0511 | 0.8010 |
| Tc-11 | 7.3007 | 8.2036 | 0.9029 |
| Tc-12 | 7.2654 | 8.2661 | 1.0007 |
| | | | 0.0000 |
| Tc-1P AB | 3.5106 | 3.7105 | 0.1999 |
| Tc-2P AB | 3.5223 | 3.9233 | 0.4010 |
| Tc-3P AB | 3.4698 | 4.1718 | 0.7020 |
| Tc-4P AB | 3.538 | 4.5387 | 1.0007 |

6/25

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| | | | |
|---------|--------|--------|--------|
| Tc-1P G | 3.4791 | 3.6789 | 0.1998 |
| Tc-2P G | 3.4971 | 3.8979 | 0.4008 |
| Tc-3P G | 3.4926 | 4.1944 | 0.7018 |
| Tc-4P G | 3.5395 | 4.5403 | 1.0008 |
| Tc-4 G | 7.2542 | 7.4536 | 0.1994 |
| Tc-6 G | 7.2821 | 7.6834 | 0.4013 |
| Tc-9 G | 7.2336 | 7.9348 | 0.7012 |
| Tc-12 G | 7.3103 | 8.3105 | 1.0002 |

24 Jun 99 15:40

ALPHA/BETA - 1.02

Page

Protocol #:43

TC-99 CPM

User : Ji

Time: 10.00

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: None

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|------|
| Region A: | 0.0 - 300 | | 0 | 0.0 | 0.00 |
| Region B: | 2.0 - 300 | | 0 | 0.0 | 0.00 |
| Region C: | 0.0 - 2000 | | 0 | 0.0 | 0.00 |

Quench Indicator: SIS

Beta cpm TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|----|-------|---------|-------|---------|-------|---------|-------|--------|-------|
| 1 | 10.00 | 1047.50 | 1.95 | 1039.80 | 1.96 | 1051.90 | 1.95 | 210.58 | Tc-3 |
| 2 | 10.00 | 2067.00 | 1.39 | 2047.00 | 1.40 | 2072.30 | 1.39 | 198.72 | |
| 3 | 10.00 | 3081.00 | 1.14 | 3052.00 | 1.14 | 3086.40 | 1.14 | 187.98 | |
| 4 | 10.00 | 4096.50 | 0.99 | 4054.80 | 0.99 | 4101.40 | 0.99 | 181.48 | |
| 5 | 10.00 | 5097.70 | 0.89 | 5049.40 | 0.89 | 5103.40 | 0.89 | 177.42 | |
| 6 | 10.00 | 6098.70 | 0.81 | 6036.40 | 0.81 | 6105.40 | 0.81 | 173.59 | |
| 7 | 10.00 | 7166.30 | 0.75 | 7090.10 | 0.75 | 7172.50 | 0.75 | 171.34 | |
| 8 | 10.00 | 8162.40 | 0.70 | 8073.70 | 0.70 | 8169.00 | 0.70 | 167.81 | |
| 9 | 10.00 | 9171.20 | 0.66 | 9070.70 | 0.66 | 9178.10 | 0.66 | 164.28 | |
| 10 | 10.00 | 10167.2 | 0.63 | 10053.0 | 0.63 | 10173.9 | 0.63 | 161.12 | Tc-12 |

24 Jun 99 12:46 ALPHA/BETA - 1.02
Protocol #:12 C-14 CPM

TC-1

Time: 4.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

| | LL | UL | LCR | 25% | BKG |
|-----------|-------|-----|-----|-----|------|
| Region A: | 0.0 - | 156 | 0 | 0.0 | 0.00 |
| Region B: | 4.0 - | 156 | 0 | 0.0 | 0.00 |
| Region C: | 0.0 - | 350 | 0 | 0.0 | 0.00 |

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA | CPMB | CPMC | SIS FLAG | tSIE |
|----|------|---------|---------|---------|----------|--------|
| 1 | 4.00 | 3894.25 | 3836.50 | 5143.50 | 122.58 | 486.78 |

24 Jun 99 13:06 ALPHA/BETA - 1.02
Protocol #:14 C-14 DPM TC-1 & TC-2

Time: 5.00
Data Mode: DPM Nuclide: 14C
Background Subtract: None

| | LL | UL | LCR | 25% | BKG |
|-----------|-------|-----|-----|-----|------|
| Region A: | 0.0 - | 156 | 0 | 0.5 | 0.00 |
| Region B: | 4.0 - | 156 | 0 | 0.0 | 0.00 |
| Region C: | 0.0 - | 0.0 | 0 | 0.0 | 0.00 |

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count

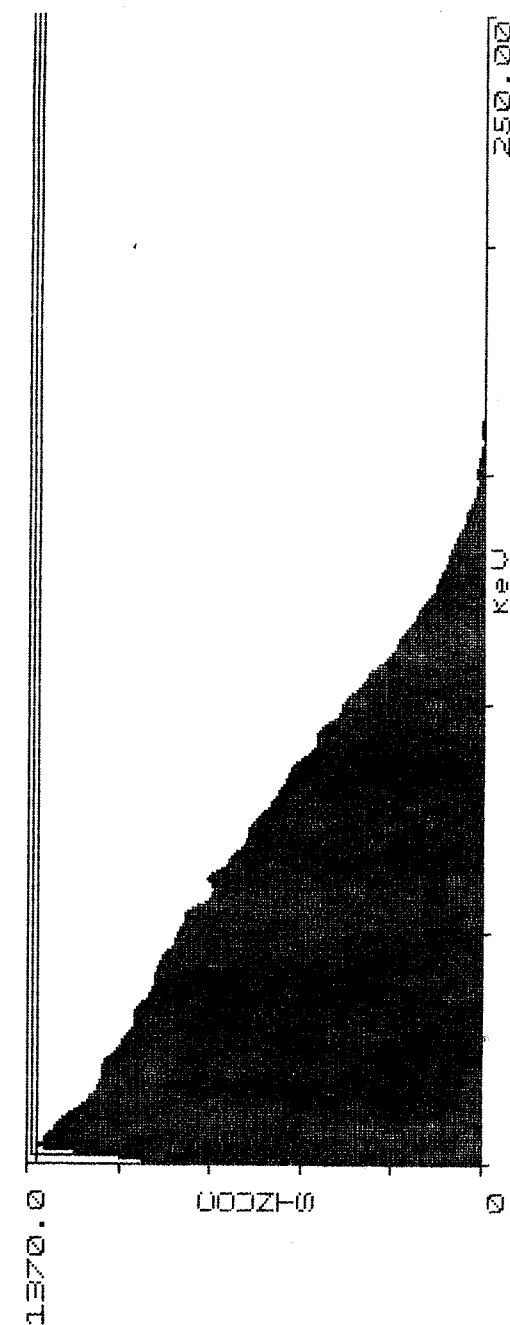
| S# | TIME | CPMA | DPM1 | SIS | tSIE | FLAG |
|----|------|---------|---------|--------|--------|---------|
| 1 | 5.00 | 3911.20 | 4118.41 | 122.92 | 486.79 | 0.00012 |
| 2 | 5.00 | 3897.60 | 4104.13 | 123.51 | 486.48 | 0.00012 |

PH of starting spike #43 = 5.4

25 Jun 99 12:25 ALPHA/BETA - 1.02 Page #3
Protocol #:43 TC-99 CPM User: Jim Prikrlyl

Sample #: 36
Count Time: 10.00
AEC Off

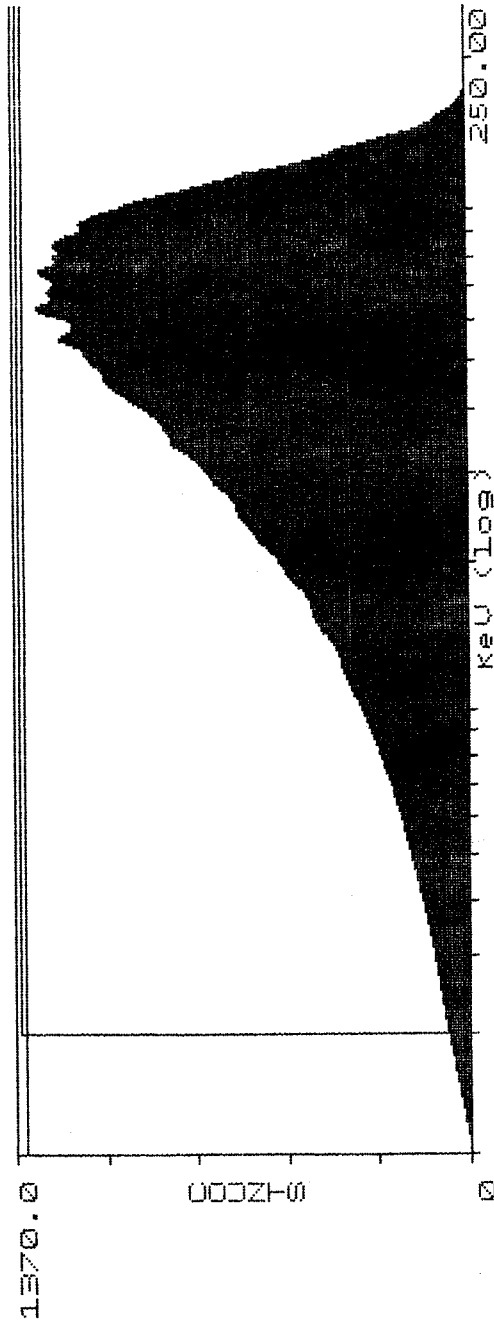
| | LL | UL | CPM | 25% |
|-----------|-----|--------|----------|-----|
| Region A: | 0.0 | 300.0 | 10167.20 | 0.6 |
| Region B: | 2.0 | 300.0 | 10053.00 | 0.6 |
| Region C: | 0.0 | 2000.0 | 10173.90 | 0.6 |



25 Jun 99 12:24 ALPHA/BETA - 1.02 Page #2
 Protocol #:43 TC-99 CPM User : Jim Prikrly

Sample #: 36
 Count Time: 10.00
 AEC Off

| | LL | UL | CPM | 25% |
|-----------|-----|--------|----------|-----|
| Region A: | 0.0 | 300.0 | 10167.20 | 0.6 |
| Region B: | 2.0 | 300.0 | 10053.00 | 0.6 |
| Region C: | 0.0 | 2000.0 | 10173.90 | 0.6 |



25 Jun 99 15:07
 Protocol #:43

ALPHA/BETA - 1.02
 TC-99 CPM

Page #
 User : Jim P

Time: 10.00
 Data Mode: CPM
 Background Subtract: None

Nuclide: MANUAL

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|------|
| Region A: | 0.0 - 300 | | 0 | 0.0 | 0.00 |
| Region B: | 2.0 - 300 | | 0 | 0.0 | 0.00 |
| Region C: | 0.0 - 2000 | | 0 | 0.0 | 0.00 |

