Geochemistry
Research laboratory
Notebook

Vol. GC-05

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Vol GC-08

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11/25/92 JP

URANIUM SORPTION EXPERIMENT B-8005
Kd vs pH: Equilibrium with atmospheric pCO_2; Initial ΣU=5 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: Nov 25, 1992

OBJECTIVE: to investigate the importance of surface area on uranium sorption as a function of solution pH

EQUIPMENT:
- Gyratory shaker
- EG&G alpha-spectrometer
- ORION ph/mV/ISE/ C meter
- Combination ph electrode
- Automatic termperature compensator probe
- Analytical balance

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
1	125 ml PP bottle (to contain B-8005*IU)
39	125 ml PP bottles (to contain experimental mixtures and control solutions)
78	50 ml centrifuge tubes (to contain samples for U analysis)
1	100 ml volumetric pipet (to pipet 5 ppb U solution into PP bottles)
1	10 ml volumetric pipet (to prepare 5 ppb U solution)
1	5 ml volumetric pipet (to pipet 0.02M HNO_3 into 50 ml centrifuge tubes)
1	4000 ml plastic bottle (for preparation of 5 ppb U solution)
3	glass droppers (for adjusting pH by addition of HNO_3)
	weighing paper
1	Eppendorf pipet (for taking 5 ml samples)
	8005 Alpha Alumina (surface area 2.09 m^2/g; from NIST)
	reagent grade $NaHCO_3$ (22K)
	500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike
4L	0.1 m $NaNO_3$ stock solution
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3
	ultrapure water

PREPARATION:

0900 hrs JP

1. Preclean:

40	125 ml PP bottles
78	50 ml centrifuge tubes
1	100 ml volumetric pipet
1	10 ml volumetric pipet
1	5 ml volumetric pipet
1	4000 ml plastic bottle
3	glass droppers

2. Prepare:

1400 hrs JP

	500 ppb U stock solution from 50 ppm ^{233}U commercial spike
4L	0.1 m $NaNO_3$ stock solution (lot No. 7808 KCCL)
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3

PROCEDURE:

Solution B-8005 (1 bottle for each pH value)

- Initial ΣU = 5 ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 100 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8005 alpha alumina to use = 0.200±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

1530 hrs JP

a) In a pre-cleaned 4L plastic bottle, prepare 4000 g of 5 ppb U solution by diluting 40 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 4000 g by carefully taring 0.1 m $NaNO_3$ solution into a plastic bottle on a Mettler 4600 balance.

11/30/92
1345 hrs JP

b) Into each of 29 125 ml PP bottles labeled B-8005*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 100 g of the 5 ppb U solution.

1550 hrs JP

Into each of 10 125 ml PP bottles labeled B-8005-C*pH*i*a (or b) [where *i* is 2, 4, 6, 8, or 9.5, representing the approximate initial pH of the solution], tare 100 g of the 5 ppb U solution. These are control solutions to determine uranium loss to the container walls as a function of pH.

Transfer the remaining solution into a 125 ml PP bottle labeled B-8005*IU. Take two 5 ml samples from B-8005*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8005-IU*a (or b)] and pre-weighed 50 ml centrifuge tubes containing 5 g of a 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

12/2/92 JP
0830 hrs

Sample	Wt. of tube	Wt of tube + 5g 0.02M HNO_3	Wt of tube + 5g 0.02M HNO_3 + 5 ml B-8005*IU
B-8005*IU*a	10.73g	15.73g	20.75g
B-8005*IU*b	10.73g	15.74g	20.75g

0900 hrs

c) 1. For each solution B-8005*pH*i* and B-8005-C*pH*i*, where *i*≤5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). The automatic temperature compensator (ATC) probe should be immersed in water in a separate container. Adjust the pH to the value *i* of each solution by adding ~~dropwise with a glass droppper~~ with Eppendorf pipetors HNO_3 solution, the concentration and approximate amount of which is given in Table B-III-1. Swirl the solutions by hand. Record ~~the number of drops~~ amount and concentration of solution added. Also record the attained pH. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically, together with the bottles prepared in step 2 below.

12/2/92 JP
8005 JP 12/2/92
12/2/92

1500 hrs.

2. For each solution B-8005*pH*i* and B-8005-C*pH*i*, where *i*>5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). Tare onto weighing paper reagent grade $NaHCO_3$ in the amounts listed in Table B-III-1 and transfer into the respective PP bottles. Swirl the solutions by hand. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically until the pH becomes constant and in equilibrium with atmospheric CO_2(g). This equilibrium process may take at least ten days.

8005 JP 12/2/92

Table B-8005

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8005*pH2.00	5.16	2.00	1.21	1.00	2.00
B-8005*pH2.25	5.13	2.25	0.67	1.00	2.25
B-8005*pH2.50	5.07	2.50	0.36	1.00	2.49
B-8005*pH2.75	5.20	2.75	0.23	1.00	2.75
B-8005*pH3.00	5.03	3.00	1.10	0.10	3.00
B-8005*pH3.25	5.07	3.25	0.64	0.10	3.25
B-8005*pH3.50	5.19	3.50	0.40	0.10	3.50
B-8005*pH3.75	5.02	3.75	0.21	0.10	3.74
B-8005*pH4.00	5.09	4.00	0.12	0.10	3.96
B-8005*pH4.25	5.19	4.25	0.66	0.01	4.24
B-8005*pH4.50	5.24	4.50	0.36	0.01	4.50
B-8005*pH4.75	5.19	4.75	0.20	0.01	4.68
B-8005*pH5.00	5.25	5.00	0.06	0.01	5.00
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8005*pH5.25	5.13	5.25	0.000022		
B-8005*pH5.50	5.16	5.50	0.000053		
B-8005*pH5.75	5.16	5.75	0.000086		
B-8005*pH6.00	5.12	6.00	0.000118		
B-8005*pH6.25	5.20	6.25	0.000169		
B-8005*pH6.50	5.18	6.50	0.00025		
B-8005*pH6.75	5.21	6.75	0.0004		
B-8005*pH7.00	5.19	7.00	0.00066		
B-8005*pH7.25	5.14	7.25	0.001135		
B-8005*pH7.50	5.13	7.50	0.001964		
B-8005*pH7.75	5.06	7.75	0.00346		
B-8005*pH8.00	5.09	8.00	0.00619		
B-8005*pH8.25	5.03	8.25	0.01105		
B-8005*pH8.50	5.16	8.50	0.020275		
B-8005*pH8.75	5.21	8.75	0.03774		
B-8005*pH9.00	5.20	9.00	0.07274		

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8005-C*pH2*a	5.12	2.00	1.20	1.00	2.00
B-8005-C*pH2*b	5.11	2.00	1.21	1.00	2.00
B-8005-C*pH4*a	5.12	4.00	0.12	0.10	4.00
B-8005-C*pH4*b	5.15	4.00	0.12	0.10	3.98
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8005-C*pH6*a	5.19	6.00	0.000119		
B-8005-C*pH6*b	5.23	6.00	0.000116		
B-8005-C*pH8*a	5.19	8.00	0.00618		
B-8005-C*pH8*b	5.21	8.00	0.00615		
B-8005-C*pH9.5*a	5.19	9.50	0.33511		
B-8005-C*pH9.5*b	5.20	9.50	0.3349		

12/17/92
1500hrs
DD

12/30/92
0900hrs
DD

d) Tare 0.200±0.001 g of 8005 alpha alumina onto weighing paper, and carefully transfer into each of the B-8005*pH*i* (**not** the B-8005-C*pH*i*) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker. Filtered thru Dynagard 0.2µm Filter (lot 75-68A)

e) After equilibrium is reached (at least 10 days), take two 5 ml samples from each bottle B-8005*pH*i* and B-8005-C*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8005*pH*i*a (or b)] and pre-weighed 50 ml centrifuge tubes containing ~5 g of 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

Measure and record the pH and temperature of solutions B-8005*pH*i* and B-8005-C*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

f) Analyze the U concentration by alpha-spectrometry.

B-8005 data Table

Sample	Weight of tube	Weight of tube plus ~5g 0.02M HNO3	Weight of tube plus ~5g 0.02M HNO3 plus 5ml of sample
B-8005*pH2.00*a	10.70g	15.72g	20.79g
B-8005*pH2.00*b	10.70g	15.70	20.73g
B-8005*pH2.25*a	10.76g	15.74	20.78
B-8005*pH2.25*b	10.74	15.73	20.77
B-8005*pH2.50*a	10.75	15.74	20.78
B-8005*pH2.50*b	10.73	15.72	20.75
B-8005*pH2.75*a	10.73	15.73	20.76
B-8005*pH2.75*b	10.83	15.82	20.83
B-8005*pH3.00*a	10.64	15.64	20.65
B-8005*pH3.00*b	10.77	15.76	20.76
B-8005*pH3.25*a	10.71	15.71	20.74
B-8005*pH3.25*b	10.76	15.76	20.78
B-8005*pH3.50*a	10.73	15.72	20.74
B-8005*pH3.50*b	10.74	15.73	20.75
B-8005*pH3.75*a	10.73	15.73	20.77
B-8005*pH3.75*b	10.74	15.73	20.76
B-8005*pH4.00*a	10.81	15.82	20.85
B-8005*pH4.00*b	10.93	15.91	20.92
B-8005*pH4.25*a	10.88	15.84	20.87
B-8005*pH4.25*b	10.76	15.75	20.77
B-8005*pH4.50*a	10.70	15.70	20.73
B-8005*pH4.50*b	10.74	15.74	20.78
B-8005*pH4.75*a	10.87	15.86	20.88
B-8005*pH4.75*b	10.64	15.63	20.65
B-8005*pH5.00*a	10.76	15.75	20.80
B-8005*pH5.00*b	10.75	15.73	20.77
B-8005*pH5.25*a	10.72	15.73	20.76
B-8005*pH5.25*b	10.82	15.81	20.85
B-8005*pH5.50*a	10.65	15.63	20.63
B-8005*pH5.50*b	10.74	15.72	20.73
B-8005*pH5.75*a	10.73	15.71	20.74
B-8005*pH5.75*b	10.72	15.71	20.74
B-8005*pH6.00*a	10.74	15.73	20.75
B-8005*pH6.00*b	10.72	15.71	20.73
B-8005*pH6.25*a	10.76	15.75	20.76
B-8005*pH6.25*b	10.73	15.70	20.72
B-8005*pH6.50*a	10.74	15.71	20.70
B-8005*pH6.50*b	10.92	15.90	20.91
B-8005*pH6.75*a	10.82	15.79	20.79
B-8005*pH6.75*b	10.73	15.75	20.77
B-8005*pH7.00*a	10.93	15.92	20.93
B-8005*pH7.00*b	10.74	15.72	20.73

B-8005 data Table

B-8005*pH7.25*a	10.70	15.68	20.70
B-8005*pH7.25*b	10.82	15.80	20.82
B-8005*pH7.50*a	10.72	15.71	20.72
B-8005*pH7.50*b	10.74	15.73	20.76
B-8005*pH7.75*a	10.75	15.74	20.77
B-8005*pH7.75*b	10.74	15.72	20.75
B-8005*pH8.00*a	10.70	15.68	20.70
B-8005*pH8.00*b	10.93	15.92	20.93
B-8005*pH8.25*a	10.75	15.74	20.76
B-8005*pH8.25*b	10.74	15.72	20.75
B-8005*pH8.50*a	10.64	15.65	20.66
B-8005*pH8.50*b	10.74	15.73	20.75
B-8005*pH8.75*a	10.71	15.72	20.73
B-8005*pH8.75*b	10.70	15.70	20.73
B-8005*pH9.00*a	10.73	15.73	20.74
B-8005*pH9.00*b	10.73	15.71	20.73
B-8005-C*pH2*a1	10.82	15.83	20.86
B-8005-C*pH2*a2	10.73	15.73	20.77
B-8005-C*pH2*b1	10.74	15.74	20.75
B-8005-C*pH2*b2	10.77	15.76	20.80
B-8005-C*pH4*a1	10.93	15.95	20.98
B-8005-C*pH4*a2	10.76	15.77	20.82
B-8005-C*pH4*b1	10.70	15.71	20.72
B-8005-C*pH4*b2	10.74	15.73	20.77
B-8005-C*pH6*a1	10.77	15.77	20.78
B-8005-C*pH6*a2	10.77	15.77	20.79
B-8005-C*pH6*b1	10.77	15.77	20.80
B-8005-C*pH6*b2	10.74	15.72	20.76
B-8005-C*pH8*a1	10.73	15.73	20.76
B-8005-C*pH8*a2	10.72	15.72	20.76
B-8005-C*pH8*b1	10.72	15.73	20.74
B-8005-C*pH8*b2	10.73	15.73	20.76
B-8005-C*pH9.5*a1	10.72	15.70	20.72
B-8005-C*pH9.5*a2	10.88	15.87	20.90
B-8005-C*pH9.5*b1	10.71	15.69	20.74
B-8005-C*pH9.5*b2	10.74	15.74	20.78