Quench Indicator: SIS
 Beta cpm TC-99
 Coincidence Time(ns): 18
 Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|-------------------|-------|---------|-------|---------|-------|---------|-------|--------|------|
| 1 | 10.00 | 1028.60 | 1.97 | 1019.60 | 1.98 | 1034.70 | 1.97 | 209.63 | |
| 2 | 10.00 | 2057.30 | 1.39 | 2036.20 | 1.40 | 2062.80 | 1.39 | 195.03 | |
| 3 | 10.00 | 3056.10 | 1.14 | 3027.40 | 1.15 | 3063.00 | 1.14 | 186.76 | |
| 4 | 10.00 | 4091.80 | 0.99 | 4046.60 | 0.99 | 4098.90 | 0.99 | 181.01 | |
| 5 | 10.00 | 5088.00 | 0.89 | 5036.30 | 0.89 | 5093.90 | 0.89 | 176.41 | |
| 6 | 10.00 | 6100.50 | 0.81 | 6032.80 | 0.81 | 6106.70 | 0.81 | 172.58 | |
| 7 | 10.00 | 7175.00 | 0.75 | 7099.20 | 0.75 | 7180.90 | 0.75 | 169.69 | |
| 8 | 10.00 | 8117.50 | 0.70 | 8031.20 | 0.71 | 8122.80 | 0.70 | 165.82 | |
| 9 | 10.00 | 9189.70 | 0.66 | 9083.10 | 0.66 | 9196.10 | 0.66 | 163.58 | |
| 10 | 10.00 | 10174.5 | 0.63 | 10066.3 | 0.63 | 10180.0 | 0.63 | 160.92 | |
| 8 MISSING TUBE(S) | | | | | | | | | |
| 19 | 10.00 | 2038.60 | 1.40 | 2018.30 | 1.41 | 2043.90 | 1.40 | 183.59 | |
| 20 | 10.00 | 4090.10 | 0.99 | 4044.50 | 0.99 | 4096.90 | 0.99 | 172.54 | |
| 21 | 10.00 | 7141.70 | 0.75 | 7058.60 | 0.75 | 7148.10 | 0.75 | 166.97 | |
| 22 | 10.00 | 10190.3 | 0.63 | 10077.5 | 0.63 | 10197.8 | 0.63 | 165.41 | |
| 1 MISSING TUBE(S) | | | | | | | | | |
| 24 | 10.00 | 2040.00 | 1.40 | 2022.10 | 1.41 | 2043.80 | 1.40 | 215.83 | |
| 25 | 10.00 | 4108.30 | 0.99 | 4072.40 | 0.99 | 4114.00 | 0.99 | 199.93 | |

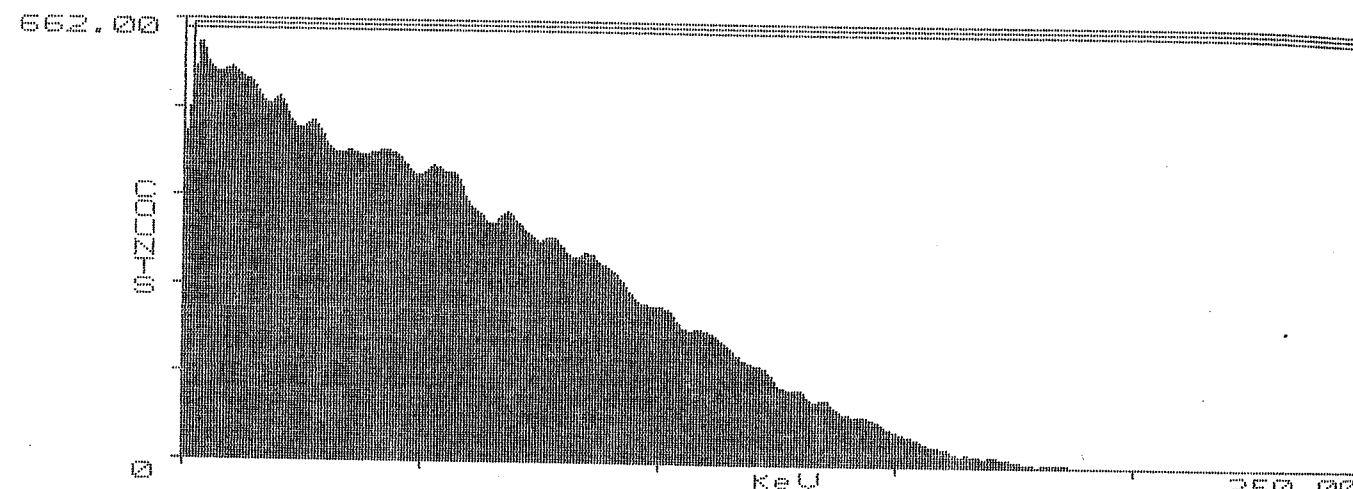
TC-3
 0.1000 ml
 0.2000 ml
 glass
 AB
 cocktail
 TC-12
 TC-49
 TC-69
 TC-99
 TC-129
 TC-1P AB cocktail
 TC-27AB Vials
 AB cocktail

5 Jun 99 15:24 ALPHA/BETA - 1.02
Protocol #:43 TC-99 CPM

Page #2
User : Jim Priky

Sample #: 26
Count Time: 7.30
AEC Off

| | LL | UL | CPM | 25% |
|-----------|-----|--------|---------|-----|
| Region A: | 0.0 | 300.0 | 7161.37 | 0.9 |
| Region B: | 2.0 | 300.0 | 7089.04 | 0.9 |
| Region C: | 0.0 | 2000.0 | 7166.99 | 0.9 |



| | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|-----------------|-------|---------|-------|---------|-------|---------|-------|--------|----------------------|
| 26 | 10.00 | 7153.10 | 0.75 | 7081.00 | 0.75 | 7159.20 | 0.75 | 182.37 | Tc-3PAB plastic vial |
| 27 | 10.00 | 10195.2 | 0.63 | 10091.5 | 0.63 | 10200.7 | 0.63 | 171.13 | Tc-4PAB cocktail |
| MISSING TUBE(S) | | | | | | | | | |
| 29 | 10.00 | 2050.50 | 1.40 | 2029.10 | 1.40 | 2057.50 | 1.39 | 199.29 | Tc-1P9 Gold cocktail |
| 30 | 10.00 | 4110.20 | 0.99 | 4069.90 | 0.99 | 4115.90 | 0.99 | 190.70 | Tc-2P9 Elastic vial |
| 31 | 10.00 | 7191.70 | 0.75 | 7121.10 | 0.75 | 7198.10 | 0.75 | 188.76 | Tc-3P9 |
| MISSING TUBE(S) | | | | | | | | | |
| 32 | 10.00 | 10189.2 | 0.63 | 10085.0 | 0.63 | 10196.2 | 0.63 | 177.05 | Tc-4P9 |

25 Jun 99 17:04 ALPHA/BETA - 1.02
Protocol #:43 TC-99 CPM

Page #
User : Jim F

Time: 100.00
Data Mode: CPM
Sigma Coincidence On
Background Subtract: None
Nuclide: MANUAL

| | LL | UL | LCR | 25% | BKG |
|-----------|-----|------|-----|-----|------|
| Region A: | 0.0 | 300 | 0 | 0.0 | 0.00 |
| Region B: | 2.0 | 300 | 0 | 1.0 | 0.00 |
| Region C: | 0.0 | 2000 | 0 | 0.0 | 0.00 |

Quench Indicator: SIS
Beta cpm TC-99
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|-----------------|-------|---------|-------|---------|-------|---------|-------|--------|---------|
| 1 | 38.90 | 1038.05 | 1.00 | 1028.46 | 1.00 | 1043.60 | 0.99 | 209.10 | Tc-3 |
| 2 | 19.66 | 2053.36 | 1.00 | 2034.74 | 1.00 | 2059.26 | 0.99 | 195.74 | |
| 3 | 13.04 | 3096.40 | 1.00 | 3068.64 | 1.00 | 3102.53 | 0.99 | 187.10 | |
| 4 | 9.97 | 4057.47 | 0.99 | 4013.54 | 1.00 | 4064.69 | 0.99 | 182.18 | |
| 5 | 7.98 | 5068.42 | 0.99 | 5013.53 | 1.00 | 5075.44 | 0.99 | 176.48 | |
| 6 | 6.60 | 6130.30 | 0.99 | 6066.97 | 1.00 | 6137.12 | 0.99 | 172.32 | |
| 7 | 5.59 | 7232.92 | 0.99 | 7155.99 | 1.00 | 7239.71 | 0.99 | 168.74 | |
| 8 | 5.00 | 8094.20 | 0.99 | 8011.00 | 1.00 | 8103.00 | 0.99 | 165.58 | |
| 9 | 4.38 | 9235.39 | 0.99 | 9134.70 | 1.00 | 9241.78 | 0.99 | 163.11 | |
| 10 | 3.97 | 10192.7 | 0.99 | 10085.1 | 1.00 | 10196.0 | 0.99 | 160.38 | Tc-12 |
| MISSING TUBE(S) | | | | | | | | | |
| 19 | 19.73 | 2050.63 | 0.99 | 2027.57 | 1.00 | 2057.12 | 0.99 | 181.53 | Tc-49 |
| 20 | 9.82 | 4120.67 | 0.99 | 4073.93 | 1.00 | 4127.50 | 0.99 | 173.31 | Tc-69 |
| 21 | 5.66 | 7151.94 | 0.99 | 7070.50 | 1.00 | 7158.83 | 0.99 | 166.00 | Tc-99 |
| 22 | 3.95 | 10247.6 | 0.99 | 10139.2 | 1.00 | 10254.7 | 0.99 | 166.17 | Tc-129 |
| MISSING TUBE(S) | | | | | | | | | |
| 24 | 19.67 | 2051.35 | 1.00 | 2034.62 | 1.00 | 2056.43 | 0.99 | 214.85 | Tc-1PAB |
| 25 | 9.95 | 4060.90 | 0.99 | 4023.52 | 1.00 | 4067.54 | 0.99 | 199.04 | |
| 26 | 5.64 | 7168.97 | 0.99 | 7104.26 | 1.00 | 7175.18 | 0.99 | 183.12 | |
| 27 | 3.98 | 10165.1 | 0.99 | 10057.8 | 1.00 | 10172.6 | 0.99 | 172.04 | Tc-4PAB |
| MISSING TUBE(S) | | | | | | | | | |
| 29 | 19.55 | 2066.55 | 1.00 | 2046.45 | 1.00 | 2072.79 | 0.99 | 201.35 | Tc-1P9 |
| 30 | 9.88 | 4094.33 | 0.99 | 4052.33 | 1.00 | 4100.71 | 0.99 | 191.05 | |
| 31 | 5.62 | 7194.48 | 0.99 | 7130.78 | 1.00 | 7200.18 | 0.99 | 188.81 | |
| 32 | 3.97 | 10198.0 | 0.99 | 10093.7 | 1.00 | 10204.0 | 0.99 | 177.49 | Tc-4P9 |

6/28/99 AJ

① (sample) (wt. of vial) (wt. of vial + 0.1M NaCl + sample) (wt. of vial + 0.1M NaCl + sample AJ)

| | | | |
|-------|--------|--------|--------|
| Tc-13 | 7.2946 | 7.7944 | 7.6944 |
| Tc-14 | 7.2147 | 7.7143 | 7.5145 |
| Tc-15 | 7.2862 | 7.7865 | 7.4857 |
| Tc-16 | 7.3508 | 7.8520 | 7.4505 |
| Tc-17 | 7.3169 | 7.8193 | 7.3169 |

| | wt. of 0.1M NaCl | wt. of sample added |
|-------|------------------|---------------------|
| Tc-13 | 0.3998 ml | 0.1000 ml |
| Tc-14 | 0.2998 | 0.1998 |
| Tc-15 | 0.1995 | 0.3008 |
| Tc-16 | 0.0997 | 0.4015 |
| Tc-17 | 0.0000 | 0.5024 |

Note: 0.1M NaCl was added to have same amounts of Na in all samples for counting.
d/p cocktail was used.

Result: consistent values of SIS obtained.

② PH of spike # 43 adjusted to 7.0 using 1.4 ml of 0.1M NaOH.

③ NaCl solution prepared of 0.1M. F.W. NaCl = 59 g
2.95 g of NaCl (lot # 914193) was dissolved in DI water and more H₂O was added to mark 500 ml.

28 Jun 99 12:58

ALPHA/BETA - 1.02

Page

Protocol #: 43

TC-99 CPM

User: Jim

Time: 100.00

Data Mode: CPM

Nuclide: MANUAL

Sigma Coincidence On

Background Subtract: None

| | LL | UL | LCR | 2S% | BKG |
|-----------|------------|----|-----|-----|------|
| Region A: | 0.0 - 300 | | 0 | 0.0 | 0.00 |
| Region B: | 2.0 - 300 | | 0 | 1.0 | 0.00 |
| Region C: | 0.0 - 2000 | | 0 | 0.0 | 0.00 |

Quench Indicator: SIS

Beta cpm TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|-------|------------|--------------|--------------|-------------|
| 1 | 38.72 | 1044.06 | 0.99 1033.16 | 1.00 1050.54 | 0.99 180.68 |
| 2 | 19.67 | 2054.75 | 0.99 2035.03 | 1.00 2061.01 | 0.99 178.57 |
| 3 | 13.09 | 3088.39 | 0.99 3057.37 | 1.00 3094.42 | 0.99 179.25 |
| 4 | 9.80 | 4123.37 | 0.99 4083.57 | 1.00 4130.71 | 0.99 178.72 |
| 5 | 7.93 | 5101.14 | 0.99 5046.66 | 1.00 5105.93 | 0.99 179.44 |

↑ Tc-13
↓ Tc-17

6/29/99 AJ

PH checked on Spike # 43 (1:30 PM)
PH = 9.22

6/30/99 AJ

PH checked on Spike # 43
PH = 7.09
Spike is being stirred constantly for a few hours.

| Sample | wt. of vial | wt. of vial + 0.1M NaCl | wt. of vial + sample + NaCl |
|--------|-------------|-------------------------|-----------------------------|
|--------|-------------|-------------------------|-----------------------------|

| | AJ | | |
|-------|-------------|-------------|--------|
| Tc-18 | 7.2946 2658 | 7.6889 | 7.7691 |
| Tc-19 | 7.3071 | 7.5145 6086 | 7.8097 |
| Tc-20 | 7.3467 | 7.4857 5480 | 7.8497 |
| Tc-21 | 7.2510 | 7.3506 | 7.7529 |
| Tc-22 | 7.2956 | 7.2956 | 7.7863 |

01 Jul 1999 08:20 ALPHA/BETA - 1.09
Protocol #143 TC-99 CPM

User: JI

Time: 100.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-----|------|
| Region A: | 0.0 - 300 | | 0 | 0.0 | 0.00 |
| Region B: | 2.0 - 300 | | 0 | 1.0 | 0.00 |
| Region C: | 0.0 - 2000 | | 0 | 0.0 | 0.00 |

Quench Indicator: SIS
BETA CPM TC-99

AB cocktail, glass vials, PH = 7.09

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG |
|----|-------|------------|--------------|--------------|-------------|
| 1 | 38.85 | 1041.85 | 0.99 1029.76 | 1.00 1046.80 | 0.99 174.56 |
| 2 | 19.58 | 2066.45 | 0.99 2043.87 | 1.00 2071.96 | 0.99 174.32 |
| 3 | 13.16 | 3076.52 | 0.99 3042.10 | 1.00 3081.76 | 0.99 174.66 |
| 4 | 9.81 | 4123.14 | 0.99 4079.31 | 1.00 4129.66 | 0.99 173.36 |
| 5 | 7.91 | 5112.90 | 0.99 5058.41 | 1.00 5117.70 | 0.99 174.49 |

The counts are same as for PH = 5.4 (for Spike # 43).

7/1/99

AJ

Tc-99 KINETICS EXPERIMENT

OBJECTIVE: to determine the time it takes to reach sorption equilibrium between Tc-99 solutions and gamma-alumina powder.

- Initial Σ Tc = 29.4 ppb
- Initial solution mass = 35 g
- 0.105 g of gamma alumina
- Equilibrium with atmospheric $\text{CO}_2(\text{g})$ ($p\text{CO}_2 = 10^{-3.5}$ bar)
- M/V = 30

EQUIPMENT: Liquid Scintillation Analyzer (Packard 2500TR/AB)

ORION pH/mV/ISE/ C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balances (Mettler 4600 and 240AE)
Gyratory shaker

SUPPLIES: pH buffers (pH= 2,4,7,10)
50 ml polycarbonate centrifuge tubes (43 ml capacity)
LSA vials 7ml
Repipettor for transfer of scintillation cocktail
Packard A/B cocktail
Eppendorf pipet and tips (various volumes)
gamma-alumina (lot C16F19)
1 L FEP teflon bottle
Tc-99 stock solution (**spike #43A**)
HCl (lot 956110) various concentrations; e.g., 1.0 M, 0.1 M, etc...
NaOH (lot 976631) various concentrations; e.g., 1.0 M, 0.1 M, etc...

PROCEDURE:

1. In a precleaned 1L FEP bottle, prepare 280 g of 2.95×10^{-7} m (29.4 ppm) Tc-99 solution by diluting 28 g of a 2.95×10^{-6} m (294 ppm) ^{99}Tc stock solution (**spike #43A**; in 0.1 m NaCl matrix; prepared from a commercial ^{99}Tc spike) to a total of 280 g by taring 0.1 m NaCl solution into the FEP bottle on a Mettler 4600 balance.

2.

Label 3 50 ml polycarbonate centrifuge tubes Tc-K*pHi (where i is the approximate pH of each solution, see Table) and pre-weigh. Record weight.
3.

Add 35 g of the 29.4 ppb Tc solution to each tube and record weight.
4.

Label 3 50 ml polycarbonate centrifuge tubes Tc-KC*pHi (where i is the approximate pH of each solution, see Table) and pre-weigh. Record weight.
5.

Add 40 g of the 29.4 ppb Tc solution to each tube and record weight.
6.

Transfer the remaining 29.4 ppb Tc solution (about 35 g) into a pre-weighed 50 ml centrifuge tube labeled Tc-K*ITc. Measure the pH of the solution and record weight.
7.

Sample Tc-K*ITc to determine initial Tc concentration. Label (e.g. Tc-K*ITca and Tc-K*ITcb) and preweigh two LSA vials. Withdraw two 0.5 ml samples from Tc-K*IU and transfer to the LSA vials. Reweigh vials and record weight. Add 5 ml of scintillation cocktail to each vial. Homogenize sample and set aside for LSA.
8.

For the Tc-K*pHi and Tc-KC*pHi solutions adjust pH by adding HCl or NaOH using an Eppendorf pipetter. The amount to be added to each solution is given in the Table. After pH adjustment record weight of containers. Cap loosely and place on gyratory shaker. Wait about 10 days for solutions to reach pH equilibrium.
9.

Add 0.105 g of gamma-alumina to Tc-K*pHi solutions. Tare aliquots of gamma-alumina weighing 0.105 ± 0.001 g onto weighing paper. Transfer to each Tc-K*pHi solution (not Tc-KC*pHi solutions). Reweigh and record. Replace cap loosely. Place samples on gyratory shaker.
10.

Sample kinetic and kinetic control solutions for Tc concentration and pH over various time intervals. At intervals of 2 hours, 4 hours, 1 day, 2 days, 4 days, 8 days, 12 days, 16 days, and 21 days, sample the solutions (Tc-K*pHi and Tc-KC*pHi) for pH and Tc concentration by:

- weighing and recording weight of tube

- withdrawing two 0.5 ml aliquots and transferring into preweighed and labeled LSA vials and setting aside for LSA.

- measuring pH of each solution, reweighing tubes, and replacing on gyratory shaker.
11.

Determine Tc concentration of solutions by analyzing using LSA.

| Sample label | Estimated solution pH | Volume of HCl needed, ml | Molarity of HCl to use |
|--------------|-----------------------|---------------------------|-------------------------|
| Tc-K*pH4 | 4 | 0.075 | 0.1 |
| | | Volume of NaOH needed, ml | Molarity of NaOH to use |
| Tc-K*pH7 | 7 | --- | --- |
| Tc-K*pH9.5 | 9.5 | 1.5 | 1.0 |
| | | | |
| | | Volume of HCl needed, ml | Molarity of HCl to use |
| Tc-KC*pH4 | 4 | 0.075 | 0.1 |
| | | Volume of NaOH needed, ml | Molarity of NaOH to use |
| Tc-KC*pH7 | 7 | --- | --- |
| Tc-KC*pH9.5 | 9.5 | 1.5 | 1.0 |

7/2/99

AJ

① PH of spike # 43A = 6.75

② Prepare 0.1M NaCl to dilute the spike. following the procedure^{step} # 1 on page 283, spike 43A was diluted to a conc. of $2.95 \times 10^{-7} \text{ M}$ (29.5 ppb).

| Sample Name | Original Container wt., g | Container wt. after Tc soln. Add | Container wt. after pH adjust | Container wt. after sampling | Container wt. after alumina add |
|--------------------------|---------------------------|----------------------------------|-------------------------------|------------------------------|---------------------------------|
| Tc-K* ^{pH} 4 | 22.4493 | 57.4518 | 57.5270 | | |
| Tc-K* ^{pH} 7 | 22.4422 | 57.4436 | | | |
| Tc-K* ^{pH} 9.5 | 22.4107 | 57.4157 | 59.0008 | | |
| Tc-K*ITc | 22.4349 | 57.4364 | | 56.4305 | |
| Tc-KC* ^{pH} 4 | 22.593 | 57.5932 | 57.6690 | | |
| Tc-KC* ^{pH} 7 | 22.3248 | 57.3239 | | | |
| Tc-KC* ^{pH} 9.5 | 22.2925 | 57.2995 | 57.8913 | | |
| | | | | | |
| | wt. Of vial | wt. Of vial + sample | | | |
| Tc-K*ITca | 7.2913 | 7.7947 | | | |
| Tc-K*ITcb | 7.3336 | 7.8361 | | | |

07 Jul 1999 03:58

Protocol #:43

ALPHA/BETA - 1.09

TC-99 CPM

User: J

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

Tc-K*ITc

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 300 | 0 | 0.0 | 18.89 | |
| Region B: | 2.0 - 300 | 0 | 1.0 | 17.74 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.80 | |

Quench Indicator: SIS

BETA CPM TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG | | | | |
|----|--------|------------|------------|------------|----------|--------|------|--------|---|
| 1 | 999.99 | 18.89 | 1.46 | 17.74 | 1.50 | 24.80 | 1.27 | 126.12 | B |
| 2 | 75.98 | 514.24 | 1.03 | 508.72 | 1.04 | 514.82 | 1.04 | 177.62 | |
| 3 | 76.09 | 513.48 | 1.03 | 507.98 | 1.04 | 513.97 | 1.04 | 178.20 | |

7/9/99
AJ

PH measurements on Tc-K* & Tc-KC* experiments

| | wt. before PH | measured PH | wt. after PH |
|-----------|---------------|-------------|--------------|
| Tc-K* 4.0 | 57.4020 | 3.79 | 57.3929 |

| | | | |
|-----------|---------|------|---------|
| Tc-K* 7.0 | 57.3776 | 5.73 | 57.3666 |
|-----------|---------|------|---------|

| | | | |
|-----------|---------|-------|---------|
| Tc-K* 9.5 | 58.8862 | 12.37 | 58.8776 |
|-----------|---------|-------|---------|

| | | | |
|----------|---------|------|---------|
| Tc-K*ITc | 56.3510 | 5.73 | 56.3382 |
|----------|---------|------|---------|

| | | | |
|------------|---------|------|---------|
| Tc-KC* 4.0 | 57.5734 | 3.76 | 57.5573 |
|------------|---------|------|---------|

| | | | |
|------------|---------|------|---------|
| Tc-KC* 7.0 | 57.2240 | 5.76 | 57.2086 |
|------------|---------|------|---------|

| | | | |
|------------|---------|-------|---------|
| Tc-KC* 9.5 | 58.7818 | 12.43 | 58.7757 |
|------------|---------|-------|---------|

PH of spike #43A (29.4PPb) = 6.80

The PH of Tc-K* & Tc-KC* samples (at desired PH of 9.5) was measured ~12.4 which is very high. So, these samples were kept on shaker with caps removed and covered with kimwipe to be equilibrated with atmospheric CO₂.

| | wt. without cap |
|-------------|-----------------|
| Tc-K* 9.5a | 56.1516 |
| Tc-KC* 9.5b | 56.1170 |

7/12/99

AJ

PH measurements on Tc-K* 9.5 = 11.67

Still high & again kept on shaker to equilibrate with atmospheric CO₂.