B-8005 pH Table

Sample	Date	Date	Date	Date	Date
	12/15/92	12/17/92		12/30/92	
	pH	pH	pH	pH	pH
B-8005*pH2.00	2.00	1.93		1.93 / 22.8°C	
B-8005*pH2.25	2.23	2.18		2.19 / 22.9°C	
B-8005*pH2.50	2.48	2.45		2.47 / 22.9°C	
B-8005*pH2.75	2.64	2.67		2.67 / 22.9°C	
B-8005*pH3.00	3.02	2.89		3.04 / 22.8°C	
B-8005*pH3.25	3.25	3.23		3.35 / 22.9°C	
B-8005*pH3.50	3.47	3.44		3.64 / 22.8°C	
B-8005*pH3.75	3.72	3.67		4.13 / 23.0°C	
B-8005*pH4.00	3.96	3.92		4.74 / 23.0°C	
B-8005*pH4.25	4.22	4.16		5.77 / 23.0°C	
B-8005*pH4.50	4.46	4.42		6.49 / 23.0°C	
B-8005*pH4.75	4.64	4.61		6.65 / 23.0°C	
B-8005*pH5.00	4.95	4.88		6.76 / 23.1°C	
B-8005*pH5.25	5.24	5.23		6.80 / 22.9°C	
B-8005*pH5.50	5.41	5.45		6.85 / 23.1°C	
B-8005*pH5.75	5.65	5.66		6.90 / 23.1°C	
B-8005*pH6.00	5.75	5.90		6.90 / 23.1°C	
B-8005*pH6.25	6.16	6.18		6.91 / 23.1°C	
B-8005*pH6.50	6.32	6.30		6.94 / 23.2°C	
B-8005*pH6.75	6.63	6.64	7.02	6.9 / 23.2°C	
B-8005*pH7.00	6.85	6.87		7.14 / 23.2°C	
B-8005*pH7.25	7.09	7.19		7.28 / 23.2°C	
B-8005*pH7.50	7.35	7.40		7.46 / 23.3°C	
B-8005*pH7.75	7.58	7.64		7.61 / 23.3°C	
B-8005*pH8.00	7.91	7.95		7.95 / 23.2°C	
B-8005*pH8.25	8.14	8.25		8.19 / 23.3°C	
B-8005*pH8.50	8.46	8.47		8.46 / 23.3°C	
B-8005*pH8.75	8.73	8.74		8.79 / 23.3°C	
B-8005*pH9.00	8.97	9.00		8.97 / 23.3°C	
B-8005-C*pH2*a	2.01	1.97		1.93 / 22.8°C	
B-8005-C*pH2*b	2.01	1.93		1.92 / 22.8°C	
B-8005-C*pH4*a	4.00	3.96		3.90 / 22.8°C	
B-8005-C*pH4*b	3.96	3.92		3.87 / 22.8°C	
B-8005-C*pH6*a	5.98	5.95		5.91 / 23.0°C	
B-8005-C*pH6*b	5.92	5.72		5.71 / 23.0°C	
B-8005-C*pH8*a	7.89	7.96		7.93 / 23.2°C	
B-8005-C*pH8*b	7.89	7.94		7.96 / 23.2°C	
B-8005-C*pH9.5*a	9.43	9.47		9.45 / 23.3°C	
B-8005-C*pH9.5*b	9.43	9.48		9.46 / 23.3°C	

12/4/92

URANIUM SORPTION EXPERIMENT B-8006
Kd vs pH: Equilibrium with atmospheric pCO_2; Initial ΣU=5 ppb

WRITTEN BY: R.T. PABALAN — DATE WRITTEN: Aug 5, 1992
REVISION NO.: 1 — DATE REVISED: Dec 3, 1992
REVISED BY: J.D. PRIKRYL

OBJECTIVE: to investigate the importance of surface area on uranium sorption as a function of solution pH

EQUIPMENT:
- Gyratory shaker
- EG&G alpha-spectrometer
- ORION ph/mV/ISE/ C meter
- Combination ph electrode
- Automatic termperature compensator probe
- Analytical balance

SUPPLIES:
- pH buffers (pH= 2,4,7,9,10)
- 1 — 125 ml PP bottle (to contain B-8006*IU)
- 39 — 125 ml PP bottles (to contain experimental mixtures and control solutions)
- 78 — 50 ml centrifuge tubes (to contain samples for U analysis)
- 1 — 100 ml volumetric pipet (to pipet 5 ppb U solution into PP bottles)
- 1 — 10 ml volumetric pipet (to prepare 5 ppb U solution)
- 1 — 5 ml volumetric pipet (to pipet 0.02M HNO_3 into 50 ml centrifuge tubes)
- 1 — 4000 ml plastic bottle (for preparation of 5 ppb U solution)
- 3 — glass droppers (for adjusting pH by addition of HNO_3)
- weighing paper
- 1 — Eppendorf pipet (for taking 5 ml samples)
- 8006 Alpha Alumina (surface area 0.229 m^2/g; from NIST)
- reagent grade $NaHCO_3$ (22K)
- 500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike
- 4L — 0.1 m $NaNO_3$ stock solution
- 1L — stock solution of 1.0 m HNO_3
- 1L — stock solution of 0.1 m HNO_3
- 1L — stock solution of 0.02 m HNO_3
- 1L — stock solution of 0.01 m HNO_3
- ultrapure water

PREPARATION:

1. Preclean:
   - 40 — 125 ml PP bottles
   - 78 — 50 ml centrifuge tubes
   - 1 — 100 ml volumetric pipet
   - 1 — 10 ml volumetric pipet
   - 1 — 5 ml volumetric pipet
   - 1 — 4000 ml plastic bottle
   - 3 — glass droppers
2. Prepare:
   - 500 ppb U stock solution from 50 ppm ^{233}U commercial spike
   - 4L — 0.1 m $NaNO_3$ stock solution (lot 7808 MDJE)
   - 1L — stock solution of 1.0 m HNO_3
   - 1L — stock solution of 0.1 m HNO_3
   - 1L — stock solution of 0.02 m HNO_3
   - 1L — stock solution of 0.01 m HNO_3

1100 hrs

PROCEDURE:

Solution B-8006 (1 bottle for each pH value)

- Initial ΣU = 5 ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 100 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8006 alpha alumina to use = 0.200±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

1330 hrs
gp

12/7/92
0945 hrs
gp

a) In a pre-cleaned 4L plastic bottle, prepare 4000 g of 5 ppb U solution by diluting 40 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 4000 g by carefully taring 0.1 m $NaNO_3$ solution into a plastic bottle on a Mettler 4600 balance.

b) Into each of 29 125 ml PP bottles labeled B-8006*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 100 g of the 5 ppb U solution.

Into each of 10 125 ml PP bottles labeled B-8006-C*pH*i*a (or b) [where *i* is 2, 4, 6, 8, or 9.5, representing the approximate initial pH of the solution], tare 100 g of the 5 ppb U solution. These are control solutions to determine uranium loss to the container walls as a function of pH.

1300 hrs

Transfer the remaining solution into a 125 ml PP bottle labeled B-8006*IU. Take two 5 ml samples from B-8006*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8006-IU*a (or b)] and pre-weighed 50 ml centrifuge tubes containing 5 g of a 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

Sample	Wt. of tube	Wt of tube + 5g 0.02M HNO_3	Wt of tube + 5g 0.02M HNO_3 + 5 ml B-8006*IU
B-8006-IU*a	10.77 g	15.77 g	20.79 g
B-8006-IU*b	10.77 g	15.76 g	20.81 g

12/8/92
0815 hrs
gp

c) 1. For each solution B-8006*pH*i* and B-8006-C*pH*i*, where *i*≤5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). The automatic temperature compensator (ATC) probe should be immersed in water in a separate container. Adjust the pH to the value *i* of each solution by adding with Eppendorf pipettes HNO_3 solution, the concentration and approximate amount of which is given in Table B-8006. Swirl the solutions by hand. Record the amount and concentration of solution added. Also record the attained pH. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically, together with the bottles prepared in step 2 below.

12/23/92
gp
0825 hrs

1/6/93
0930 hrs
gp

2. For each solution B-8006*pH*i* and B-8006-C*pH*i*, where *i*>5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). Tare onto weighing paper reagent grade $NaHCO_3$ in the amounts listed in Table B-8006 and transfer into the respective PP bottles. Swirl the solutions by hand. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically until the pH becomes constant and in equilibrium with atmospheric CO_2(g). This equilibrium process may take at least ten days.

d) Tare 0.200±0.001 g of 8006 alpha alumina onto weighing paper, and carefully transfer into each of the B-8006*pH*i* (**not** the B-8006-C*pH*i*) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker. Filtered than Dynaguard 0.2 um filter

e) After equilibrium is reached (at least 10 days), take two 5 ml samples from each bottle B-8006*pH*i* and B-8006-C*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8006*pH*i*a (or b)] and pre-weighed 50 ml centrifuge tubes containing 5 g of 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

Measure and record the pH and temperature of solutions B-8006*pH*i* and B-8006-C*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

f) Analyze the U concentration by alpha-spectrometry.

Table B-8006

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8006*pH2.00	5.14	2.00	1.22	1.00	2.00
B-8006*pH2.25	5.09	2.25	0.68	1.00	2.26
B-8006*pH2.50	5.07	2.50	0.36	1.00	2.50
B-8006*pH2.75	5.15	2.75	0.21	1.00	2.74
B-8006*pH3.00	5.12	3.00	1.14	0.10	3.01
B-8006*pH3.25	5.13	3.25	0.64	0.10	3.25
B-8006*pH3.50	5.18	3.50	0.40	0.10	3.46
B-8006*pH3.75	5.14	3.75	0.20	0.10	3.75
B-8006*pH4.00	5.10	4.00	0.11	0.10	4.00
B-8006*pH4.25	5.15	4.25	0.56	0.01	4.25
B-8006*pH4.50	5.13	4.50	0.28	0.01	4.49
B-8006*pH4.75	5.16	4.75	0.14	0.01	4.74
B-8006*pH5.00	5.08	5.00	0.02	0.01	5.00
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8006*pH5.25	5.10	5.25	0.000021		
B-8006*pH5.50	5.14	5.50	0.000058		
B-8006*pH5.75	5.17	5.75	0.000085		
B-8006*pH6.00	5.12	6.00	0.000121		
B-8006*pH6.25	5.09	6.25	0.000165		
B-8006*pH6.50	5.17	6.50	0.00027		
B-8006*pH6.75	5.12	6.75	0.00042		
B-8006*pH7.00	5.19	7.00	0.00066		
B-8006*pH7.25	5.15	7.25	0.001139		
B-8006*pH7.50	5.14	7.50	0.00203		
B-8006*pH7.75	5.13	7.75	0.00352		
B-8006*pH8.00	5.11	8.00	0.00615		
B-8006*pH8.25	5.15	8.25	0.01172		
B-8006*pH8.50	5.16	8.50	0.02057		
B-8006*pH8.75	5.13	8.75	0.03795		
B-8006*pH9.00	5.16	9.00	0.07294		

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8006-C*pH2*a	5.17	2.00	1.24	1.00	2.01
B-8006-C*pH2*b	5.02	2.00	1.20	1.00	1.99
B-8006-C*pH4*a	5.14	4.00	0.12	0.10	3.98
B-8006-C*pH4*b	5.11	4.00	0.12	0.10	3.98
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8006-C*pH6*a	5.17	6.00	0.00012		
B-8006-C*pH6*b	5.13	6.00	0.000118		
B-8006-C*pH8*a	5.18	8.00	0.0062		
B-8006-C*pH8*b	5.11	8.00	0.00623		
B-8006-C*pH9.5*a	5.09	9.50	0.3349		
B-8006-C*pH9.5*b	5.15	9.50	0.3353		

B-8006 pH Table

Sample	1600 hrs JP Date	1015 hrs JP Date	0800 hrs JP Date	1400 hrs JP Date	Date
	12/15/92	12/21/92	12/23/92	1/6/93	
	pH	pH	pH	pH	pH
B-8006*pH2.00	1.96	1.95		1.93/18.5°C	
B-8006*pH2.25	2.21	2.13		2.18/18.5°C	
B-8006*pH2.50	2.45	2.46		2.46/18.5°C	
B-8006*pH2.75	2.70	2.72		2.69/18.5°C	
B-8006*pH3.00	2.98	2.96		2.98/18.5°C	
B-8006*pH3.25	3.24	3.22		3.25/18.5°C	
B-8006*pH3.50	3.44	3.41		3.49/18.5°C	
B-8006*pH3.75	3.74	3.71		3.89/18.5°C	
B-8006*pH4.00	3.97	3.96		4.32/18.7°C	
B-8006*pH4.25	4.23	4.23		5.49/18.7°C	
B-8006*pH4.50	4.51	4.47		6.37/18.7°C	
B-8006*pH4.75	4.69	4.68		6.55/18.7°C	
B-8006*pH5.00	4.96	4.98		6.63/18.7°C	
B-8006*pH5.25	4.60	4.55	5.05	6.72/18.8°C	
B-8006*pH5.50	3.93	3.88	5.33	6.93/18.8°C	
B-8006*pH5.75	~~4.40~~5.57	5.51		6.73/18.8°C	
B-8006*pH6.00	5.94	~~5.90~~		6.78/18.8°C	
B-8006*pH6.25	5.65	5.60	6.10	6.81/18.9°C	
B-8006*pH6.50	4.57	4.54	6.37	7.01/18.9°C	
B-8006*pH6.75	4.40	4.38	6.66	7.07/18.9°C	
B-8006*pH7.00	6.72	6.76		7.03/19.0°C	
B-8006*pH7.25	7.07	7.02		7.22/19.0°C	
B-8006*pH7.50	7.38	7.34		7.42/19.0°C	
B-8006*pH7.75	7.59	7.57		7.61/19.0°C	
B-8006*pH8.00	7.83	7.85		7.84/19.0°C	
B-8006*pH8.25	7.99	7.96		7.96/19.0°C	
B-8006*pH8.50	8.41	8.42		8.41/19.0°C	
B-8006*pH8.75	8.64	8.69		8.65/19.0°C	
B-8006*pH9.00	8.91	8.95		8.93/19.0°C	
B-8006-C*pH2*a	1.96	1.92		1.95/18.6°C	
B-8006-C*pH2*b	1.97	1.93		1.94/18.6°C	
B-8006-C*pH4*a	3.94	3.94		3.93/18.6°C	
B-8006-C*pH4*b	3.91	3.92		3.90/18.5°C	
B-8006-C*pH6*a	5.95	5.93		5.99/18.7°C	
B-8006-C*pH6*b	5.94	5.88		5.85/18.7°C	
B-8006-C*pH8*a	7.85	7.85		7.89/18.9°C	
B-8006-C*pH8*b	7.86	7.85		7.91/18.9°C	
B-8006-C*pH9.5*a	9.31	9.42		9.45/18.9°C	
B-8006-C*pH9.5*b	9.33	9.43		9.49/18.9°C	