7/19/99

AJ

PH measurements on Tc-K* 9.5 samples.

| | | wt. of container | pH measured | after pH wt. of container |
|--|----------------|------------------------|--|------------------------------|
| | Tc-K* 9.5a | 54.1611 | 9.91 | 54.1459 |
| | Tc-K* 9.5b | 53.8785 | 9.88 | 53.8611 |
| | Tc-K* 9.5a | 56.8527 |] wt. of containers with lid after pH measurements. | |
| | Tc-K* 9.5b | 56.5041 | | |
| Following step # 9 from Tc-kinetics experiment procedure on page 284, γ -Al ₂ O ₃ was added to Tc-K*(pH) samples. (0.105 g) | | | | |
| | Sample name | wt. of container g. | wt. of container + γ -Al ₂ O ₃ , g | |
| | Tc-K* 4.0 | 57.1702 | 57.2767 | |
| | Tc-K* 7.0 | 57.2345 | 57.3414 | |
| | Tc-K* 9.5 | 56.8527 | 56.9590 | |

| | 3 hours | | | | | |
|----------------|---------|--------|--------|---------|------|---------|
| Tc-K* pH 4a | 7.2590 | 7.7578 | 0.4988 | 56.2516 | 4.34 | 55.2304 |
| Tc-K* pH 4b | 7.2328 | 7.7325 | 0.4997 | | | |
| Tc-K* pH 7a | 7.2206 | 7.7178 | 0.4972 | 56.3190 | 6.57 | 55.3115 |
| Tc-K* pH 7b | 7.2799 | 7.78 | 0.5001 | | | |
| Tc-K* pH 9.5a | 7.3778 | 7.8782 | 0.5004 | 55.9325 | 9.91 | 54.9176 |
| Tc-K* pH 9.5b | 7.2648 | 7.7674 | 0.5026 | | | |
| | | | | | | |
| Tc-KC* pH 4a | 7.2979 | 7.7974 | 0.4995 | 56.3708 | 3.77 | 55.3535 |
| Tc-KC* pH 4b | 7.2289 | 7.7322 | 0.5033 | | | |
| Tc-KC* pH 7a | 7.2794 | 7.7788 | 0.4994 | 55.9986 | 5.25 | 54.9835 |
| Tc-KC* pH 7b | 7.2757 | 7.7768 | 0.5011 | | | |
| Tc-KC* pH 9.5a | 7.2380 | 7.7391 | 0.5011 | 55.4811 | 9.91 | 54.4643 |
| Tc-KC* pH 9.5b | 7.2543 | 7.7576 | 0.5033 | | | |

7/19/99

| 7/19/99 | | | | | | |
|----------------|-------------|-------------------------|------------|--|----------------|---|
| Sample Name | wt. of vial | wt. of vial + sample | sample wt. | Container wt. before sampling & pH measuremen ts, g | Measured pH | Container wt. after sampling & pH measuremen ts, g |
| | 1 hour | | | | | |
| Tc-K* pH 4a | 7.2184 | 7.7190 | 0.5006 | 57.2744 | 4.14 | 56.2539 |
| Tc-K* pH 4b | 7.2784 | 7.7808 | 0.5024 | | | |
| Tc-K* pH 7a | 7.2625 | 7.7644 | 0.5019 | 57.3392 | 6.5 | 56.3217 |
| Tc-K* pH 7b | 7.2987 | 7.8011 | 0.5024 | | | |
| Tc-K* pH 9.5a | 7.2496 | 7.752 | 0.5024 | 56.9573 | 9.89 | 55.9351 |
| Tc-K* pH 9.5b | 7.2870 | 7.7908 | 0.5038 | | | |
| | | | | | | |
| Tc-KC* pH 4a | 7.3096 | 7.8078 | 0.4982 | 57.3862 | 3.77 | 56.3735 |
| Tc-KC* pH 4b | 7.2851 | 7.7866 | 0.5015 | | | |
| Tc-KC* pH 7a | 7.2475 | 7.7484 | 0.5009 | 57.0216 | 5.27 | 56.0013 |
| Tc-KC* pH 7b | 7.3353 | 7.8575 | 0.5222 | | | |
| Tc-KC* pH 9.5a | 7.3013 | 7.8039 | 0.5026 | 56.5021 | 9.89 | 55.4830 |
| Tc-KC* pH 9.5b | 7.3388 | 7.843 | 0.5042 | | | |

| Sample Name | wt. of vial | wt. of vial + sample | sample wt. | Container wt. before sampling & pH | Measured pH | Container wt. after sampling & pH |
|----------------|-------------|-------------------------|------------|--|----------------|---|
| | 4 hours | | | | | |
| Tc-K* pH 4a | 7.2949 | 7.7924 | 0.4975 | 55.1933 | 5.98 | 54.1612 |
| Tc-K* pH 4b | 7.3078 | 7.8065 | 0.4987 | | | |
| Tc-K* pH 7a | 7.2748 | 7.7723 | 0.4975 | 55.3091 | 6.69 | 54.2878 |
| Tc-K* pH 7b | 7.2169 | 7.7187 | 0.5018 | | | |
| Tc-K* pH 9.5a | 7.2972 | 7.7967 | 0.4995 | 54.9154 | 9.90 | 53.8998 |
| Tc-K* pH 9.5b | 7.2287 | 7.7307 | 0.5020 | | | |
| | | | | | | |
| Tc-KC* pH 4a | 7.3155 | 7.8136 | 0.4981 | 55.3517 | 3.78 | 54.3296 |
| Tc-KC* pH 4b | 7.2727 | 7.7735 | 0.5008 | | | |
| Tc-KC* pH 7a | 7.2996 | 7.7985 | 0.4989 | 54.9818 | 5.28 | 53.9597 |
| Tc-KC* pH 7b | 7.2764 | 7.7776 | 0.5012 | | | |
| Tc-KC* pH 9.5a | 7.255 | 7.7561 | 0.5011 | 54.4631 | 9.89 | 53.4309 |
| Tc-KC* pH 9.5b | 7.2627 | 7.7658 | 0.5031 | | | |

7/20/99 AJ

| | (1 day) | | | | | |
|----------------|---------|--------|--------|---------|------|---------|
| Tc-K* pH 4a | 7.3038 | 7.8047 | 0.5009 | 54.1225 | 6.24 | 53.1071 |
| Tc-K* pH 4b | 7.2953 | 7.7979 | 0.5026 | | | |
| Tc-K* pH 7a | 7.3574 | 7.8608 | 0.5034 | 54.2764 | 6.87 | 53.2641 |
| Tc-K* pH 7b | 7.2704 | 7.7703 | 0.4999 | | | |
| Tc-K* pH 9.5a | 7.3494 | 7.8553 | 0.5059 | 53.8830 | 9.84 | 52.8785 |
| Tc-K* pH 9.5b | 7.2473 | 7.7486 | 0.5013 | | | |
| Tc-KC* pH 4a | 7.2372 | 7.7373 | 0.5001 | 54.3139 | 3.78 | 53.2975 |
| Tc-KC* pH 4b | 7.3241 | 7.8262 | 0.5021 | | | |
| Tc-KC* pH 7a | 7.3254 | 7.8256 | 0.5002 | 53.9454 | 5.25 | 52.9249 |
| Tc-KC* pH 7b | 7.2660 | 7.7699 | 0.5039 | | | |
| Tc-KC* pH 9.5a | 7.3420 | 7.8448 | 0.5028 | 53.4172 | 9.85 | 52.3868 |
| Tc-KC* pH 9.5b | 7.2270 | 7.7325 | 0.5055 | | | |

| Sample Name | wt. of vial | wt. of vial + sample | sample wt. | Container wt. before sampling & pH measurements | Measured pH | Container wt. after sampling & pH measurements |
|----------------|-------------|----------------------|------------|---|-------------|--|
| 7/21/99 | (2 days) | | | | | |
| Tc-K* pH 4a | 7.2337 | 7.7373 | 0.5036 | 53.076 | 6.39 | 52.0574 |
| Tc-K* pH 4b | 7.2378 | 7.7403 | 0.5025 | | | |
| Tc-K* pH 7a | 7.2528 | 7.7524 | 0.4996 | 53.2466 | 7.02 | 52.2485 |
| Tc-K* pH 7b | 7.3386 | 7.8394 | 0.5008 | | | |
| Tc-K* pH 9.5a | 7.2498 | 7.7493 | 0.4995 | 52.8597 | 9.81 | 51.8592 |
| Tc-K* pH 9.5b | 7.2825 | 7.7838 | 0.5013 | | | |
| Tc-KC* pH 4a | 7.2594 | 7.7558 | 0.4964 | 53.281 | 3.77 | 52.2864 |
| Tc-KC* pH 4b | 7.3282 | 7.8269 | 0.4987 | | | |
| Tc-KC* pH 7a | 7.2661 | 7.7647 | 0.4986 | 52.408 | 5.25 | 51.9406 |
| Tc-KC* pH 7b | 7.3528 | 7.8526 | 0.4998 | | | |
| Tc-KC* pH 9.5a | 7.3674 | 7.8666 | 0.4992 | 52.3688 | 9.85 | 51.3803 |
| Tc-KC* pH 9.5b | 7.2719 | 7.7724 | 0.5005 | | | |

C14 Eff (0-156 keV) = 96.63 %
 C14 CHI SQUARE IPA DATA PROCESSED - 20-Jul-1999 09:42
 C14 Chi Square = 17.93
 H3 IPA DATA PROCESSED - 20-Jul-1999 09:43
 H3 Eff (0-18.6 keV) = 65.71 %
 H3 CHI SQUARE IPA DATA PROCESSED - 20-Jul-1999 09:54
 H3 Chi Square = 24.39
 BKG IPA DATA PROCESSED - 20-Jul-1999 10:54
 Bkg (0-18.6 keV) = 15.60 cpm
 Bkg (0-156 keV) = 22.08 cpm
 C14 E²/B (1-156 keV) = 557.32
 H3 E²/B (1-18.6 keV) = 275.45

21 Jul 1999 03:44 ALPHA/BETA - 1.09
 Protocol #143 TC-99 CPM User :

Time: 999.99
 Data Mode: CPM Nuclide: MANUAL
 Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 300 | 0 | 0.0 | 19.08 | |
| Region B: | 2.0 - 300 | 0 | 1.0 | 17.75 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.97 | |

Quench Indicator: SIS
 BETA CPM TC-99
 Coincidence Time(ns): 18
 Delay Before Burst(ns): Normal

1%

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS | FLAG |
|-------------------|--------|------------|------------|------------|------|--------|
| 1 | 999.99 | 19.08 | 1.45 | 17.75 | 1.50 | 24.97 |
| 2 | 76.39 | 511.35 | 1.03 | 505.91 | 1.04 | 511.44 |
| 3 | 76.24 | 512.34 | 1.03 | 506.94 | 1.04 | 512.68 |
| 4 | 75.94 | 514.70 | 1.03 | 509.02 | 1.04 | 514.68 |
| 5 | 76.42 | 511.14 | 1.03 | 505.69 | 1.04 | 511.10 |
| 6 | 75.40 | 517.67 | 1.03 | 512.78 | 1.04 | 517.45 |
| 7 | 75.33 | 518.65 | 1.03 | 513.25 | 1.04 | 518.45 |
| 1 MISSING TUBE(S) | | | | | | |
| 9 | 76.65 | 509.38 | 1.03 | 504.12 | 1.04 | 509.81 |
| 10 | 75.64 | 516.08 | 1.03 | 511.08 | 1.04 | 516.09 |
| 11 | 76.06 | 513.36 | 1.03 | 508.20 | 1.04 | 513.30 |
| 12 | 75.81 | 515.00 | 1.03 | 509.89 | 1.04 | 515.01 |
| 13 | 74.59 | 523.87 | 1.03 | 518.52 | 1.04 | 523.47 |
| 14 | 74.02 | 528.24 | 1.03 | 522.69 | 1.04 | 528.26 |
| 1 MISSING TUBE(S) | | | | | | |
| 16 | 76.74 | 508.94 | 1.03 | 503.49 | 1.04 | 509.02 |
| 17 | 77.06 | 506.53 | 1.03 | 501.42 | 1.04 | 506.83 |
| 18 | 76.37 | 511.42 | 1.03 | 506.10 | 1.04 | 510.79 |
| 19 | 76.24 | 512.38 | 1.03 | 506.94 | 1.04 | 512.43 |
| 20 | 75.79 | 515.36 | 1.03 | 510.02 | 1.04 | 515.21 |
| 21 | 75.35 | 518.75 | 1.03 | 513.13 | 1.04 | 519.19 |

Tc-K* 1 hr.
 Tc-KC* 1 hr.
 Tc-K* 3 hr.

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|-------|--------|------|--------|------|--------|------|--------|---------------|
| 23 | 76.00 | 514.29 | 1.03 | 508.57 | 1.04 | 513.95 | 1.04 | 173.63 | Tc-KC* 3hr |
| 24 | 76.40 | 511.79 | 1.03 | 505.86 | 1.04 | 511.77 | 1.04 | 172.52 | |
| 25 | 76.57 | 509.69 | 1.03 | 504.65 | 1.04 | 509.86 | 1.04 | 173.13 | |
| 26 | 76.52 | 510.52 | 1.03 | 505.03 | 1.04 | 510.66 | 1.04 | 172.31 | |
| 27 | 74.70 | 523.20 | 1.03 | 517.75 | 1.04 | 523.28 | 1.04 | 172.80 | |
| 28 | 74.65 | 523.50 | 1.03 | 518.12 | 1.04 | 523.39 | 1.04 | 172.09 | |

WARNING: USER HAS MODIFIED COUNT CONDITIONS

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|--------------|
| 30 | 8.66 | 501.36 | 3.09 | 495.88 | 3.11 | 500.55 | 3.11 | 171.29 | Tc-K* 4hr |
| 31 | 8.41 | 514.93 | 3.10 | 510.79 | 3.10 | 514.03 | 3.12 | 176.84 | |
| 32 | 8.33 | 520.90 | 3.09 | 516.22 | 3.10 | 521.25 | 3.11 | 173.93 | |
| 33 | 8.45 | 512.99 | 3.09 | 508.52 | 3.10 | 512.31 | 3.11 | 173.02 | |
| 34 | 8.33 | 522.58 | 3.09 | 515.74 | 3.10 | 521.01 | 3.11 | 169.99 | |
| 35 | 8.32 | 522.51 | 3.09 | 516.51 | 3.10 | 522.15 | 3.11 | 171.97 | |

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|--------|
| 37 | 8.40 | 518.30 | 3.09 | 511.77 | 3.10 | 518.48 | 3.10 | 173.67 | Tc-KC* |
| 38 | 8.42 | 517.38 | 3.09 | 510.64 | 3.10 | 517.31 | 3.10 | 172.33 | |
| 39 | 8.49 | 510.13 | 3.10 | 505.81 | 3.11 | 510.01 | 3.11 | 173.80 | |

22 Jul 1999 11:57
Protocol #143ALPHA/BETA - 1.09
TC-99 CPM

User

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|----|------|--------|-------|--------|-------|--------|-------|--------|------|
| 40 | 8.56 | 506.04 | 3.10 | 501.41 | 3.11 | 505.76 | 3.11 | 171.04 | 4hr |
| 41 | 8.54 | 508.56 | 3.09 | 503.09 | 3.11 | 507.94 | 3.11 | 174.13 | |
| 42 | 8.36 | 519.56 | 3.09 | 514.55 | 3.10 | 519.77 | 3.11 | 174.96 | |

12 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|---------------|
| 55 | 8.34 | 518.69 | 3.10 | 515.10 | 3.10 | 517.96 | 3.12 | 176.66 | Tc-K* 1day |
| 56 | 8.18 | 529.82 | 3.09 | 525.77 | 3.10 | 528.82 | 3.11 | 174.36 | |
| 57 | 8.50 | 511.39 | 3.09 | 505.90 | 3.10 | 513.38 | 3.10 | 174.10 | |
| 58 | 8.30 | 523.33 | 3.09 | 518.15 | 3.10 | 523.47 | 3.11 | 174.11 | |
| 59 | 8.42 | 515.13 | 3.09 | 510.16 | 3.10 | 515.65 | 3.11 | 177.26 | |
| 60 | 8.60 | 505.57 | 3.09 | 499.34 | 3.11 | 505.38 | 3.11 | 171.30 | |

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|----------------|
| 62 | 8.77 | 494.15 | 3.10 | 489.21 | 3.11 | 493.28 | 3.12 | 171.24 | Tc-KC* 1day |
| 63 | 8.20 | 529.46 | 3.09 | 524.93 | 3.10 | 529.06 | 3.11 | 174.51 | |
| 64 | 8.76 | 495.54 | 3.09 | 490.13 | 3.11 | 494.44 | 3.12 | 172.80 | |
| 65 | 8.31 | 524.00 | 3.09 | 517.15 | 3.10 | 524.61 | 3.10 | 170.18 | |
| 66 | 8.21 | 529.16 | 3.09 | 523.66 | 3.10 | 527.65 | 3.11 | 171.70 | |
| 67 | 8.22 | 529.71 | 3.09 | 523.25 | 3.10 | 530.75 | 3.10 | 173.59 | |

5 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|----------------|
| 73 | 8.79 | 492.30 | 3.10 | 487.83 | 3.11 | 492.32 | 3.12 | 171.20 | Tc-K* 2days |
| 74 | 8.47 | 511.15 | 3.10 | 507.04 | 3.11 | 510.69 | 3.12 | 175.92 | |
| 75 | 8.34 | 520.85 | 3.09 | 515.10 | 3.10 | 520.36 | 3.11 | 171.90 | |
| 76 | 8.43 | 515.80 | 3.09 | 509.65 | 3.10 | 514.41 | 3.11 | 172.98 | |
| 77 | 8.45 | 513.82 | 3.09 | 508.40 | 3.10 | 514.91 | 3.11 | 171.85 | |
| 78 | 8.13 | 534.43 | 3.09 | 529.61 | 3.10 | 532.97 | 3.11 | 169.76 | |

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|-----------------|
| 80 | 8.60 | 503.48 | 3.10 | 499.23 | 3.11 | 505.26 | 3.11 | 172.10 | Tc-KC* 2days |
| 81 | 8.46 | 513.19 | 3.09 | 507.90 | 3.10 | 512.38 | 3.11 | 170.35 | |
| 82 | 8.51 | 510.30 | 3.09 | 504.46 | 3.11 | 511.34 | 3.11 | 172.79 | |
| 83 | 8.46 | 513.55 | 3.09 | 508.37 | 3.10 | 513.33 | 3.11 | 171.71 | |
| 84 | 8.41 | 516.24 | 3.09 | 511.03 | 3.10 | 517.01 | 3.11 | 174.39 | |
| 85 | 8.45 | 513.94 | 3.09 | 508.29 | 3.11 | 513.73 | 3.11 | 174.47 | |

7/23/99

AJ

| 7/23/99 | 4 days | | | | | |
|----------------|--------|--------|--------|---------|------|---------|
| Tc-K* pH 4a | 7.2923 | 7.7957 | 0.5034 | 52.0098 | 6.52 | 50.9927 |
| Tc-K* pH 4b | 7.2568 | 7.7588 | 0.5389 | | | |
| Tc-K* pH 7a | 7.3053 | 7.8073 | 0.4535 | 52.2226 | 6.92 | 51.1344 |
| Tc-K* pH 7b | 7.2453 | 7.7454 | 0.562 | | | |
| Tc-K* pH 9.5a | 7.2807 | 7.7847 | 0.4647 | 51.8228 | 9.81 | 50.7633 |
| Tc-K* pH 9.5b | 7.2641 | 7.7653 | 0.5206 | | | |
| | | | | | | |
| Tc-KC* pH 4a | 7.3293 | 7.8321 | 0.5028 | 52.2532 | 3.75 | 51.2223 |
| Tc-KC* pH 4b | 7.2513 | 7.7554 | 0.5041 | | | |
| Tc-KC* pH 7a | 7.3309 | 7.8333 | 0.5024 | 51.908 | 5.29 | 50.8846 |
| Tc-KC* pH 7b | 7.276 | 7.7806 | 0.5046 | | | |
| Tc-KC* pH 9.5a | 7.3037 | 7.808 | 0.5043 | 51.3452 | | 50.3463 |
| Tc-KC* pH 9.5b | 7.2147 | 7.7189 | 0.5042 | | 9.85 | |

24 Jul 1999 05:49 ALPHA/BETA - 1.09
Protocol #:43 TC-99 CPM

User :

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 300 | 0 | 0.0 | 18.97 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.74 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.73 | |

Quench Indicator: SIS
BETA CPM TC-99
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|-------------------|--------|--------|-------|--------|-------|--------|-------|--------|--------|
| 1 | 999.99 | 18.97 | 1.45 | 17.74 | 1.50 | 24.73 | 1.27 | 124.89 | B |
| 2 | 8.70 | 498.27 | 3.10 | 493.41 | 3.11 | 498.95 | 3.11 | 176.08 | |
| 3 | 8.44 | 515.51 | 3.09 | 509.04 | 3.10 | 514.49 | 3.11 | 173.63 | Tc-K* |
| 4 | 8.56 | 506.96 | 3.09 | 501.42 | 3.11 | 505.76 | 3.11 | 177.92 | 1 hr. |
| 5 | 8.27 | 526.25 | 3.09 | 519.75 | 3.10 | 526.30 | 3.10 | 171.83 | |
| 6 | 8.21 | 528.29 | 3.09 | 523.80 | 3.10 | 528.74 | 3.11 | 173.48 | |
| 7 | 8.39 | 517.74 | 3.09 | 512.65 | 3.10 | 518.06 | 3.11 | 176.21 | |
| 1 MISSING TUBE(S) | | | | | | | | | |
| 9 | 8.54 | 509.25 | 3.09 | 502.64 | 3.11 | 509.93 | 3.10 | 173.88 | |
| 10 | 8.46 | 512.12 | 3.09 | 507.67 | 3.11 | 511.56 | 3.11 | 178.27 | Tc-KC* |
| 11 | 8.62 | 504.23 | 3.09 | 498.04 | 3.11 | 503.93 | 3.11 | 171.03 | 1 hr. |
| 12 | 8.42 | 516.90 | 3.09 | 510.41 | 3.10 | 516.96 | 3.10 | 174.11 | |
| 13 | 8.25 | 526.24 | 3.09 | 521.29 | 3.10 | 527.15 | 3.10 | 175.04 | |
| 14 | 8.32 | 522.86 | 3.09 | 516.88 | 3.10 | 521.91 | 3.11 | 178.49 | |
| 1 MISSING TUBE(S) | | | | | | | | | |
| 16 | 8.31 | 523.87 | 3.09 | 517.52 | 3.10 | 524.13 | 3.10 | 174.61 | |
| 17 | 8.28 | 524.87 | 3.09 | 519.34 | 3.10 | 523.58 | 3.11 | 175.50 | Tc-K* |
| 18 | 8.56 | 507.20 | 3.09 | 501.42 | 3.11 | 506.35 | 3.11 | 171.73 | 3 hr. |
| 19 | 8.90 | 488.33 | 3.09 | 481.81 | 3.11 | 487.41 | 3.11 | 174.34 | |
| 20 | 8.29 | 523.49 | 3.09 | 518.93 | 3.10 | 524.00 | 3.11 | 174.93 | |
| 21 | 8.58 | 506.55 | 3.09 | 500.68 | 3.11 | 507.90 | 3.10 | 176.20 | |
| 1 MISSING TUBE(S) | | | | | | | | | |
| 23 | 8.70 | 500.00 | 3.09 | 493.41 | 3.11 | 499.64 | 3.11 | 172.28 | |
| 24 | 8.32 | 521.53 | 3.09 | 516.76 | 3.10 | 520.46 | 3.11 | 174.13 | Tc-KC* |
| 25 | 8.66 | 501.47 | 3.09 | 495.77 | 3.11 | 500.33 | 3.11 | 176.24 | 3 hr. |
| 26 | 8.51 | 511.58 | 3.09 | 504.94 | 3.10 | 511.46 | 3.10 | 176.26 | |
| 27 | 8.35 | 520.55 | 3.09 | 514.60 | 3.10 | 518.74 | 3.11 | 173.08 | |
| 28 | 8.29 | 523.85 | 3.09 | 518.57 | 3.10 | 524.37 | 3.10 | 175.92 | |
| 8 MISSING TUBE(S) | | | | | | | | | |