B-8006 data Table

Sample	Weight of tube (g)	Weight of tube plus 5g 0.02M HNO3 (g)	Weight of tube plus 5g 0.02M HNO3 plus 5ml of sample (g)
B-8006*pH2.00*a	10.65	15.68	20.77
B-8006*pH2.00*b	10.76	15.78	20.84
B-8006*pH2.25*a	10.74	15.75	20.80
B-8006*pH2.25*b	10.76	15.77	20.82
B-8006*pH2.50*a	10.77	15.79	20.82
B-8006*pH2.50*b	10.73	15.73	20.78
B-8006*pH2.75*a	10.74	15.75	20.79
B-8006*pH2.75*b	10.65	15.64	20.69
B-8006*pH3.00*a	10.65	15.65	20.67
B-8006*pH3.00*b	10.73	15.73	20.76
B-8006*pH3.25*a	10.73	15.72	20.76
B-8006*pH3.25*b	10.83	15.82	20.85
B-8006*pH3.50*a	10.74	15.73	20.75
B-8006*pH3.50*b	10.74	15.73	20.77
B-8006*pH3.75*a	10.71	15.71	20.73
B-8006*pH3.75*b	10.93	15.92	20.96
B-8006*pH4.00*a	10.76	15.75	20.78
B-8006*pH4.00*b	10.87	15.86	20.89
B-8006*pH4.25*a	10.71	15.70	20.73
B-8006*pH4.25*b	10.93	15.92	20.96
B-8006*pH4.50*a	10.74	15.73	20.75
B-8006*pH4.50*b	10.74	15.73	20.76
B-8006*pH4.75*a	10.73	15.73	20.73
B-8006*pH4.75*b	10.79	15.77	20.81
B-8006*pH5.00*a	10.70	15.69	20.70
B-8006*pH5.00*b	10.83	15.80	20.83
B-8006*pH5.25*a	10.73	15.74	20.75
B-8006*pH5.25*b	10.92	15.93	20.96
B-8006*pH5.50*a	10.93	15.92	20.92
B-8006*pH5.50*b	10.72	15.70	20.71
B-8006*pH5.75*a	10.72	15.69	20.69
B-8006*pH5.75*b	10.87	15.86	20.87
B-8006*pH6.00*a	10.73	15.69	20.71
B-8006*pH6.00*b	10.73	15.70	20.72
B-8006*pH6.25*a	10.70	15.66	20.66
B-8006*pH6.25*b	10.70	15.68	20.70
B-8006*pH6.50*a	10.64	15.62	20.62
B-8006*pH6.50*b	10.74	15.70	20.71
B-8006*pH6.75*a	10.87	15.86	20.87
B-8006*pH6.75*b	10.64	15.63	20.65
B-8006*pH7.00*a	10.73	15.72	20.74
B-8006*pH7.00*b	10.73	15.71	20.73

B-8006 data Table

B-8006*pH7.25*a	10.73	15.72	20.70
B-8006*pH7.25*b	10.74	15.71	20.74
B-8006*pH7.50*a	10.71	15.70	20.70
B-8006*pH7.50*b	10.74	15.73	20.74
B-8006*pH7.75*a	10.71	15.71	20.72
B-8006*pH7.75*b	10.74	15.70	20.72
B-8006*pH8.00*a	10.74	15.72	20.75
B-8006*pH8.00*b	10.65	15.63	20.66
B-8006*pH8.25*a	10.73	15.70	20.71
B-8006*pH8.25*b	10.87	15.84	20.87
B-8006*pH8.50*a	10.64	15.63	20.65
B-8006*pH8.50*b	10.74	15.72	20.74
B-8006*pH8.75*a	10.73	15.72	20.71
B-8006*pH8.75*b	10.74	15.72	20.73
B-8006*pH9.00*a	10.72	15.71	20.72
B-8006*pH9.00*b	10.86	15.85	20.88
B-8006-C*pH2*a1	10.73	15.73	20.77
B-8006-C*pH2*a2	10.73	15.74	20.78
B-8006-C*pH2*b1	10.73	15.72	20.75
B-8006-C*pH2*b2	10.70	15.70	20.74
B-8006-C*pH4*a1	10.70	15.70	20.71
B-8006-C*pH4*a2	10.82	15.82	20.84
B-8006-C*pH4*b1	10.93	15.92	20.95
B-8006-C*pH4*b2	10.77	15.76	20.78
B-8006-C*pH6*a1	10.69	15.68	20.71
B-8006-C*pH6*a2	10.77	15.76	20.79
B-8006-C*pH6*b1	10.65	15.64	20.68
B-8006-C*pH6*b2	10.73	15.73	20.75
B-8006-C*pH8*a1	10.73	15.73	20.77
B-8006-C*pH8*a2	10.65	15.64	20.68
B-8006-C*pH8*b1	10.76	15.75	20.78
B-8006-C*pH8*b2	10.73	15.72	20.74
B-8006-C*pH9.5*a1	10.88	15.87	20.88
B-8006-C*pH9.5*a2	10.93	15.92	20.94
B-8006-C*pH9.5*b1	10.74	15.72	20.75
B-8006-C*pH9.5*b2	10.94	15.93	20.96

12/8/92

URANIUM SORPTION EXPERIMENT B-8007
Kd vs pH: Equilibrium with atmospheric pCO_2; Initial ΣU=5 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: Dec 3, 1992

OBJECTIVE: to investigate the importance of surface area on uranium sorption as a function of solution pH

EQUIPMENT:
- Gyratory shaker
- EG&G alpha-spectrometer
- ORION ph/mV/ISE/ C meter
- Combination ph electrode
- Automatic termperature compensator probe
- Analytical balance

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
1	125 ml PP bottle (to contain B-8007*IU)
39	125 ml PP bottles (to contain experimental mixtures and control solutions)
78	50 ml centrifuge tubes (to contain samples for U analysis)
1	100 ml volumetric pipet (to pipet 5 ppb U solution into PP bottles)
1	10 ml volumetric pipet (to prepare 5 ppb U solution)
1	5 ml volumetric pipet (to pipet 0.02M HNO_3 into 50 ml centrifuge tubes)
1	4000 ml plastic bottle (for preparation of 5 ppb U solution)
3	glass droppers (for adjusting pH by addition of HNO_3)
	weighing paper
1	Eppendorf pipet (for taking 5 ml samples)
	8007 Alpha Alumina (surface area 0.0686 m^2/g; from NIST)
	reagent grade $NaHCO_3$ (23A)
	500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike
4L	0.1 m $NaNO_3$ stock solution
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3
	ultrapure water

PREPARATION:

1. Preclean:

40	125 ml PP bottles
78	50 ml centrifuge tubes
1	100 ml volumetric pipet
1	10 ml volumetric pipet
1	5 ml volumetric pipet
1	4000 ml plastic bottle
3	glass droppers

2. Prepare:
   - 500 ppb U stock solution from 50 ppm ^{233}U commercial spike
   - 4L 0.1 m $NaNO_3$ stock solution (lot 7308 KDJE)
   - 1L stock solution of 1.0 m HNO_3
   - 1L stock solution of 0.1 m HNO_3
   - 1L stock solution of 0.02 m HNO_3
   - 1L stock solution of 0.01 m HNO_3

1600ml.

PROCEDURE:

Solution B-8007 (1 bottle for each pH value)

- Initial ΣU = 5 ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 100 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8006 alpha alumina to use = 0.200±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

12/8/92 1630hrs JP

a) In a pre-cleaned 4L plastic bottle, prepare 4000 g of 5 ppb U solution by diluting 40 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 4000 g by carefully taring 0.1 m $NaNO_3$ solution into a plastic bottle on a Mettler 4600 balance.

12/9/92 1330hrs JP

b) Into each of 29 125 ml PP bottles labeled B-8007*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 100 g of the 5 ppb U solution.

Into each of 10 125 ml PP bottles labeled B-8007-C*pH*i*a (or b) [where *i* is 2, 4, 6, 8, or 9.5, representing the approximate initial pH of the solution], tare 100 g of the 5 ppb U solution. These are control solutions to determine uranium loss to the container walls as a function of pH.

Transfer the remaining solution into a 125 ml PP bottle labeled B-8007*IU. Take two 5 ml samples from B-8007*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8007-IU*a (or b)] and pre-weighed 50 ml centrifuge tubes containing 5 g of a 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

Sample	Wt. of tube	Wt of tube + 5g 0.02M HNO_3	Wt of tube + 5g 0.02M HNO_3 + 5 ml B-8007*IU
B-8007*IU*a	10.73	15.73	20.71
B-8007*IU*b	10.73	15.74	20.77

12/10/92 1100hrs JP

c) 1. For each solution B-8007*pH*i* and B-8007-C*pH*i*, where *i*≤5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). The automatic temperature compensator (ATC) probe should be immersed in water in a separate container. Adjust the pH to the value *i* of each solution by adding with Eppendorf pipettes HNO_3 solution, the concentration and approximate amount of which is given in Table B-8007. Swirl the solutions by hand. Record the amount and concentration of solution added. Also record the attained pH. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically, together with the bottles prepared in step 2 below.

2. For each solution B-8007*pH*i* and B-8007-C*pH*i*, where *i*>5.1:

Measure and record the initial pH (~5.1, based on EQ3 calculation). Tare onto weighing paper reagent grade $NaHCO_3$ in the amounts listed in Table B-8007 and transfer into the respective PP bottles. Swirl the solutions by hand. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to ~120 rpm. Monitor the pH periodically until the pH becomes constant and in equilibrium with atmospheric $CO_2(g)$. This equilibrium process may take at least ten days.

12/23/92 1030hrs JP

d) Tare 0.200±0.001 g of 8007 alpha alumina onto weighing paper, and carefully transfer into each of the B-8007*pH*i* (**not** the B-8007-C*pH*i*) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker.

Filtered thru Dynaguard 0.2 um filter.

e) After equilibrium is reached (at least 10 days), take two 5 ml samples from each bottle B-8007*pH*i* and B-8007-C*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8007*pH*i*a (or b)] and pre-weighed 50 ml centrifuge tubes containing 5 g of 0.02 M HNO_3. Swirl each tube and save for later analysis of uranium concentration by alpha-spectrometry.

1/13/93 0920 hrs. JP

Measure and record the pH and temperature of solutions B-8007*pH*i* and B-8007-C*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

f) Analyze the U concentration by alpha-spectrometry.

Table B-8007

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8007*pH2.00	5.07	2.00	1.24	1.00	2.01
B-8007*pH2.25	5.14	2.25	0.70	1.00	2.27
B-8007*pH2.50	5.15	2.50	0.40	1.00	2.52
B-8007*pH2.75	5.02	2.75	0.21	1.00	2.71
B-8007*pH3.00	5.07	3.00	1.10	0.10	3.00
B-8007*pH3.25	5.08	3.25	0.64	0.10	3.26
B-8007*pH3.50	5.11	3.50	0.40	0.10	3.48
B-8007*pH3.75	5.16	3.75	0.23	0.10	3.75
B-8007*pH4.00	5.15	4.00	0.14	0.10	4.00
B-8007*pH4.25	5.14	4.25	0.64	0.01	4.25
B-8007*pH4.50	5.04	4.50	0.30	0.01	4.45
B-8007*pH4.75	5.11	4.75	0.16	0.01	4.75
B-8007*pH5.00	5.12	5.00	0.04	0.01	4.95
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8007*pH5.25	5.11	5.25	0.000025		
B-8007*pH5.50	5.16	5.50	0.000056		
B-8007*pH5.75	5.15	5.75	0.000083		
B-8007*pH6.00	5.14	6.00	0.000122		
B-8007*pH6.25	5.13	6.25	0.000169		
B-8007*pH6.50	5.13	6.50	0.000261		
B-8007*pH6.75	5.12	6.75	0.0004		
B-8007*pH7.00	5.14	7.00	0.000649		
B-8007*pH7.25	5.13	7.25	0.00118		
B-8007*pH7.50	5.16	7.50	0.00192		
B-8007*pH7.75	5.06	7.75	0.00357		
B-8007*pH8.00	5.10	8.00	0.00632		
B-8007*pH8.25	5.07	8.25	0.01165		
B-8007*pH8.50	5.12	8.50	0.020275		
B-8007*pH8.75	5.17	8.75	0.038		
B-8007*pH9.00	5.18	9.00	0.07275		

Sample	Initial pH	Target pH	Volume HNO3 added (ml)	Molarity of HNO3 used	pH attained
B-8007-C*pH2*a	5.05	2.00	1.12	1.00	2.00
B-8007-C*pH2*b	5.04	2.00	1.10	1.00	1.99
B-8007-C*pH4*a	5.10	4.00	0.11	0.10	4.00
B-8007-C*pH4*b	5.12	4.00	0.12	0.10	3.99
			Amount of NaHCO3 added (grams)	(lot 897186A)	
B-8007-C*pH6*a	5.12	6.00	0.000118		
B-8007-C*pH6*b	5.14	6.00	0.000119		
B-8007-C*pH8*a	5.09	8.00	0.00626		
B-8007-C*pH8*b	5.08	8.00	0.00615		
B-8007-C*pH9.5*a	5.12	9.50	0.33481		
B-8007-C*pH9.5*b	5.15	9.50	0.33487		

B-8007 pH Table

Sample	Date (1000hrs)	Date (1345hrs)	Date (1330hrs)	Date	Date
	12/16/92	12/21/92	1/13/93		
	pH	pH	pH	pH	pH
B-8007*pH2.00	1.94	1.93	1.92/18.8°C		
B-8007*pH2.25	2.18	2.17	2.16/18.8°C		
B-8007*pH2.50	2.43	2.42	2.37/18.8°C		
B-8007*pH2.75	2.68	2.69	2.66/18.8°C		
B-8007*pH3.00	2.97	2.96	2.92/18.8°C		
B-8007*pH3.25	3.21	3.23	3.19/18.9°C		
B-8007*pH3.50	3.41	3.43	3.41/18.9°C		
B-8007*pH3.75	3.64	3.65	3.65/18.9°C		
B-8007*pH4.00	3.88	3.86	3.87/18.9°C		
B-8007*pH4.25	4.17	4.22	4.26/19.0°C		
B-8007*pH4.50	4.47	4.49	4.59/19.0°C		
B-8007*pH4.75	4.70	4.71	4.85/19.0°C		
B-8007*pH5.00	5.00	4.96	5.17/19.0°C		
B-8007*pH5.25	5.25	5.25	5.51/19.0°C		
B-8007*pH5.50	5.42	5.33	5.49/19.0°C		
B-8007*pH5.75	5.63	5.63	5.84/19.0°C		
B-8007*pH6.00	~~5.99~~ 5.69	5.65	6.04/19.1°C		
B-8007*pH6.25	5.99	5.96	6.25/19.1°C		
B-8007*pH6.50	6.45	6.40	6.54/19.1°C		
B-8007*pH6.75	6.56	6.57	6.75/19.1°C		
B-8007*pH7.00	6.78	6.72	6.81/19.2°C		
B-8007*pH7.25	7.06	7.08	7.06/19.5°C		
B-8007*pH7.50	7.25	7.24	7.25/19.5°C		
B-8007*pH7.75	7.58	7.60	7.57/19.4°C		
B-8007*pH8.00	7.85	7.86	7.84/19.4°C		
B-8007*pH8.25	8.12	8.13	8.14/19.4°C		
B-8007*pH8.50	8.39	8.40	8.43/19.4°C		
B-8007*pH8.75	8.65	8.66	8.68/19.4°C		
B-8007*pH9.00	8.92	8.92	8.92/19.4°C		
B-8007-C*pH2*a	1.99	1.98	2.03/19.2°C		
B-8007-C*pH2*b	1.99	1.99	2.04/19.3°C		
B-8007-C*pH4*a	3.97	3.97	4.01/19.2°C		
B-8007-C*pH4*b	3.93	3.93	3.97/19.2°C		
B-8007-C*pH6*a	5.85	5.86	5.97/19.2°C		
B-8007-C*pH6*b	5.74	5.74	5.85/19.2°C		
B-8007-C*pH8*a	7.85	7.85	7.88/19.3°C		
B-8007-C*pH8*b	7.82	7.84	7.88/19.3°C		
B-8007-C*pH9.5*a	9.24	9.35	9.43/19.4°C		
B-8007-C*pH9.5*b	9.21	9.35	9.44/19.4°C		

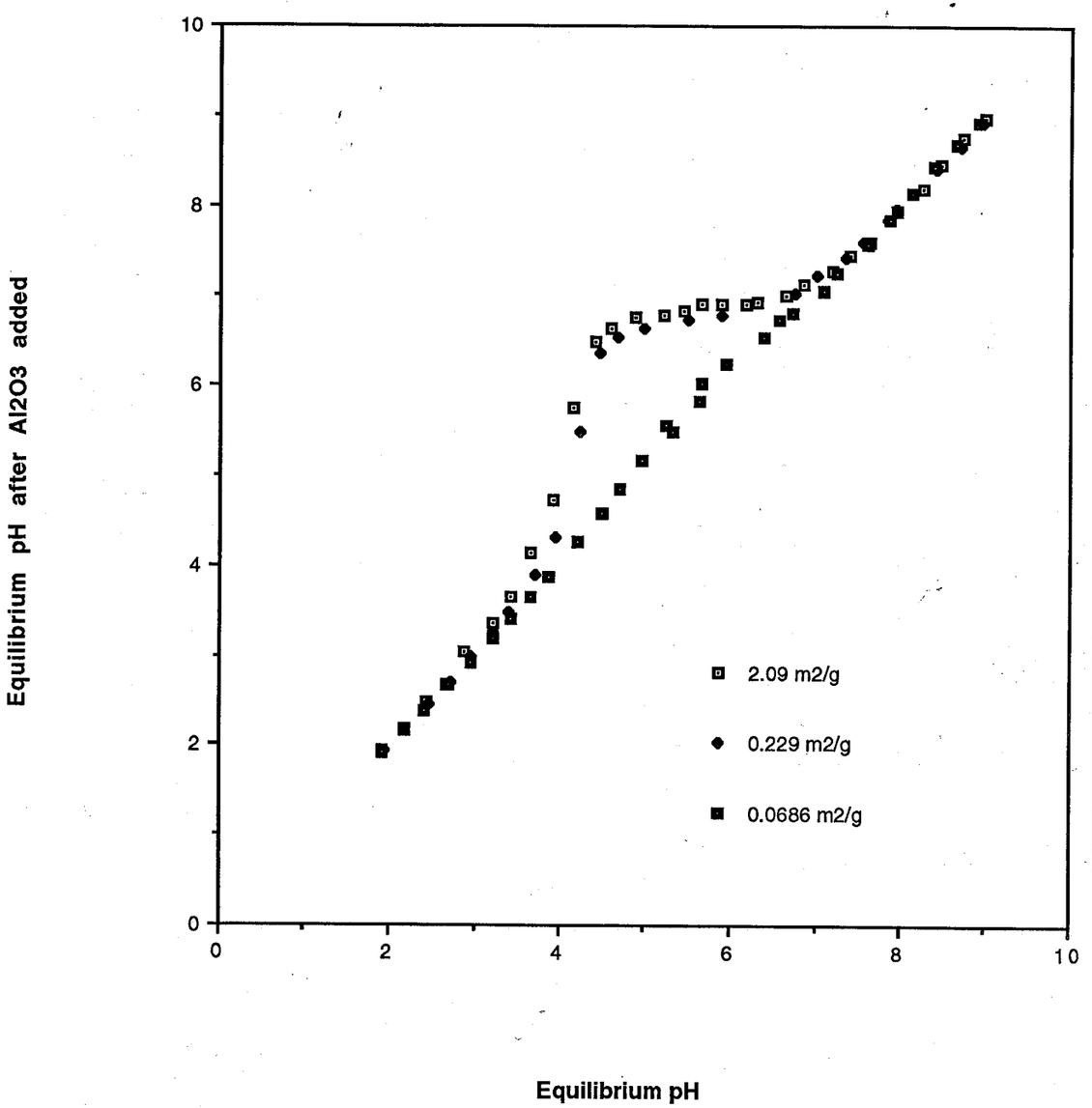
B-8007 data Table

Sample	Weight of tube	Weight of tube plus 5g 0.02M HNO3	Weight of tube plus 5g 0.02M HNO3 plus 5ml of sample
B-8007*pH2.00*a	10.77	15.79	20.83
B-8007*pH2.00*b	10.72	15.73	20.77
B-8007*pH2.25*a	10.77	15.76	20.79
B-8007*pH2.25*b	10.77	15.79	20.83
B-8007*pH2.50*a	10.87	15.87	20.90
B-8007*pH2.50*b	10.87	15.86	20.91
B-8007*pH2.75*a	10.72	15.70	20.72
B-8007*pH2.75*b	10.92	15.92	20.96
B-8007*pH3.00*a	10.72	15.70	20.73
B-8007*pH3.00*b	10.72	15.71	20.75
B-8007*pH3.25*a	10.76	15.74	20.76
B-8007*pH3.25*b	10.87	15.85	20.88
B-8007*pH3.50*a	10.76	15.74	20.76
B-8007*pH3.50*b	10.77	15.74	20.77
B-8007*pH3.75*a	10.76	15.76	20.76
B-8007*pH3.75*b	10.72	15.70	20.74
B-8007*pH4.00*a	10.72	15.72	20.73
B-8007*pH4.00*b	10.76	15.74	20.77
B-8007*pH4.25*a	10.73	15.72	20.73
B-8007*pH4.25*b	10.82	15.80	20.84
B-8007*pH4.50*a	10.77	15.77	20.78
B-8007*pH4.50*b	10.73	15.72	20.75
B-8007*pH4.75*a	10.65	15.63	20.66
B-8007*pH4.75*b	10.76	15.74	20.76
B-8007*pH5.00*a	10.92	15.91	20.91
B-8007*pH5.00*b	10.73	15.72	20.75
B-8007*pH5.25*a	10.71	15.68	20.69
B-8007*pH5.25*b	10.72	15.68	20.71
B-8007*pH5.50*a	10.71	15.70	20.70
B-8007*pH5.50*b	10.73	15.72	20.74
B-8007*pH5.75*a	10.70	15.69	20.71
B-8007*pH5.75*b	10.86	15.86	20.87
B-8007*pH6.00*a	10.77	15.75	20.77
B-8007*pH6.00*b	10.64	15.63	20.67
B-8007*pH6.25*a	10.72	15.70	20.71
B-8007*pH6.25*b	10.76	15.75	20.78
B-8007*pH6.50*a	10.70	15.69	20.71
B-8007*pH6.50*b	10.93	15.92	20.95
B-8007*pH6.75*a	10.65	15.63	20.64
B-8007*pH6.75*b	10.87	15.86	20.88
B-8007*pH7.00*a	10.80	15.79	20.81
B-8007*pH7.00*b	10.73	15.71	20.74

B-8007 data Table

B-8007*pH7.25*a	10.86	15.86	20.88
B-8007*pH7.25*b	10.69	15.68	20.71
B-8007*pH7.50*a	10.74	15.73	20.74
B-8007*pH7.50*b	10.72	15.71	20.73
B-8007*pH7.75*a	10.74	15.73	20.74
B-8007*pH7.75*b	10.90	15.90	20.92
B-8007*pH8.00*a	10.87	15.86	20.89
B-8007*pH8.00*b	10.79	15.78	20.80
B-8007*pH8.25*a	10.72	15.71	20.71
B-8007*pH8.25*b	10.70	15.68	20.71
B-8007*pH8.50*a	10.71	15.72	20.72
B-8007*pH8.50*b	10.86	15.84	20.88
B-8007*pH8.75*a	10.77	15.76	20.77
B-8007*pH8.75*b	10.77	15.77	20.80
B-8007*pH9.00*a	10.73	15.72	20.73
B-8007*pH9.00*b	10.81	15.81	20.84
B-8007-C*pH2*a1	10.73	15.73	20.76
B-8007-C*pH2*a2	10.72	15.71	20.75
B-8007-C*pH2*b1	10.78	15.76	20.78
B-8007-C*pH2*b2	10.78	15.75	20.79
B-8007-C*pH4*a1	10.73	15.72	20.74
B-8007-C*pH4*a2	10.71	15.68	20.71
B-8007-C*pH4*b1	10.66	15.63	20.66
B-8007-C*pH4*b2	10.76	15.75	20.78
B-8007-C*pH6*a1	10.78	15.77	20.80
B-8007-C*pH6*a2	10.73	15.73	20.75
B-8007-C*pH6*b1	10.78	15.76	20.77
B-8007-C*pH6*b2	10.83	15.81	20.82
B-8007-C*pH8*a1	10.73	15.71	20.72
B-8007-C*pH8*a2	10.72	15.72	20.74
B-8007-C*pH8*b1	10.72	15.72	20.75
B-8007-C*pH8*b2	10.75	15.75	20.77
B-8007-C*pH9.5*a1	10.73	15.72	20.76
B-8007-C*pH9.5*a2	10.74	15.73	20.76
B-8007-C*pH9.5*b1	10.65	15.65	20.69
B-8007-C*pH9.5*b2	10.92	15.91	20.95

1/18/93 gp Graph of eq pH before and after Al_2O_3 addition for the 3 surface area sizes.



1/22/93 gp Fill in samples for exp B-8006
Prepared to fill in blank areas of pH diagram

Took sample B-8006-C*pH6*b at pH 5.88 and added .09 ml of 0.01 m HNO_3 to reach pH5.23.
Relabelled sample B-8006*pH5.25A.
Took sample B-8007-C*pH6*b at pH 5.85 and added 0.01 ml of 0.01 m HNO_3 to reach pH5.75
Relabelled sample B-8006*pH 5.50A
Took sample B-8006*C*pH6*a and added about .00006 g $NaHCO_3$ (lot #971186A) to reach pH6.45.
Added 0.1 ml of 0.01 m HNO_3 to reach pH6.20.
Relabelled sample B-8006*pH6.25A.
Took sample B-8007-C*pH6*a and added about .0001 g $NaHCO_3$ (lot #971186A) to reach pH 6.55.
Added .03 ml of 0.01 M HNO_3 to reach pH6.48.
Relabelled sample B-8006*pH6.50A.