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|--------|
| 37 | 8.34 | 521.20 | 3.09 | 515.35 | 3.10 | 521.31 | 3.10 | 178.52 | |
| 38 | 8.50 | 510.32 | 3.09 | 505.44 | 3.10 | 510.80 | 3.11 | 176.98 | Tc-K* |
| 39 | 8.46 | 513.54 | 3.09 | 507.91 | 3.10 | 515.11 | 3.10 | 179.66 | 4 days |
| 40 | 8.47 | 513.03 | 3.09 | 507.05 | 3.11 | 511.52 | 3.11 | 176.67 | |
| 41 | 8.43 | 514.24 | 3.09 | 509.54 | 3.10 | 514.89 | 3.11 | 175.26 | |
| 42 | 8.50 | 510.32 | 3.09 | 505.08 | 3.11 | 512.09 | 3.10 | 174.41 | |

1 MISSING TUBE(S)

| | | | | | | | | | |
|----|------|--------|------|--------|------|--------|------|--------|--------|
| 44 | 8.28 | 523.66 | 3.09 | 519.34 | 3.10 | 523.82 | 3.11 | 175.31 | |
| 45 | 8.45 | 512.39 | 3.10 | 508.53 | 3.10 | 513.02 | 3.11 | 177.33 | Tc-K* |
| 46 | 8.53 | 507.52 | 3.10 | 503.48 | 3.11 | 506.34 | 3.12 | 177.79 | 4 days |
| 47 | 8.12 | 536.20 | 3.08 | 529.80 | 3.10 | 535.25 | 3.10 | 174.62 | |
| 48 | 8.17 | 531.58 | 3.09 | 526.45 | 3.10 | 532.43 | 3.10 | 173.09 | |

24 Jul 1999 11:06 ALPHA/BETA - 1.09
Protocol #:43 TC-99 CPM

User :

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|----|------|--------|-------|--------|-------|--------|-------|--------|------|
| 49 | 8.20 | 529.93 | 3.09 | 524.58 | 3.10 | 529.30 | 3.11 | 171.27 | |

| 7/27/99 | | | | | |
|----------------|-------------|----------------------|------------------------------------|-------------|-----------------------------------|
| Sample Name | wt. of vial | wt. of vial + sample | Container wt. before sampling & pH | Measured pH | Container wt. after sampling & pH |
| | 8 days | | | | |
| Tc-K* pH 4a | 7.3479 | 7.8512 | 50.8921 | 6.52 | 49.8349 |
| Tc-K* pH 4b | 7.2764 | 7.7818 | | | |
| Tc-K* pH 7a | 7.2221 | 7.727 | 51.0435 | 7.08 | 50.0197 |
| Tc-K* pH 7b | 7.2583 | 7.7636 | | | |
| Tc-K* pH 9.5a | 7.3426 | 7.8492 | 50.6729 | 9.83 | 49.6367 |
| Tc-K* pH 9.5b | 7.2481 | 7.7559 | | | |
| | | | | | |
| Tc-KC* pH 4a | 7.3645 | 7.8685 | 51.1447 | 3.78 | 50.1166 |
| Tc-KC* pH 4b | 7.2812 | 7.7868 | | | |
| Tc-KC* pH 7a | 7.3286 | 7.8329 | 50.8103 | 5.77 | 49.7732 |
| Tc-KC* pH 7b | 7.1917 | 7.697 | | | |
| Tc-KC* pH 9.5a | 7.323 | 7.8276 | 50.2721 | 9.8 | 49.2361 |
| Tc-KC* pH 9.5b | 7.2655 | 7.7723 | | | |

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 27-Jul-1999 11:11
C14 Eff (0-156 keV) = 96.32 %
C14 CHI SQUARE IPA DATA PROCESSED - 27-Jul-1999 11:22
C14 Chi Square = 11.34
H3 IPA DATA PROCESSED - 27-Jul-1999 11:23
H3 Eff (0-18.6 keV) = 65.45 %
H3 CHI SQUARE IPA DATA PROCESSED - 27-Jul-1999 11:34
H3 Chi Square = 23.27
BKG IPA DATA PROCESSED - 27-Jul-1999 12:34
Bkg (0-18.6 keV) = 16.93 cpm
Bkg (0-156 keV) = 23.80 cpm
C14 E²/B (1-156 keV) = 510.59
H3 E²/B (1-18.6 keV) = 251.21

28 Jul 1999 05:23

ALPHA/BETA - 1.09

Protocol #:43

TC-99 CPM

User

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 300 | 0 | 0.0 | 18.71 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.40 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.45 | |

Quench Indicator: SIS

BETA CPM TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS | FLAG |
|-------------------|--------|------------|------------|------------|------|---------------------|
| 1 | 999.99 | 18.71 | 1.46 | 17.40 | 1.52 | 24.45 1.28 123.36 B |
| 2 | 8.49 | 510.97 | 3.09 | 506.63 | 3.10 | 509.82 3.11 179.20 |
| 3 | 8.55 | 506.79 | 3.09 | 502.37 | 3.10 | 507.36 3.11 173.89 |
| 4 | 8.34 | 520.74 | 3.09 | 515.69 | 3.10 | 520.15 3.11 173.33 |
| 5 | 8.37 | 519.64 | 3.09 | 514.38 | 3.10 | 518.92 3.11 173.62 |
| 6 | 8.15 | 532.95 | 3.09 | 528.25 | 3.10 | 532.36 3.11 180.85 |
| 7 | 8.24 | 528.98 | 3.08 | 522.65 | 3.10 | 530.52 3.09 173.45 |
| 1 MISSING TUBE(S) | | | | | | |
| 9 | 8.45 | 514.07 | 3.09 | 509.11 | 3.10 | 512.47 3.11 176.94 |
| 10 | 8.35 | 520.21 | 3.09 | 515.18 | 3.10 | 520.70 3.10 175.29 |
| 11 | 8.48 | 511.12 | 3.09 | 506.78 | 3.10 | 511.87 3.11 178.78 |
| 12 | 8.28 | 524.53 | 3.09 | 519.80 | 3.10 | 524.22 3.11 174.49 |
| 13 | 8.26 | 528.51 | 3.08 | 521.34 | 3.10 | 529.30 3.09 173.56 |
| 14 | 8.21 | 529.16 | 3.09 | 524.38 | 3.10 | 528.41 3.11 177.72 |
| 2 MISSING TUBE(S) | | | | | | |
| 17 | 8.52 | 508.64 | 3.09 | 504.55 | 3.10 | 508.29 3.11 175.92 |
| 18 | 8.48 | 512.54 | 3.09 | 506.89 | 3.10 | 511.16 3.11 175.71 |

*Tc-K**
8 days

*Tc-K**
9 days

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 29-Jul-1999 14:38
C14 Eff (0-156 keV) = 96.59 %
C14 CHI SQUARE IPA DATA PROCESSED - 29-Jul-1999 14:48
C14 Chi Square = 13.08
H3 IPA DATA PROCESSED - 29-Jul-1999 14:50
H3 Eff (0-18.6 keV) = 65.53 %
H3 CHI SQUARE IPA DATA PROCESSED - 29-Jul-1999 15:00
H3 Chi Square = 12.18
BKG IPA DATA PROCESSED - 29-Jul-1999 16:01
Bkg (0-18.6 keV) = 15.80 cpm
Bkg (0-156 keV) = 22.55 cpm
C14 E²/B (1-156 keV) = 537.25
H3 E²/B (1-18.6 keV) = 268.29

30 Jul 1999 08:50

ALPHA/BETA - 1.09

Protocol #:43

TC-99 CPM

User

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 300 | 0 | 0.0 | 19.09 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.85 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.81 | |

Quench Indicator: SIS

BETA CPM TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS | FLAG |
|----|--------|------------|------------|------------|------|---------------------|
| 1 | 999.99 | 19.09 | 1.45 | 17.85 | 1.50 | 24.81 1.27 127.49 B |
| 2 | 0.90 | 5019.79 | 2.98 | 4965.49 | 3.00 | 5022.97 2.98 177.45 |
| 3 | 0.89 | 5073.04 | 2.98 | 5014.74 | 3.00 | 5071.82 2.98 174.86 |
| 4 | 8.47 | 511.60 | 3.09 | 506.83 | 3.11 | 510.02 3.12 176.65 |
| 5 | 8.65 | 502.06 | 3.09 | 496.02 | 3.11 | 501.78 3.11 176.41 |

*TC-P**

300 ppb
Tc-99

30 ppb
Tc-99

| Sample Name | wt. of vial | wt. of vial + sample | Container wt. before sampling & pH | Measured pH | Container wt. after sampling & pH |
|----------------|----------------|----------------------|------------------------------------|-------------|-----------------------------------|
| 7/30/99 | 11 days | | | | |
| Tc-K* pH 4a | 7.3493 | 7.8512 | 49.7435 | 6.58 | 48.707 |
| Tc-K* pH 4b | 7.4338 | 7.9356 | | | |
| Tc-K* pH 7a | 7.3036 | 7.8088 | 49.9463 | 7.15 | 48.8969 |
| Tc-K* pH 7b | 7.2929 | 7.7958 | | | |
| Tc-K* pH 9.5a | 7.2909 | 7.7941 | 49.5636 | 9.78 | 48.5287 |
| Tc-K* pH 9.5b | 7.3159 | 7.8218 | | | |
| Tc-KC* pH 4a | 7.3778 | 7.8833 | 50.0607 | 3.83 | 49.0282 |
| Tc-KC* pH 4b | 7.2791 | 7.7826 | | | |
| Tc-KC* pH 7a | 7.3528 | 7.8565 | 49.7171 | 5.75 | 48.6835 |
| Tc-KC* pH 7b | 7.3274 | 7.8323 | | | |
| Tc-KC* pH 9.5a | 7.3447 | 7.8552 | 49.1749 | 9.77 | 48.1343 |
| Tc-KC* pH 9.5b | 7.2646 | 7.7721 | | | |
| 8/5/99 | 17 days | | | | |
| Tc-K* pH 4a | 7.3316 | 7.8358 | 48.5187 | 6.62 | 47.4876 |
| Tc-K* pH 4b | 7.2551 | 7.7581 | | | |
| Tc-K* pH 7a | 7.3093 | 7.7902 | 48.7494 | 7.09 | 47.7151 |
| Tc-K* pH 7b | 7.3544 | 7.8597 | | | |
| Tc-K* pH 9.5a | 7.3807 | 7.8859 | 48.3938 | 9.64 | 47.3442 |
| Tc-K* pH 9.5b | 7.371 | 7.8779 | | | |
| Tc-KC* pH 4a | 7.3428 | 7.8471 | 48.887 | 3.82 | 47.8603 |
| Tc-KC* pH 4b | 7.2226 | 7.7266 | | | |
| Tc-KC* pH 7a | 7.3282 | 7.8309 | 48.5358 | 5.77 | 47.5123 |
| Tc-KC* pH 7b | 7.2619 | 7.7668 | | | |
| Tc-KC* pH 9.5a | 7.3226 | 7.8244 | 48.0179 | 9.65 | 46.9898 |
| Tc-KC* pH 9.5b | 7.3712 | 7.8774 | | | |

31 Jul 1999 08:49
Protocol #:43

ALPHA/BETA - 1.09
TC-99 CPM

User 19

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 300 | 0 | 0.0 | 19.12 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.91 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.93 | |