1/25/93 gp Added .200 ± .001 g of α alumina (8006) to each of the above samples. Samples were covered + placed on shaker.

pH of samples was measured periodically to determine equilibrium with air.

	2/18/93 pH	2/22/93 pH	2/24/93 pH	2/25/93
B-8006*pH5.25A	6.82	6.93	6.89	6.89/18.6°C
B-8006*pH5.50A	6.86	6.92	6.91	6.92/19.8°C
B-8006*pH6.25A	6.89	7.01	6.98	6.98/19.6°C
B-8006*pH6.50A	6.90	7.05	7.05	7.04/19.7°C

2/25/93 gp
Took two 5ml samples filtered thru Dynagard 0.2 μm filter from each bottle with an Eppendorf pipet, transfer into pre-labeled & pre-weighed 50 ml centrifuge tubes containing 5 g of 0.02 M HNO_3.

Sample	Weight of Tube	Weight of tube + 5g 0.02m HNO_3	Weight of tube + 5g 0.02m HNO_3 + 5ml of sample
B-8006*pH5.25A*a	10.68	15.75	20.82
B-8006*pH5.25A*b	10.86	15.91	20.97
B-8006*pH5.50A*a	10.75	15.75	20.82
B-8006*pH5.50A*b	10.86	15.86	20.90
B-8006*pH6.25A*a	10.80	15.82	20.82
B-8006*pH6.25A*b	10.77	15.76	20.80
B-8006*pH6.50A*a	10.75	15.75	20.78
B-8006*pH6.50A*b	10.73	15.76	20.79

1/27/93 RP

U-233 Alpha Spectrometry Analysis Procedure

Preparation:

1. All glass should be washed in a hot acid bath (4 liter beaker with cover glass) of fuming nitric acid over night (> 4 hours). Once the temperature of the acid bath is near room temperature, the acid washed glass should be quickly inserted into nanopure H_2O in another large beaker inside the fumehood. Two pairs of gloves and goggles should be used during this operation as there is a possibility of splashing and nitric acid is a real hazard. The rinsed glassware should then be rinsed at least three more times in the sink with nanopure water. The rinsed dishes can either be air dried on a covered tray or placed in the drying oven.

1 column = 1 25 ml glass beaker + sample

Waste Disposal:

1. Used ion exchange resins will be stored in nonbreakable, screw-top containers. Containers will be clearly labelled. Radioisotope storage containers will be labelled with the following information: a) radioisotope; b) physical form; c) type of emission; d) activity in Curies; and e) dose rate at container surface. The exchange resins will be regenerated to reduce the level of radioactivity and to reduce the volume that will be disposed. Responsibility for regeneration and disposal of the ion exchange resins will be assumed by the RPC.

2 Disposal of the solutions containing several isotopes (*i.e.* solutions containing both ^{232}U and ^{233}U) down the sewer is restricted by the release limits set forth in TRC 21.303 Appendix 21-A (Note 1). This note states that the release is limited so that the sum of the the ratios in the solution to that of the limiting concentration for each isotope in the solution will be < 1. For example, in the case of two isotopes (^{232}U and ^{233}U) present in concentrations C_{232U} and C_{233U} with maximum permissible concentrations MPC_{232U} and MPC_{233U} : $(C_{232U}/MPC_{232U}) + (C_{233U}/MPC_{233U}) \leq 1$. Note that the limits for MPC_{232U} and MPC_{233U} are 0.8 nCi/ml and 0.9 nCi/ml, respectively.

3. All waste solutions derived during ion exchange column manipulations either contain high concentrations of N (nitrate and ammonium (NH_4), which readily converts to nitrate) or acid and require liberal dilution prior to and during disposal via the sink.

Analytical Procedure:

A. Spiking and co-precipitation

1. Spike all samples in a rack requiring the same spike at one time (*i.e.* all spike 25 C at same time). Use the centrifuge tube stand in glass beaker to hold the tube being spiked. Use the syringe stand to hold the pasteur pipet between sample spikings. Tare a pre-folded sample boat (plastic) on balance. Using a pasteur pipet (a new one for each time a series of samples will be spiked with a different ^{232}U solution) and a small rubber bulb add predetermined mass of spike solution to sample boat and record weight. Error in spike mass should be to the low side. Dispose of pasteur pipet in special radioactive wastebasket after samples have been spiked.

2. Transfer spike quantitatively to sample centrifuge tube using squirt bottle of 0.1 N HNO_3. Use enough of 0.1 N HNO_3 during each spike transfer (~ 5 ml) to obtain roughly equal volumes in the centrifuge tubes. Dispose of sample boat into special radioactive wastebasket.

3. Homogenize sample after addition of spike by replacing lid and swirling the centrifuge tube. Allow spike to equilibrate at least 24 hours before next step.

4. Add carrier to one row of tubes in a rack at a time. Add 1 ml of Fe carrier using Eppendorf fixed volume pipet and disposable tips. Homogenize sample after addition of carrier by replacing lid and swirling the centrifuge tube. Allow sample to equilibrate for 24 hours.

5. Start step B1 now and continue with step B2 simultaneously with the following steps of A (A5 - A7) so that once you have completed step A7 you can immediately proceed with step B3. Bring the pH up to 7 with the addition of concentrated NH_4OH from the reagent bottle using 6 to 7 drops from the plastic pipet attached to the NH_4OH reagent bottle. This is strongly exothermic and the solution should be gently swirled during the addition of the base. Note that the NH_4OH should be from a closed reagent container (*eg.* new 2 l bottle) since this will ensure that minimal CO_2 will be present in the base which would lower yield. This step precipitates about 2.5 ml of $Fe(OH)_3$.

6. The solution can then be centrifuged (labeled 50 ml centrifuge tubes) and the supernate is discarded into a waste beaker.

7. The $Fe(OH)_3$ precipitates are washed with about 10 ml of ultrapure water and agitated using the hand homogenizer, centrifuged and washings discarded. This step is repeated once. This is necessary to get rid of the excess NH_4.

B. Uranium-thorium separation

1. Prepare a labeled (Sample ID) anion ion exchange column (8 -10 cm high, 1 cm diameter) by placing a glass wool plug in the bottom of the column and adding a slurry (in ultrapure water) of Dowex 1 x 8 100-200 mesh chloride form ion exchange resin. Use the teflon column holders and collect the waste liquid in a plastic beaker (100 ml of greater).

2. Add 30-40 ml of 8 N Ammonium Nitrate - 0.1 N Nitric acid into anion exchange column and elute into plastic waste beaker.

3. The samples will be lowered to pH =1 using 250 µl of 1.0 N HNO_3 and diluting to 5.0 ml with 0.1 N HNO_3 using squirt bottle. Make sure $Fe(OH)_3$ is entirely dissolved (agitate) prior to adding the ammonium nitrate. Next, saturate the solution with 5.9 g of ammonium nitrate and shake until all is dissolved. This reaction is highly endothermic.

4. Add the sample onto the column and allow to drain into 100 ml plastic beaker. Elute Fe into the beaker using 80 ml, added in 20 ml increments using squirt bottle, of 8 N NH_4NO_3 - 0.1 N HNO_3. Rinse the centrifuge tube with the 20

ml aliquot and pour onto column and mark the level of the fluid. Test for the presence of Fe after 70 to 80 ml have drained by placing a drop of NH_4CSN on the convex side of watch glass and allowing a drop of the eluate to come in contact with ammonium cyanide. If Fe is present then the solution will turn red. Continue to eluate the Fe until there is no red upon testing for Fe. Empty the contents of the 100 ml plastic beaker into a waste beaker.

5. Next add 100 ml, in 20 ml aliquots, of 8 N HCL acid and elute the thorium into the same beaker. Dispose of solution in beaker down the sink with liberal flushing.

6. Using a squirt bottle, add no more than 5 ml of 0.1 N nitric acid to the column and allow to **drain into waste beaker**. The column changes color slightly from orange to slightly yellow. Next, add 50 ml of 0.1 N nitric acid in 10 ml increments and drain the uranium into a labeled and acid washed cleaned **100 ml glass beaker**. The solution is then placed on a hot plate to reach just to dryness. Allow beaker to cool moderately and then immediately proceed with step C1. As long as the beaker is slightly warm and 0.001 N HNO_3 is added the U is more easily put into solution and the pH is closer to the desired pH.

stop point overnight

C. Solvent extraction and plating

1. A total of 4 pasteur pipets are used in the following steps and it is imperative to keep them separate by labeling the rubber bulbs used with them. The dried U eluate is taken up with two ml of 0.001 N HNO_3 (pH 3) issued from a squirt bottle. Using a new pasteur pipet (W on bulb) carefully and completely wash the beaker with the acid. Transfer the acid solution to a labeled glass 12 ml centrifuge tube. Repeat this step twice more with 0.5 - 1 ml of the acid.

2. Add two drops of Methyl Orange indicator solution using a pasteur pipet (MO on bulb) to solution in centrifuge tube. Bring pH up to 3.0 with dilute 0.1 N NaOH solution using a pasteur pipet (NaO on bulb) by adding a few drops at a time and homogenizing the contents of the tube with pasteur pipet used to transfer the U eluate (W on bulb). The color should change from pink to slightly orange. The colors can be seen in test tube set which covers the pH range. If steps in B were done properly the total volume should be 5-6 ml.

3. Add 1-2 ml of the 0.4 M TTA in benzene solution to the centrifuge tube.

4. Homogenize and extract the U using the same pasteur pipet used in step C1 (W on bulb). The TTA solution should be red or orange (depending on U concentration) and this should be quickly evident (within 30 seconds to 1 minute).

5. Solution is centrifuged for 1-2 minutes, making sure that the tube is covered with Parafilm.

6. Carefully clean a stainless steel planchet and label it with a sharp pointed object. The label should have sample ID, date, initials of the person plating and U. Label a glassine stamp envelope with the same information. The mounting ring

should be on the hot plate and the hot plate should be set to red mark (~3). Place the plate, labeled side down, on the mounting ring just prior to next step.

7. The TTA solution is carefully separated using a clean pasteur pipet (P on bulb), taking care not to include any of the HNO_3 solution. This is most easily accomplished by slightly tilting the tube so that the TTA bulges on top of the acid. A total of two aliquots using the pasteur pipet (P on bulb) are used to retrieve all the TTA.

8. The TTA is evaporated drop-wise (vertically) on heated steel plates that have been placed on mounting ring, making sure that spattering is avoided.

9. Remove the plate from the mounting ring and repeat the TTA extraction (Steps 3 - 8).

10. Pass the plate through the flame of a propane flame inside the fume hood to burn off the organic deposit. Allow the plate to cool on the edge of the hot plate.

11. Place plates in labeled glassine stamp envelopes and count samples ASAP after plating, recording the channel in which the sample is counted on the envelope.

D. Cleaning up

1. The solution remaining in the centrifuge tube should be placed into a glass waste beaker. The centrifuge tube should be rinsed with 0.1 N HNO_3 and the rinse should also be placed into the waste beaker. This beaker should remain inside the fumehood. The volume of the solution should be reduced by gentle heating until dry.

2. The used anion exchange resin should be rinsed with water from a squirt bottle into the used resin bottle (see instructions above).

3. All glassware should then be washed using alconox. The glassware should be rinsed with the single pass water. The glassware is then ready for the fuming nitric acid bath.

1/27/93 ^{232}U spiking information for experiments B-8005, B-8006, and B-8007.

Identification - sample no. or id

Spike # - ^{232}U spike #
25B = 204.88 pCi/g
25C = 20.477 pCi/g

Init. act./g - initial activity in nCi/g of sample. Since all samples have 5 ppb U initial activity is 0.04796 nCi/g.

% sorb - percent of U sorbed onto the α-alumina (estimated from previous experiments on clinoptilolite).

Final act./g - final activity in nCi/g of sample (Init act/g x (1 - %sorb))

Aliqt (g) - mass of aliquot taken for analysis from each sample.

Act (total) - Final act/g x Aliqt

Wt (g) - mass of ^{232}U spike added to sample.

B-8005 Anticipate

Sorption Experiment B-8005							
Initial U is 5 ppb		500 ppb = 4.796 nCi/g					
Identification	**Init. act./g**	**% sorb**	**Finl act./g**	**Aliqt (g)**	**Act. total**	**Spike#**	**Wt (g)**
B-8005*pH2.00*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005*pH2.00*b	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005*pH2.25*a	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8005*pH2.25*b	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8005*pH2.50*a	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8005*pH2.50*b	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8005*pH2.75*a	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8005*pH2.75*b	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8005*pH3.00*a	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8005*pH3.00*b	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8005*pH3.25*a	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8005*pH3.25*b	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8005*pH3.50*a	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8005*pH3.50*b	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8005*pH3.75*a	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005*pH3.75*b	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005*pH4.00*a	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8005*pH4.00*b	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8005*pH4.25*a	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8005*pH4.25*b	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8005*pH4.50*a	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8005*pH4.50*b	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8005*pH4.75*a	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8005*pH4.75*b	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8005*pH5.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH5.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH6.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*

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B-8005 Anticipate

Identification	Init. act./g	% sorb	Finl act./g	Aliqt (g)	Act. total	Spike#	Wt (g)
B-8005*pH6.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH7.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH7.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH7.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH7.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8005*pH7.50*a	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8005*pH7.50*b	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8005*pH7.75*a	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8005*pH7.75*b	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8005*pH8.00*a	0.04796	70	0.0144	5	0.0719	*25C*	*0.7026*
B-8005*pH8.00*b	0.04796	70	0.0144	5	0.0719	*25C*	*0.7026*
B-8005*pH8.25*a	0.04796	55	0.0216	5	0.1079	*25C*	*1.0540*
B-8005*pH8.25*b	0.04796	55	0.0216	5	0.1079	*25C*	*1.0540*
B-8005*pH8.50*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005*pH8.50*b	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005*pH8.75*a	0.04796	15	0.0408	5	0.2038	**25B**	**0.1990**
B-8005*pH8.75*b	0.04796	15	0.0408	5	0.2038	**25B**	**0.1990**
B-8005*pH9.00*a	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005*pH9.00*b	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH2*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH2*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH2*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH2*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH4*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH4*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH4*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH4*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8005-C*pH6*a1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005-C*pH6*a2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005-C*pH6*b1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005-C*pH6*b2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8005-C*pH8*a1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005-C*pH8*a2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005-C*pH8*b1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005-C*pH8*b2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8005-C*pH9.5*a1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8005-C*pH9.5*a2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8005-C*pH9.5*b1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8005-C*pH9.5*b2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8005*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
B-8005*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
	SUM 25B	**5.5245**	*SUM 25C*	*28.504*			

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1/27/93 gp 1300hr B-8005 samples spiked

B-8005 U-232 Spiking

Sorption Experiment B-8005

Initial U is 5 ppb 500 ppb = 4.796 nCi/g

Identification	Spike#	Wt (g)	Actual wt(g)
B-8005*IUa	**25B**	0.234	.243
B-8005*IUa	**25B**	0.234	.243
B-8005-C*pH2*a1	**25B**	0.211	.205
B-8005-C*pH2*a2	**25B**	0.211	.225
B-8005-C*pH2*b1	**25B**	0.211	.210
B-8005-C*pH2*b2	**25B**	0.211	.214
B-8005-C*pH4*a1	**25B**	0.211	.216
B-8005-C*pH4*a2	**25B**	0.211	.205
B-8005-C*pH4*b1	**25B**	0.211	.206
B-8005-C*pH4*b2	**25B**	0.211	.207
B-8005-C*pH6*a1	**25B**	0.187	.191
B-8005-C*pH6*a2	**25B**	0.187	.190
B-8005-C*pH6*b1	**25B**	0.187	.192
B-8005-C*pH6*b2	**25B**	0.187	.194
B-8005-C*pH8*a1	*25C*	1.405	1.401
B-8005-C*pH8*a2	*25C*	1.405	1.408
B-8005-C*pH8*b1	*25C*	1.405	1.409
B-8005-C*pH8*b2	*25C*	1.405	1.401
B-8005-C*pH9.5*a1	*25C*	0.937	.934
B-8005-C*pH9.5*a2	*25C*	0.937	.935
B-8005-C*pH9.5*b1	*25C*	0.937	.935
B-8005-C*pH9.5*b2	*25C*	0.937	.935
B-8005*pH2.00*a	**25B**	0.187	.187
B-8005*pH2.00*b	**25B**	0.187	.187
B-8005*pH2.25*a	**25B**	0.181	.184
B-8005*pH2.25*b	**25B**	0.181	.182
B-8005*pH2.50*a	**25B**	0.176	.181
B-8005*pH2.50*b	**25B**	0.176	.185
B-8005*pH2.75*a	**25B**	0.170	.170
B-8005*pH2.75*b	**25B**	0.170	.179
B-8005*pH3.00*a	*25C*	1.639	1.645
B-8005*pH3.00*b	*25C*	1.639	1.640
B-8005*pH3.25*a	*25C*	1.546	1.542
B-8005*pH3.25*b	*25C*	1.546	1.551
B-8005*pH3.50*a	*25C*	1.499	1.494
B-8005*pH3.50*b	*25C*	1.499	1.494
B-8005*pH3.75*a	*25C*	1.405	1.404
B-8005*pH3.75*b	*25C*	1.405	1.406
B-8005*pH4.00*a	*25C*	0.820	.823
B-8005*pH4.00*b	*25C*	0.820	.826
B-8005*pH4.25*a	*25C*	0.586	.588
B-8005*pH4.25*b	*25C*	0.586	.589

Identification	Spike#	Wt (g)	Actual wt(g)
B-8005*pH4.50*a	*25C*	0.422	.422 .425
B-8005*pH4.50*b	*25C*	0.422	.420 1/27/93
B-8005*pH4.75*a	*25C*	0.187	.187
B-8005*pH4.75*b	*25C*	0.187	.190
B-8005*pH5.00*a	*25C*	0.117	.123
B-8005*pH5.00*b	*25C*	0.117	.118
B-8005*pH5.25*a	*25C*	0.117	.121
B-8005*pH5.25*b	*25C*	0.117	.118
B-8005*pH5.50*a	*25C*	0.117	.118
B-8005*pH5.50*b	*25C*	0.117	.117
B-8005*pH5.75*a	*25C*	0.117	.117
B-8005*pH5.75*b	*25C*	0.117	.118
B-8005*pH6.00*a	*25C*	0.117	.118
B-8005*pH6.00*b	*25C*	0.117	.119
B-8005*pH6.25*a	*25C*	0.117	.121
B-8005*pH6.25*b	*25C*	0.117	.118
B-8005*pH6.50*a	*25C*	0.117	.118
B-8005*pH6.50*b	*25C*	0.117	.117
B-8005*pH6.75*a	*25C*	0.117	.119
B-8005*pH6.75*b	*25C*	0.117	.118
B-8005*pH7.00*a	*25C*	0.117	.118
B-8005*pH7.00*b	*25C*	0.117	.119
B-8005*pH7.25*a	*25C*	0.117	.122
B-8005*pH7.25*b	*25C*	0.117	.121
B-8005*pH7.50*a	*25C*	0.234	.235
B-8005*pH7.50*b	*25C*	0.234	.233
B-8005*pH7.75*a	*25C*	0.468	.467
B-8005*pH7.75*b	*25C*	0.468	.469
B-8005*pH8.00*a	*25C*	0.703	.701
B-8005*pH8.00*b	*25C*	0.703	.703
B-8005*pH8.25*a	*25C*	1.054	1.054
B-8005*pH8.25*b	*25C*	1.054	1.052
B-8005*pH8.50*a	**25B**	0.187	.190
B-8005*pH8.50*b	**25B**	0.187	.188
B-8005*pH8.75*a	**25B**	0.199	.197
B-8005*pH8.75*b	**25B**	0.199	.207
B-8005*pH9.00*a	**25B**	0.211	.210
B-8005*pH9.00*b	**25B**	0.211	.218

25B is 204.88 pCi/g
25C is 20.477 pCi/g

Actual Sum
SUM 25B **5.524** → 5.606g
SUM 25C *28.504* → 32.874g

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Sorption Experiment B-8006							
Initial U is 5 ppb		500 ppb = 4.796 nCi/g					
Identification	**Init. act./g**	**% sorb**	**Finl act./g**	**Aliqt (g)**	**Act. total**	**Spike#**	**Wt (g)**
B-8006*pH2.00*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006*pH2.00*b	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006*pH2.25*a	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8006*pH2.25*b	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8006*pH2.50*a	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8006*pH2.50*b	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8006*pH2.75*a	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8006*pH2.75*b	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8006*pH3.00*a	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8006*pH3.00*b	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8006*pH3.25*a	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8006*pH3.25*b	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8006*pH3.50*a	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8006*pH3.50*b	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8006*pH3.75*a	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006*pH3.75*b	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006*pH4.00*a	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8006*pH4.00*b	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8006*pH4.25*a	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8006*pH4.25*b	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8006*pH4.50*a	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8006*pH4.50*b	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8006*pH4.75*a	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8006*pH4.75*b	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8006*pH5.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH5.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH6.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*

Identification	Init. act./g	% sorb	Finl act./g	Aliqt (g)	Act. total	Spike#	Wt (g)
B-8006*pH6.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH7.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH7.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH7.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH7.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8006*pH7.50*a	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8006*pH7.50*b	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8006*pH7.75*a	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8006*pH7.75*b	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8006*pH8.00*a	0.04796	70	0.0144	5	0.0719	*25C*	*0.7026*
B-8006*pH8.00*b	0.04796	70	0.0144	5	0.0719	*25C*	*0.7026*
B-8006*pH8.25*a	0.04796	55	0.0216	5	0.1079	*25C*	*1.0540*
B-8006*pH8.25*b	0.04796	55	0.0216	5	0.1079	*25C*	*1.0540*
B-8006*pH8.50*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006*pH8.50*b	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006*pH8.75*a	0.04796	15	0.0408	5	0.2038	**25B**	**0.1990**
B-8006*pH8.75*b	0.04796	15	0.0408	5	0.2038	**25B**	**0.1990**
B-8006*pH9.00*a	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006*pH9.00*b	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH2*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH2*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH2*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH2*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH4*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH4*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH4*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH4*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8006-C*pH6*a1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006-C*pH6*a2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006-C*pH6*b1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006-C*pH6*b2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8006-C*pH8*a1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006-C*pH8*a2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006-C*pH8*b1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006-C*pH8*b2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8006-C*pH9.5*a1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8006-C*pH9.5*a2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8006-C*pH9.5*b1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8006-C*pH9.5*b2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8006*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
B-8006*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
	SUM 25B	**5.5245**	*SUM 25C*	*28.504*			

B-8006 U-232 Spiking

Sorption Experiment B-8006

Initial U is 5 ppb 500 ppb = 4.796 nCi/g

Identification	Spike#	Wt (g)
B-8006*IUa	**25B**	0.234
B-8006*IUa	**25B**	0.234
B-8006-C*pH2*a1	**25B**	0.211
B-8006-C*pH2*a2	**25B**	0.211
B-8006-C*pH2*b1	**25B**	0.211
B-8006-C*pH2*b2	**25B**	0.211
B-8006-C*pH4*a1	**25B**	0.211
B-8006-C*pH4*a2	**25B**	0.211
B-8006-C*pH4*b1	**25B**	0.211
B-8006-C*pH4*b2	**25B**	0.211
B-8006-C*pH6*a1	**25B**	0.187
B-8006-C*pH6*a2	**25B**	0.187
B-8006-C*pH6*b1	**25B**	0.187
B-8006-C*pH6*b2	**25B**	0.187
B-8006-C*pH8*a1	*25C*	1.405
B-8006-C*pH8*a2	*25C*	1.405
B-8006-C*pH8*b1	*25C*	1.405
B-8006-C*pH8*b2	*25C*	1.405
B-8006-C*pH9.5*a1	*25C*	0.937
B-8006-C*pH9.5*a2	*25C*	0.937
B-8006-C*pH9.5*b1	*25C*	0.937
B-8006-C*pH9.5*b2	*25C*	0.937
B-8006*pH2.00*a	**25B**	0.187
B-8006*pH2.00*b	**25B**	0.187
B-8006*pH2.25*a	**25B**	0.181
B-8006*pH2.25*b	**25B**	0.181
B-8006*pH2.50*a	**25B**	0.176
B-8006*pH2.50*b	**25B**	0.176
B-8006*pH2.75*a	**25B**	0.170
B-8006*pH2.75*b	**25B**	0.170
B-8006*pH3.00*a	*25C*	1.639
B-8006*pH3.00*b	*25C*	1.639
B-8006*pH3.25*a	*25C*	1.546
B-8006*pH3.25*b	*25C*	1.546
B-8006*pH3.50*a	*25C*	1.499
B-8006*pH3.50*b	*25C*	1.499
B-8006*pH3.75*a	*25C*	1.405
B-8006*pH3.75*b	*25C*	1.405
B-8006*pH4.00*a	*25C*	0.820
B-8006*pH4.00*b	*25C*	0.820
B-8006*pH4.25*a	*25C*	0.586
B-8006*pH4.25*b	*25C*	0.586

.234
.229

Identification	Spike#	Wt (g)
B-8006*pH4.50*a	*25C*	0.422
B-8006*pH4.50*b	*25C*	0.422
B-8006*pH4.75*a	*25C*	0.187
B-8006*pH4.75*b	*25C*	0.187
B-8006*pH5.00*a	*25C*	0.117
B-8006*pH5.00*b	*25C*	0.117
B-8006*pH5.25*a	*25C*	0.117
B-8006*pH5.25*b	*25C*	0.117
B-8006*pH5.50*a	*25C*	0.117
B-8006*pH5.50*b	*25C*	0.117
B-8006*pH5.75*a	*25C*	0.117
B-8006*pH5.75*b	*25C*	0.117
B-8006*pH6.00*a	*25C*	0.117
B-8006*pH6.00*b	*25C*	0.117
B-8006*pH6.25*a	*25C*	0.117
B-8006*pH6.25*b	*25C*	0.117
B-8006*pH6.50*a	*25C*	0.117
B-8006*pH6.50*b	*25C*	0.117
B-8006*pH6.75*a	*25C*	0.117
B-8006*pH6.75*b	*25C*	0.117
B-8006*pH7.00*a	*25C*	0.117
B-8006*pH7.00*b	*25C*	0.117
B-8006*pH7.25*a	*25C*	0.117
B-8006*pH7.25*b	*25C*	0.117
B-8006*pH7.50*a	*25C*	0.234
B-8006*pH7.50*b	*25C*	0.234
B-8006*pH7.75*a	*25C*	0.468
B-8006*pH7.75*b	*25C*	0.468
B-8006*pH8.00*a	*25C*	0.703
B-8006*pH8.00*b	*25C*	0.703
B-8006*pH8.25*a	*25C*	1.054
B-8006*pH8.25*b	*25C*	1.054
B-8006*pH8.50*a	**25B**	0.187
B-8006*pH8.50*b	**25B**	0.187
B-8006*pH8.75*a	**25B**	0.199
B-8006*pH8.75*b	**25B**	0.199
B-8006*pH9.00*a	**25B**	0.211
B-8006*pH9.00*b	**25B**	0.211