Quench Indicator: SIS
BETA CPM TC-99
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|----|-----------------|---------|-------|---------|-------|---------|-------|--------|-------------------|
| 1 | 999.99 | 19.12 | 1.45 | 17.91 | 1.49 | 24.93 | 1.27 | 124.40 | B |
| 2 | 8.34 | 519.49 | 3.09 | 515.31 | 3.10 | 519.67 | 3.11 | 177.00 | |
| 3 | 8.28 | 525.08 | 3.09 | 519.29 | 3.10 | 523.98 | 3.11 | 173.92 | |
| 4 | 8.43 | 513.86 | 3.10 | 509.85 | 3.10 | 514.21 | 3.11 | 172.94 | Tc-K* 11 days |
| 5 | 8.45 | 511.88 | 3.10 | 508.25 | 3.11 | 511.52 | 3.12 | 175.29 | |
| 6 | 8.45 | 514.49 | 3.09 | 508.13 | 3.11 | 514.83 | 3.11 | 172.65 | |
| 7 | 8.37 | 520.19 | 3.09 | 513.52 | 3.10 | 522.02 | 3.10 | 175.11 | |
| 1 | MISSING TUBE(S) | | | | | | | | |
| 9 | 8.42 | 516.39 | 3.09 | 510.48 | 3.10 | 516.40 | 3.11 | 175.18 | |
| 10 | 8.22 | 527.35 | 3.09 | 522.85 | 3.10 | 527.02 | 3.11 | 174.49 | |
| 11 | 8.20 | 529.66 | 3.09 | 524.41 | 3.10 | 529.46 | 3.11 | 174.72 | Tc-KC* 11 days |
| 12 | 8.46 | 513.74 | 3.09 | 507.98 | 3.10 | 514.55 | 3.10 | 175.41 | |
| 13 | 8.22 | 530.51 | 3.08 | 523.82 | 3.10 | 531.88 | 3.10 | 176.80 | |
| 14 | 8.09 | 536.13 | 3.09 | 531.54 | 3.10 | 536.01 | 3.11 | 177.37 | |
| 1 | MISSING TUBE(S) | | | | | | | | |
| 16 | 8.45 | 515.32 | 3.09 | 508.25 | 3.11 | 514.48 | 3.11 | 176.50 | |
| 17 | 8.49 | 512.09 | 3.09 | 505.53 | 3.11 | 512.41 | 3.11 | 179.70 | |
| 1 | MISSING TUBE(S) | | | | | | | | |
| 19 | 8.54 | 508.75 | 3.09 | 502.94 | 3.11 | 507.39 | 3.11 | 173.38 | |
| 20 | 8.32 | 522.35 | 3.09 | 516.47 | 3.10 | 522.18 | 3.11 | 176.46 | |
| 21 | 8.54 | 510.04 | 3.09 | 502.59 | 3.11 | 509.61 | 3.11 | 176.62 | |
| 22 | 8.58 | 506.99 | 3.09 | 500.63 | 3.11 | 506.42 | 3.11 | 173.79 | |
| 23 | 8.42 | 517.22 | 3.09 | 510.95 | 3.10 | 517.59 | 3.10 | 172.70 | |
| 24 | 8.57 | 507.02 | 3.09 | 501.35 | 3.11 | 507.86 | 3.11 | 174.36 | Tc-P* 30PPb 0 day |
| 25 | 8.32 | 522.47 | 3.09 | 516.83 | 3.10 | 522.42 | 3.11 | 172.77 | |
| 26 | 8.20 | 530.39 | 3.09 | 524.78 | 3.10 | 530.80 | 3.10 | 177.76 | |
| 27 | 8.44 | 513.34 | 3.10 | 508.75 | 3.11 | 513.10 | 3.11 | 174.90 | |
| 28 | 8.45 | 514.72 | 3.09 | 508.01 | 3.11 | 515.42 | 3.10 | 172.49 | |
| 8 | MISSING TUBE(S) | | | | | | | | |
| 37 | 0.88 | 5122.92 | 2.98 | 5065.05 | 3.00 | 5122.80 | 2.99 | 173.46 | |
| 38 | 0.90 | 4991.99 | 2.99 | 4947.65 | 3.00 | 4991.74 | 2.99 | 172.66 | |
| 39 | 0.90 | 4988.66 | 2.99 | 4935.43 | 3.01 | 4987.29 | 2.99 | 176.36 | |
| 40 | 0.92 | 4922.18 | 2.98 | 4869.05 | 2.99 | 4920.72 | 2.98 | 173.36 | Tc-P* 30PPb 0 day |
| 41 | 0.90 | 5026.43 | 2.98 | 4967.65 | 3.00 | 5027.29 | 2.98 | 175.53 | |
| 42 | 0.90 | 5033.10 | 2.98 | 4986.54 | 2.99 | 5033.96 | 2.98 | 175.04 | |
| 43 | 1.00 | 5057.88 | 2.82 | 5005.09 | 2.83 | 5057.07 | 2.82 | 175.15 | |
| 44 | 0.88 | 5117.24 | 2.99 | 5066.19 | 3.00 | 5115.98 | 2.99 | 173.90 | |
| 45 | 0.91 | 4919.34 | 3.00 | 4882.09 | 3.01 | 4921.22 | 3.00 | 174.06 | |
| 46 | 0.86 | 5232.04 | 2.99 | 5176.28 | 3.00 | 5229.72 | 2.99 | 175.07 | |

7/30/99

AJ

Objective:- To investigate the precipitation of technetium in 300 ppb & 30 ppb Tc-99 spike solution at pH range 3-7.

EQUIPMENT: Liquid Scintillation Analyzer (Packard 2500TR/AB)
ORION ph/mV/ISE/ C meter
Combination ph electrode
Automatic temperature compensator probe
Analytical balances (Mettler 4600 and 240AE)
Gyratory shaker

SUPPLIES: pH buffers (pH= 4,7,10)
50 ml polycarbonate centrifuge tubes (43 ml capacity)
LSA vials 7ml
Repipettor for transfer of scintillation cocktail
Packard A/B cocktail
Eppendorf pipet and tips (various volumes)
gamma-alumina (lot C16F19)
1 L FEP teflon bottle
Tc-99 stock solution (**spike #43A**)
HCl (lot 956110) various concentrations; e.g., 1.0 M, 0.1 M, etc...
NaOH (lot 976631) various concentrations; e.g., 1.0 M, 0.1 M, etc...

PROCEDURE:

1. In a precleaned 1L FEP bottle, prepare 280 g of 2.95×10^{-7} m (29.4 ppb) Tc-99 solution by diluting 28 g of a 2.95×10^{-6} m (294 ppb) ^{99}Tc stock solution (**spike #43A**; in 0.1 m NaCl matrix; prepared from a commercial ^{99}Tc spike) to a total of 280 g by taring 0.1 m NaCl solution into the FEP bottle on a Mettler 4600 balance.
2. Label 3 50 ml polycarbonate centrifuge tubes Tc-P* pH_i (where i is the approximate pH of each solution, see Table) and pre-weigh. Record weight.
3. Add 35 g of the 29.4 ppb Tc solution to each tube and record weight.
4. Label 3 50 ml polycarbonate centrifuge tubes Tc-P* pH_i (where i is the approximate pH of each solution, see Table) and pre-weigh. Record weight.

5. Add 40 g of the 294 ppb Tc solution to each tube and record weight.
6. Sample Tc-P* to determine initial Tc concentration of both spikes, 294 ppb and 29.4 ppb. Label (e.g. Tc-P*a 30 ppb and Tc-P*b 30 ppb) and preweigh two LSA vials. Withdraw

two 0.5 ml samples and transfer to the LSA vials. Reweigh vials and record weight. Add 5 ml of scintillation cocktail to each vial. Homogenize sample and set aside for LSA.

7. Adjust pH by adding HCl or NaOH using an Eppendorf pipetter. The amount to be added to each solution is given in the Table. After pH adjustment record weight of containers.

| | 7/29/99 | 7/30/99 | 7/30/99 | 7/30/99 | 7/30/99 | 7/30/99 |
|----------------|-------------------|------------------------|-------------------------|-------------|------------------------------|---------------------------|
| exp soln | container orig wt | container wt + Tc soln | wt of Tc solution added | measured pH | container wt after pH adjust | amount of acid & base add |
| 300 ppb | | | | | | |
| Tc-P* pH 3.0 | 22.1778 | 42.1807 | 20.0029 | 3.01 | 42.1807 | 0.230 (0.1M HCl) |
| Tc-P* pH 4.0 | 22.2749 | 42.2848 | 20.0099 | 4.01 | 42.2848 | 0.370 (0.01M HCl) |
| Tc-P* pH 5.0 | 22.2996 | 42.3175 | 20.0179 | 4.96 | 42.3175 | 0.150 (0.01M HCl) |
| Tc-P* pH 6.0 | 22.0208 | 42.0232 | 20.0024 | 6.02 | 42.0232 | 0.050 (0.01M HCl) |
| Tc-P* pH 7.0 | 21.9401 | 41.954 | 20.0139 | 6.82 | 41.9540 | ----- |
| 30 ppb | | | | | | |
| Tc-P* pH 3.0 | 22.03 | 42.0368 | 20.0068 | 3.01 | 42.0368 | 0.230 (0.1M HCl) |
| Tc-P* pH 4.0 | 22.0235 | 42.0300 | 20.0065 | 4.01 | 42.0300 | 0.250 (0.01M HCl) |
| Tc-P* pH 5.0 | 22.3276 | 42.3318 | 20.0042 | 4.82 | 42.3318 | 0.050 (0.01M HCl) |
| Tc-P* pH 6.0 | 22.1521 | 42.1604 | 20.0083 | 5.92 | 42.1604 | ----- |
| Tc-P* pH 7.0 | 21.9991 | 42.0158 | 20.0167 | 7.00 | 42.0158 | 0.115 (0.01M NaOH) |

| | wt vial + | | | wt vial + | |
|----------------|------------|--------|--|---------------|------------|
| sample | wt of vial | sample | | sample | wt of vial |
| 300 ppb | | | | 30 ppb | |
| Tc-P*ITca | 7.3090 | 7.8111 | | Tc-P*ITca | 7.3493 |
| Tc-P*ITcb | 7.3131 | 7.8153 | | Tc-P*ITcb | 7.3284 |
| | | | | | 7.8515 |
| | | | | | 7.8316 |

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AJ

| sample name | weight vial | wt vial + sample |
|------------------------|-------------|------------------|
| 300 ppb 8/30/99 | | |
| Tc-P* pH 3a | 7.3634 | 7.8628 |
| Tc-P* pH 3b | 7.3613 | 7.8613 |
| Tc-P* pH 4a | 7.3348 | 7.8336 |
| Tc-P* pH 4b | 7.2885 | 7.7887 |
| Tc-P* pH 5a | 7.3109 | 7.8106 |
| Tc-P* pH 5b | 7.3222 | 7.8220 |
| Tc-P* pH 6a | 7.2578 | 7.7572 |
| Tc-P* pH 6b | 7.3258 | 7.8253 |
| Tc-P* pH 7a | 7.2268 | 7.7249 |
| Tc-P* pH 7b | 7.2809 | 7.7803 |
| 30 ppb | | |
| Tc-P* pH 3a | 7.2367 | 7.7364 |
| Tc-P* pH 3b | 7.3231 | 7.825 |
| Tc-P* pH 4a | 7.2644 | 7.765 |
| Tc-P* pH 4b | 7.2824 | 7.7845 |
| Tc-P* pH 5a | 7.3096 | 7.8088 |
| Tc-P* pH 5b | 7.2725 | 7.7731 |
| Tc-P* pH 6a | 7.2841 | 7.7832 |
| Tc-P* pH 6b | 7.2649 | 7.7656 |
| Tc-P* pH 7a | 7.2855 | 7.7828 |
| Tc-P* pH 7b | 7.3405 | 7.8403 |