25B is 204.88 pCi/g
25C is 20.477 pCi/g

SUM 25B **5.524**
SUM 25C *28.504*

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B-8007 Anticipate

Sorption Experiment B-8007

Initial U is 5 ppb 500 ppb = 4.796 nCi/g

Identification	Init. act./g	% sorb	Finl act./g	Aliqt (g)	Act. total	Spike#	Wt (g)
B-8007*pH2.00*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007*pH2.00*b	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007*pH2.25*a	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8007*pH2.25*b	0.04796	22.5	0.0372	5	0.1858	**25B**	**0.1814**
B-8007*pH2.50*a	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8007*pH2.50*b	0.04796	25	0.0360	5	0.1799	**25B**	**0.1756**
B-8007*pH2.75*a	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8007*pH2.75*b	0.04796	27.5	0.0348	5	0.1739	**25B**	**0.1697**
B-8007*pH3.00*a	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8007*pH3.00*b	0.04796	30	0.0336	5	0.1679	*25C*	*1.6395*
B-8007*pH3.25*a	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8007*pH3.25*b	0.04796	34	0.0317	5	0.1583	*25C*	*1.5458*
B-8007*pH3.50*a	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8007*pH3.50*b	0.04796	36	0.0307	5	0.1535	*25C*	*1.4990*
B-8007*pH3.75*a	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007*pH3.75*b	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007*pH4.00*a	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8007*pH4.00*b	0.04796	65	0.0168	5	0.0839	*25C*	*0.8197*
B-8007*pH4.25*a	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8007*pH4.25*b	0.04796	75	0.0120	5	0.0600	*25C*	*0.5855*
B-8007*pH4.50*a	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8007*pH4.50*b	0.04796	82	0.0086	5	0.0432	*25C*	*0.4216*
B-8007*pH4.75*a	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8007*pH4.75*b	0.04796	92	0.0038	5	0.0192	*25C*	*0.1874*
B-8007*pH5.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH5.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.50*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.50*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH6.75*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*

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B-8007 Anticipate

Identification	Init. act./g	% sorb	Finl act./g	Aliqt (g)	Act. total	Spike#	Wt (g)
B-8007*pH6.75*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH7.00*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH7.00*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH7.25*a	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH7.25*b	0.04796	95	0.0024	5	0.0120	*25C*	*0.1171*
B-8007*pH7.50*a	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8007*pH7.50*b	0.04796	90	0.0048	5	0.0240	*25C*	*0.2342*
B-8007*pH7.75*a	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8007*pH7.75*b	0.04796	80	0.0096	5	0.0480	*25C*	*0.4684*
B-8007*pH8.00*a	0.04796	70	0.0144	5	0.0719	*25C*	*0.7026*
B-8007*pH8.00*b	0.04796	70	0.0144	5	0.1079	*25C*	*0.7026*
B-8007*pH8.25*a	0.04796	55	0.0216	5	0.1079	*25C*	*1.0540*
B-8007*pH8.25*b	0.04796	55	0.0216	5	0.1918	**25B**	**0.1873**
B-8007*pH8.50*a	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007*pH8.50*b	0.04796	20	0.0384	5	0.2038	**25B**	**0.1990**
B-8007*pH8.75*a	0.04796	15	0.0408	5	0.2038	**25B**	**0.1990**
B-8007*pH8.75*b	0.04796	15	0.0408	5	0.2158	**25B**	**0.2107**
B-8007*pH9.00*a	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007*pH9.00*b	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH2*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH2*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH2*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH2*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH4*a1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH4*a2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH4*b1	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH4*b2	0.04796	10	0.0432	5	0.2158	**25B**	**0.2107**
B-8007-C*pH6*a1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007-C*pH6*a2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007-C*pH6*b1	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007-C*pH6*b2	0.04796	20	0.0384	5	0.1918	**25B**	**0.1873**
B-8007-C*pH8*a1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007-C*pH8*a2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007-C*pH8*b1	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007-C*pH8*b2	0.04796	40	0.0288	5	0.1439	*25C*	*1.4053*
B-8007-C*pH9.5*a1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8007-C*pH9.5*a2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8007-C*pH9.5*b1	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8007-C*pH9.5*b2	0.04796	60	0.0192	5	0.0959	*25C*	*0.9369*
B-8007*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
B-8007*IUa	0.04796	0	0.0480	5	0.2398	**25B**	**0.2341**
	SUM 25B	**5.5245**	*SUM 25C*	*28.504*			

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1/28/93 0900 hrs GP B-8007 samples spiked

B-8007 U-232 Spiking

Sorption Experiment B-8007

Initial U is 5 ppb 500 ppb = 4.796 nCi/g

Identification	Spike#	Wt (g)	Actual wt g
B-8007*IUa	**25B**	0.234	.232
B-8007*IUa	**25B**	0.234	.238
B-8007-C*pH2*a1	**25B**	0.211	.211
B-8007-C*pH2*a2	**25B**	0.211	.210
B-8007-C*pH2*b1	**25B**	0.211	.214
B-8007-C*pH2*b2	**25B**	0.211	.216
B-8007-C*pH4*a1	**25B**	0.211	.214
B-8007-C*pH4*a2	**25B**	0.211	.214
B-8007-C*pH4*b1	**25B**	0.211	.214
B-8007-C*pH4*b2	**25B**	0.211	.212
B-8007-C*pH6*a1	**25B**	0.187	.187
B-8007-C*pH6*a2	**25B**	0.187	.189
B-8007-C*pH6*b1	**25B**	0.187	.186
B-8007-C*pH6*b2	**25B**	0.187	.186
B-8007-C*pH8*a1	*25C*	1.405	1.400
B-8007-C*pH8*a2	*25C*	1.405	1.404
B-8007-C*pH8*b1	*25C*	1.405	1.403
B-8007-C*pH8*b2	*25C*	1.405	1.410
B-8007-C*pH9.5*a1	*25C*	0.937	.932
B-8007-C*pH9.5*a2	*25C*	0.937	.934
B-8007-C*pH9.5*b1	*25C*	0.937	.936
B-8007-C*pH9.5*b2	*25C*	0.937	.939
B-8007*pH2.00*a	**25B**	0.187	.192
B-8007*pH2.00*b	**25B**	0.187	.188
B-8007*pH2.25*a	**25B**	0.181	.185
B-8007*pH2.25*b	**25B**	0.181	.181
B-8007*pH2.50*a	**25B**	0.176	.175
B-8007*pH2.50*b	**25B**	0.176	.176
B-8007*pH2.75*a	**25B**	0.170	.168
B-8007*pH2.75*b	**25B**	0.170	.173
B-8007*pH3.00*a	*25C*	1.639	1.633
B-8007*pH3.00*b	*25C*	1.639	1.645
B-8007*pH3.25*a	*25C*	1.546	1.546
B-8007*pH3.25*b	*25C*	1.546	1.548
B-8007*pH3.50*a	*25C*	1.499	1.496
B-8007*pH3.50*b	*25C*	1.499	1.494
B-8007*pH3.75*a	*25C*	1.405	1.403
B-8007*pH3.75*b	*25C*	1.405	1.404
B-8007*pH4.00*a	*25C*	0.820	.821
B-8007*pH4.00*b	*25C*	0.820	.817
B-8007*pH4.25*a	*25C*	0.586	.584
B-8007*pH4.25*b	*25C*	0.586	.585

Identification	Spike#	Wt (g)	Actual wt(g)
B-8007*pH4.50*a	*25C*	0.422	.423
B-8007*pH4.50*b	*25C*	0.422	.424
B-8007*pH4.75*a	*25C*	0.187	.187
B-8007*pH4.75*b	*25C*	0.187	.191
B-8007*pH5.00*a	*25C*	0.117	.118
B-8007*pH5.00*b	*25C*	0.117	.116
B-8007*pH5.25*a	*25C*	0.117	.117
B-8007*pH5.25*b	*25C*	0.117	.120
B-8007*pH5.50*a	*25C*	0.117	.116
B-8007*pH5.50*b	*25C*	0.117	.119
B-8007*pH5.75*a	*25C*	0.117	.119
B-8007*pH5.75*b	*25C*	0.117	.116
B-8007*pH6.00*a	*25C*	0.117	.118
B-8007*pH6.00*b	*25C*	0.117	.121
B-8007*pH6.25*a	*25C*	0.117	.119
B-8007*pH6.25*b	*25C*	0.117	.119
B-8007*pH6.50*a	*25C*	0.117	.115
B-8007*pH6.50*b	*25C*	0.117	.119
B-8007*pH6.75*a	*25C*	0.117	.116
B-8007*pH6.75*b	*25C*	0.117	.119
B-8007*pH7.00*a	*25C*	0.117	.118
B-8007*pH7.00*b	*25C*	0.117	.120
B-8007*pH7.25*a	*25C*	0.117	.118
B-8007*pH7.25*b	*25C*	0.117	.119
B-8007*pH7.50*a	*25C*	0.234	.235
B-8007*pH7.50*b	*25C*	0.234	.237
B-8007*pH7.75*a	*25C*	0.468	.468
B-8007*pH7.75*b	*25C*	0.468	.469
B-8007*pH8.00*a	*25C*	0.703	.701
B-8007*pH8.00*b	*25C*	0.703	.709
B-8007*pH8.25*a	*25C*	1.054	1.055
B-8007*pH8.25*b	*25C*	1.054	1.061
B-8007*pH8.50*a	**25B**	0.187	.191
B-8007*pH8.50*b	**25B**	0.187	.192
B-8007*pH8.75*a	**25B**	0.199	.201
B-8007*pH8.75*b	**25B**	0.199	.200
B-8007*pH9.00*a	**25B**	0.211	.207
B-8007*pH9.00*b	**25B**	0.211	.214

25B is 204.88 pCi/g
25C is 20.477 pCi/g

		Actual Sum
SUM 25B	**5.524**	5.566 g
SUM 25C	*28.504*	32.856 g

1/27/93

1/29/93 0900hr GP

Fe carrier was added to B-8005 samples.
Step A-4 of procedure
To each sample 1ml of a 24.3 mg/ml Fe
solution was added with an Eppendorf
pipet

1510 hr GP

Fe carrier was added to B-8007 samples
1ml of 24.3 mg/ml Fe solution

2/1/93 0815hr GP

Samples B-8005*pH2.00*a + B-8005*pH2.00*b
were processed and analyzed by α-spectrometry.

2/1/93 1400hr GP

8N HCl was prepared by placing
~678 ml of Nanopure H_2O in a 2L
volumetric flask and adding ~1322 ml
of conc. (~12.1N) HCl (lot #905802).
The solution was mixed and transferred
to an acid bottle kept below the
radioisotope fume hood.

2/3/93 GP Samples B-8005* pH 2.25*a + B-8005* pH 2.50 *a
were processed and analyzed by
α-spectrometry.

2/4/93 GP Samples B-8005* pH 2.75*a, B-8005 *pH 3.00 *a,
B-8005* pH 3.25*a, and B-8005 *pH 3.50*a
were processed + analyzed by
α-spectrometry.

2/8/93 GP Samples B-8005 *pH 4.00*a, B-8005 * pH 5.00*a,
B-8005 * pH 6.00*a, B-8005 * pH 7.00*a,
B-8005 * pH 8.00*a, + B-8005 * pH 9.00*a,
were processed + analyzed by
α-spectrometry.

2/11/93 GP Prepared additional 0.4M TTA in Benzene
soln for α-spectrometry. (lot 904728)
Placed 8.71 g of Thenoyl trifluoroacetone
in a 100 ml volumetric flask. 100 ml
of benzene (lot 920979) was added
to flask. Soln was shaken +
labeled. Flask was covered in
foil to avoid light.

U-233 Liquid Scintillation Analysis Procedure

BACKGROUND:

Liquid Scintillation Analysis (LSA) is a method usually used to measure beta-emitting isotopes and is not generally used to measure alpha emitting isotopes. However, LSA is perfect for a one alpha-emitting isotope solution (^{233}U) since it counts all alpha particles with 100% efficiency, but it has terrible energy resolution. Thus for samples containing multiple alpha-emitting isotopes LSA can not be used quantitatively. The concept behind liquid scintillation counting is straight forward. Energy associated with each decay event is transfered to scintillators (solute) carried in an organic matrix (solvent). The scintillator then emits a pulse of light which is counted by a photomultiplier tube. More information on LSA is provided in the attached reprint of the Packard Liquid Scintillation Analysis Science and Technology publication. Sample preparation is easy and the amount of sample solution that can be added to a specific cocktail is limited. The specifications of the Ultima Gold cocktail that will be used in these experiments is also given in the attachment. Familiarize yourself with operation of the Repipetor which will be used to add cocktail to each sample (instruction manual is kept next to LSA on shelf unit). **Disposal of the cocktail solution will have to be rigorously documented.**

In an effort to balance precision versus counting time, different levels of precision will be used dependent upon the concentration of the the U solution used in the experiment (5, 50 or 500 ppb U). These experiments should be counted initially with the following protocols. Protocol # 4 is 1% 2 sigma counting error to be used for 500 ppb experiments. Protocol # 5 is 3% 2 sigma counting error to be used for 50 ppb experiments. Protocol # 6 is 5% 2 sigma counting error to be used for 5 ppb experiments. These protocols will allow that all samples in a single experiment to be counted to their level of precision in 1 - 3 days of counting time. Samples can be recounted later using higher precision protocols (#4 and #5) as time permits.