8 d centrifuge at 12,000 for 15 minutes
300 ppb 8/6/99

| | | |
|---------------|--------|--------|
| Tc-P* pH 3a | 7.2705 | 7.7698 |
| Tc-P* pH 3b | 7.3269 | 7.8327 |
| Tc-P* pH 4a | 7.2901 | 7.7897 |
| Tc-P* pH 4b | 7.2605 | 7.7668 |
| Tc-P* pH 5a | 7.3234 | 7.8236 |
| Tc-P* pH 5b | 7.2914 | 7.7988 |
| Tc-P* pH 6a | 7.3633 | 7.8711 |
| Tc-P* pH 6b | 7.3546 | 7.8626 |
| Tc-P* pH 7a | 7.3262 | 7.8236 |
| Tc-P* pH 7b | 7.3788 | 7.8695 |
| 30 ppb | | |
| Tc-P* pH 3a | 7.3464 | 7.8431 |
| Tc-P* pH 3b | 7.3129 | 7.8168 |
| Tc-P* pH 4a | 7.3518 | 7.8552 |
| Tc-P* pH 4b | 7.4015 | 7.9033 |
| Tc-P* pH 5a | 7.2912 | 7.7881 |
| Tc-P* pH 5b | 7.2954 | 7.7929 |
| Tc-P* pH 6a | 7.3371 | 7.8314 |
| Tc-P* pH 6b | 7.2973 | 7.7962 |
| Tc-P* pH 7a | 7.3405 | 7.8363 |
| Tc-P* pH 7b | 7.2835 | 7.7909 |

| sample name | weight vial | wt vial + sample |
|-------------------------------------|-------------|------------------|
| 3 days 8/1/99 300 ppb | | |
| Tc-P* pH 3a | 7.2726 | 7.7714 |
| Tc-P* pH 3b | 7.2269 | 7.7282 |
| Tc-P* pH 4a | 7.3548 | 7.8537 |
| Tc-P* pH 4b | 7.2624 | 7.761 |
| Tc-P* pH 5a | 7.3229 | 7.820 |
| Tc-P* pH 5b | 7.2319 | 7.7287 |
| Tc-P* pH 6a | 7.3448 | 7.8430 |
| Tc-P* pH 6b | 7.2607 | 7.7592 |
| Tc-P* pH 7a | 7.3058 | 7.8043 |
| Tc-P* pH 7b | 7.3170 | 7.8167 |
| 30 ppb | | |
| Tc-P* pH 3a | 7.3447 | 7.8459 |
| Tc-P* pH 3b | 7.3540 | 7.8555 |
| Tc-P* pH 4a | 7.2945 | 7.7949 |
| Tc-P* pH 4b | 7.3361 | 7.8371 |
| Tc-P* pH 5a | 7.3175 | 7.8187 |
| Tc-P* pH 5b | 7.3004 | 7.8005 |
| Tc-P* pH 6a | 7.3756 | 7.8752 |
| Tc-P* pH 6b | 7.3220 | 7.824 |
| Tc-P* pH 7a | 7.2981 | 7.7978 |
| Tc-P* pH 7b | 7.3053 | 7.8060 |

14 d centrifuge at 12,000 for 30 minutes
300 ppb 8/12/99

| | | |
|---------------|--------|--------|
| Tc-P* pH 3a | 7.3104 | 7.8195 |
| Tc-P* pH 3a | 7.3563 | 7.8657 |
| Tc-P* pH 3b | 7.2925 | 7.7915 |
| Tc-P* pH 4a | 7.2851 | 7.7935 |
| Tc-P* pH 4b | 7.3028 | 7.7944 |
| Tc-P* pH 5a | 7.2814 | 7.7499 |
| Tc-P* pH 5b | 7.3518 | 7.8519 |
| Tc-P* pH 6a | 7.3490 | 7.8566 |
| Tc-P* pH 6b | 7.2905 | 7.7890 |
| Tc-P* pH 7a | 7.3217 | 7.8289 |
| Tc-P* pH 7b | | |
| 30 ppb | | |
| Tc-P* pH 3a | 7.3195 | 7.8131 |
| Tc-P* pH 3b | 7.3686 | 7.8793 |
| Tc-P* pH 4a | 7.3426 | 7.8366 |
| Tc-P* pH 4b | 7.2892 | 7.8025 |
| Tc-P* pH 5a | 7.2834 | 7.7795 |
| Tc-P* pH 5b | 7.4070 | 7.9204 |
| Tc-P* pH 6a | 7.3814 | 7.871 |
| Tc-P* pH 6b | 7.3036 | 7.8151 |
| Tc-P* pH 7a | 7.3146 | 7.8211 |
| Tc-P* pH 7b | 7.3489 | 7.847 |

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03 Aug 1999 12:52 ALPHA/BETA - 1.09
Protocol #143 TC-99 CPM User

Time: 999.99
Data Mode: CPM Nuclide: MANUAL
Background Subtract: 1st Vial

| | | | | | |
|-----------|------------|----|-----|-------|-----|
| | LL | UL | LCR | 25% | BKG |
| Region A: | 0.0 - 300 | 0 | 0.0 | 19.06 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.83 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.91 | |

Quench Indicator: SIS
BETA CPM TC-99
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal

| S# | TIME | CPMA | A:25% | CPMB | B:25% | CPMC | C:25% | SIS | FLAG |
|-------------------|--------|---------|-------|---------|-------|---------|-------|--------|------|
| 1 | 999.99 | 19.06 | 1.45 | 17.83 | 1.50 | 24.91 | 1.27 | 129.31 | B |
| 2 | 0.89 | 5071.96 | 2.98 | 5018.12 | 3.00 | 5070.60 | 2.98 | 175.07 | |
| 3 | 0.87 | 5151.06 | 2.99 | 5090.21 | 3.01 | 5149.81 | 3.00 | 173.96 | |
| 4 | 0.89 | 5071.96 | 2.98 | 5002.39 | 3.00 | 5071.72 | 2.98 | 174.07 | |
| 5 | 0.97 | 4933.52 | 2.90 | 4880.10 | 2.91 | 4932.83 | 2.90 | 168.91 | |
| 6 | 0.89 | 5032.63 | 2.99 | 4991.16 | 3.01 | 5031.27 | 3.00 | 173.16 | |
| 7 | 0.91 | 4982.04 | 2.98 | 4912.94 | 3.00 | 4978.39 | 2.98 | 174.20 | |
| 8 | 0.87 | 5168.30 | 2.99 | 5104.01 | 3.01 | 5168.20 | 2.99 | 176.06 | |
| 9 | 0.87 | 5172.90 | 2.99 | 5113.20 | 3.00 | 5175.09 | 2.99 | 173.10 | |
| 10 | 0.89 | 5046.11 | 2.99 | 5000.14 | 3.00 | 5048.13 | 2.99 | 171.53 | |
| 11 | 0.88 | 5105.94 | 2.99 | 5041.26 | 3.01 | 5110.32 | 2.99 | 175.80 | |
| 1 MISSING TUBE(S) | | | | | | | | | |
| 13 | 8.72 | 496.54 | 3.10 | 492.14 | 3.11 | 496.43 | 3.12 | 175.83 | |
| 14 | 8.48 | 511.96 | 3.09 | 506.46 | 3.11 | 511.65 | 3.11 | 177.23 | |
| 15 | 8.53 | 508.73 | 3.09 | 503.15 | 3.11 | 508.86 | 3.11 | 176.12 | |
| 16 | 8.58 | 505.42 | 3.09 | 500.12 | 3.11 | 505.40 | 3.11 | 173.56 | |
| 17 | 8.45 | 514.08 | 3.09 | 508.32 | 3.11 | 512.85 | 3.11 | 172.88 | |
| 18 | 8.14 | 533.40 | 3.09 | 528.24 | 3.10 | 533.08 | 3.11 | 176.00 | |
| 19 | 8.43 | 512.85 | 3.10 | 509.45 | 3.11 | 513.05 | 3.11 | 171.96 | |
| 20 | 8.61 | 503.94 | 3.09 | 498.54 | 3.11 | 503.78 | 3.11 | 169.62 | |
| 21 | 8.31 | 522.82 | 3.09 | 517.67 | 3.10 | 522.39 | 3.11 | 173.83 | |
| 22 | 8.63 | 503.42 | 3.09 | 497.23 | 3.11 | 502.67 | 3.11 | 170.25 | |

14 days filtered through **0.2 micron**

| | | |
|----------------|--------|--------|
| 300 ppb | | |
| Tc-P* pH 3a | 7.3236 | 8.2438 |
| Tc-P* pH 3b | 7.4016 | 8.2187 |
| Tc-P* pH 4a | 7.3618 | 8.2800 |
| Tc-P* pH 4b | 7.2741 | 8.0824 |
| Tc-P* pH 5a | 7.2962 | 8.1477 |
| Tc-P* pH 5b | 7.2941 | 8.1044 |

Tc-P*
300 ppb
3 dayTc-P*
300 ppb
3 day

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 05-Aug-1999 11:44
 C14 Eff (0-156 keV) = 96.72 %
 C14 CHI SQUARE IPA DATA PROCESSED - 05-Aug-1999 11:55
 C14 Chi Square = 19.32
 H3 IPA DATA PROCESSED - 05-Aug-1999 11:56
 H3 Eff (0-18.6 keV) = 65.26 %
 H3 CHI SQUARE IPA DATA PROCESSED - 05-Aug-1999 12:07
 H3 Chi Square = 25.37
 BKG IPA DATA PROCESSED - 05-Aug-1999 13:07
 Bkg (0-18.6 keV) = 15.83 cpm
 Bkg (0-156 keV) = 22.58 cpm
 C14 E²/B (1-156 keV) = 542.93
 H3 E²/B (1-18.6 keV) = 267.42

06 Aug 1999 05:57

ALPHA/BETA - 1.09

Protocol #:43

TC-99 CPM

User

Time: 999.99

Data Mode: CPM

Nuclide: MANUAL

Background Subtract: 1st Vial

| | LL | UL | LCR | 25% | BKG |
|-----------|------------|----|-----|-------|-----|
| Region A: | 0.0 - 300 | 0 | 0.0 | 19.10 | |
| Region B: | 2.0 - 300 | 0 | 3.0 | 17.86 | |
| Region C: | 0.0 - 2000 | 0 | 0.0 | 24.92 | |

Quench Indicator: SIS

BETA CPM TC-99

Coincidence Time(ns): 18

Delay Before Burst(ns): Normal

| S# | TIME | CPMA A:25% | CPMB B:25% | CPMC C:25% | SIS FLAG | | | | |
|----|-----------------|------------|------------|------------|----------|--------|------|--------|---|
| 1 | 999.99 | 19.10 | 1.45 | 17.86 | 1.50 | 24.92 | 1.27 | 126.56 | B |
| 2 | 8.42 | 515.58 | 3.09 | 510.52 | 3.10 | 516.64 | 3.11 | 177.72 | |
| 3 | 8.38 | 518.73 | 3.09 | 512.93 | 3.10 | 519.11 | 3.10 | 174.80 | |
| 4 | 8.86 | 490.04 | 3.09 | 484.40 | 3.11 | 489.41 | 3.11 | 174.29 | |
| 5 | 8.48 | 512.62 | 3.09 | 506.43 | 3.11 | 512.22 | 3.11 | 173.69 | |
| 6 | 8.14 | 534.34 | 3.09 | 528.94 | 3.10 | 534.90 | 3.10 | 175.29 | |
| 7 | 8.07 | 539.51 | 3.08 | 532.94 | 3.10 | 538.77 | 3.10 | 172.26 | |
| 1 | MISSING TUBE(S) | | | | | | | | |
| 8 | 8.12 | 535.33 | 3.09 | 529.55 | 3.10 | 535.30 | 3.10 | 177.82 | |
| 10 | 8.36 | 518.22 | 3.09 | 513.84 | 3.10 | 518.02 | 3.11 | 175.40 | |
| 11 | 8.03 | 540.68 | 3.09 | 535.69 | 3.10 | 540.58 | 3.11 | 172.89 | |
| 12 | 8.05 | 541.02 | 3.08 | 534.44 | 3.10 | 541.91 | 3.10 | 174.98 | |
| 13 | 8.24 | 527.87 | 3.09 | 521.94 | 3.10 | 526.77 | 3.11 | 176.02 | |
| 14 | 8.17 | 532.92 | 3.09 | 526.08 | 3.10 | 531.75 | 3.11 | 174.44 | |

TC-K*
17 daysTC-K*
17 days

This Notebook means
 to copy with QAP-001.
 E.C. Pen
 1/13/2000