Also attached are two sample spectrum and a sample output which show what a typical ^{233}U spectrum should look like. In your analysis, spectrum will not need to be acquired (printed out). The protocols are set up to give the CPM in each region of interest and the associated% 2 sigma error. In your manipulations of data we are interested is CPM in Region B (CPMB). Note that the CPM=DPM, since the alpha emissions are counted at 100% efficiency. It should be noted that all radiological safety precautions prescribed in URANIUM SORPTION EXPERIMENTS UTILIZING U-233: RADIOLOGICAL SAFETY CONSIDERATIONS: WRITTEN BY: B. W. LESLIE: AUGUST 21,1992 will be followed (*eg.* record keeping, safety, surface contamination surveys, and solid waste disposal).

Waste Disposal:

1. Disposal of liquid scintillation cocktails will be via the sanitary sewer, however, rigorous documentation will be required to ensure that disposal complies with

the pertinent regulations. The release limits for solutions containing ^{233}U and the pertinent rules for disposal of such solutions can be found in TRC 21.303 and TRC Appendix 21-A and B (Note 1). Since the Ultima Gold scintillation cocktail is biodegradable, and the ^{233}U and cocktail are soluble and dispersible, the cocktails can be disposed of down the sewer, only if sufficient dilution is maintained prior to disposal down the drain. The maximum permissible concentration of ^{233}U that can be placed down the drain is **0.9 nCi/ml**. The directions for disposal of Ultima Gold scintillation cocktail solutions requires that the ratio of running **water to flow of solution exceed 25:1** and it is suggested that the **cocktail solutions be first dispersed in a large volume of water.**

2. The most concentrated cocktail (500 ppb ^{233}U) would be on the order of 3 nCi/ml, and would require only a 1:5 dilution to be below the limits for radioactivity. However, each cocktail solution will be dilute to ≥ 250 ml prior to dumping down drain. In addition the faucet should be running when the diluted cocktail is allowed to flow down the drain. This will allow for adequate dispersal of the organic solvent. The date of disposal for each cocktail solution will be recorded on a copy of the data output. Upon completion of disposal of all solution for an experiment each investigator will be responsible for presenting the disposal record to the Radiation Safety Point of Contact (Bret Leslie). This record will then be given to the Institute Radiation Safety Officer and become part of the permanent files. Please document disposal method and samples disposed of in the appropriate Scientific Notebook.

Analytical Procedure:

A. Making the sample cocktail

1. Label lids of 7 ml glass LSA vials with sample identification using thin pen marker.

2. Weigh the labeled vial on the Mettler AE240 balance and record the weight.

3. Remove sample aliquot from one row of 50 ml centrifuge tubes in a rack at a time. Add 1 ml of sample to the appropriate vial using Eppendorf fixed volume pipet and disposable tips. Cap vial. Repeat procedure for each centrifuge tube in a row. Repeat procedure until all samples have been completed

4. Reweigh each vial containing sample aliquot and record weight.

5. Carefully add 5 ml of the Ultima Gold liquid scintillation cocktail to each sample vial using the Repipetor. The Repipetor and the Ultima Gold are stored under the fumehood next to the balance.

6. Prepare a counting vial with 1 ml of a 0.02 N HNO_3 solution and 5 ml of the Ultima Gold cocktail. This is the background vial.

7. After all the vials have had cocktail added then thoroughly homogenize samples until a single-phase solution is obtained (it may still contain bubbles, but should not contain schlieren).

B. Counting

1. Place each vial into one of the 7 ml counting racks in some order, allowing for one blank space at the front of the first rack for the background vial. The number of the rack should be on the right side of the counter. Fill each rack and record the position of the sample in the rack.

2. Place the appropriate protocol flag into the left side of the rack. Push the flag to the left on it and the SNC rack. Place the SNC rack in front of your samples.

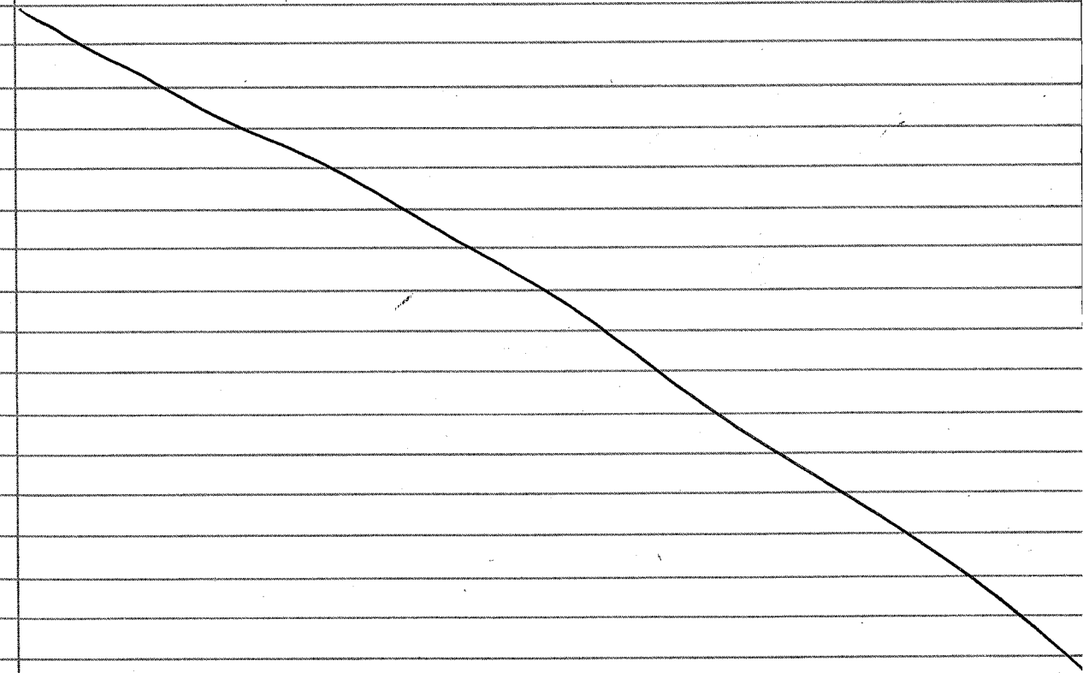
3. Close the lid of the counter and allow the samples to sit overnight. This will allow for the sample's luminescence associated with the exposure of each vial to light in the room to dissipate and allow for any bubbles which formed during homogenization to dissolve.

4. The next morning press F2. Collect data at end of run. Note that the sample DPM recorded as CPMB has already been corrected for background. Make a copy of the data for waste disposal purposes (see above). Do not dispose of cocktail until approval from the primary investigator and the RSPOC has been received.

C. Calculations

1. Convert CPMB of sample to dpm/g, remember this includes a correction for the mass added to LSA vial, and a correction for the amounts of acid and sample originally placed into the 50 ml centrifuge tubes.

2. Convert ^{233}U dpm/g to ^{233}U atoms/g to U g/g. Plot away!



2/26/93 gp

0830hrs Experiment B-8006 samples were prepared for LSA analysis following analytical procedure on p 40.

1630hrs LSA data collection was begun for experiment B-8006.

Following pages contain information concerning LSA analysis of B-8006 samples

B8006 LSA sample weight

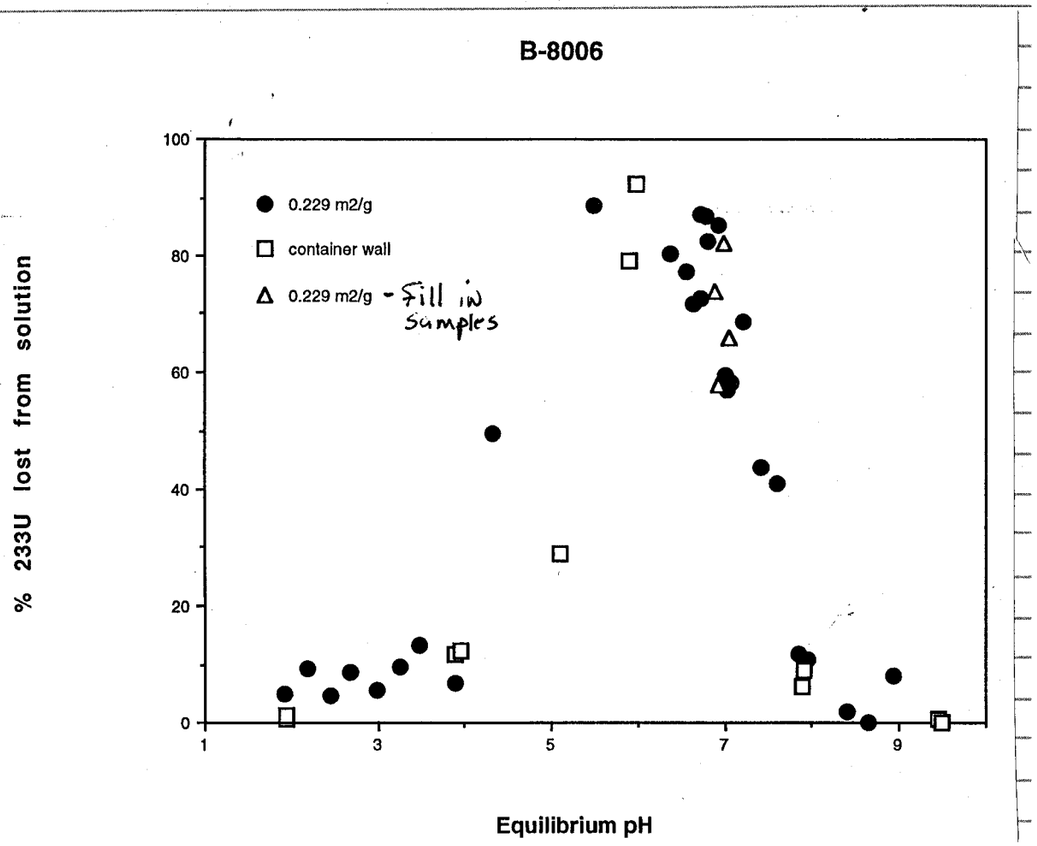
Identification	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U ppb
B-8006*pH2.00*a	7.349	8.355	12	2	1.006	55.89	4.88
B-8006*pH2.00*b	7.356	8.357	12	3	1.001	52.383	4.63
B-8006*pH2.25*a	7.325	8.321	12	4	0.996	50.519	4.49
B-8006*pH2.25*b	7.326	8.325	12	5	0.999	51.709	4.59
B-8006*pH2.50*a	7.407	8.404	12	6	0.997	51.491	4.59
B-8006*pH2.50*b	7.415	8.414	12	7	0.999	55.759	4.94
B-8006*pH2.75*a	7.388	8.386	12	8	0.998	49.581	4.41
B-8006*pH2.75*b	7.311	8.31	12	9	0.999	53.435	4.74
B-8006*pH3.00*a	7.394	8.393	12	10	0.999	49.913	4.45
B-8006*pH3.00*b	7.349	8.342	12	11	0.993	55.753	4.99
B-8006*pH3.25*a	7.442	8.438	12	12	0.996	50.682	4.52
B-8006*pH3.25*b	7.375	8.371	12	13	0.996	50.658	4.52
B-8006*pH3.50*a	7.333	8.334	12	14	1.001	50.197	4.47
B-8006*pH3.50*b	7.303	8.298	12	15	0.995	47.316	4.22
B-8006*pH3.75*a	7.386	8.383	12	16	0.997	53.221	4.76
B-8006*pH3.75*b	7.377	8.375	12	17	0.998	51.342	4.57
B-8006*pH4.00*a	7.303	8.302	12	18	0.999	28.637	2.55
B-8006*pH4.00*b	7.354	8.352	13	1	0.998	27.904	2.49
B-8006*pH4.25*a	7.363	8.354	13	2	0.991	6.414	0.58
B-8006*pH4.25*b	7.433	8.429	13	3	0.996	6.478	0.58
B-8006*pH4.50*a	7.34	8.34	13	4	1.000	11.015	0.98
B-8006*pH4.50*b	7.424	8.42	13	5	0.996	10.918	0.97
B-8006*pH4.75*a	7.396	8.396	13	6	1.000	13.032	1.17
B-8006*pH4.75*b	7.399	8.395	13	7	0.996	12.543	1.12
B-8006*pH5.00*a	7.416	8.415	13	8	0.999	15.49	1.38
B-8006*pH5.00*b	7.445	8.442	13	9	0.997	16.063	1.43
B-8006*pH5.25*a	7.39	8.386	13	10	0.996	7.613	0.68
B-8006*pH5.25*b	7.425	8.419	13	11	0.994	6.947	0.62
B-8006*pH5.50*a	7.35	8.349	13	12	0.999	8.166	0.73
B-8006*pH5.50*b	7.44	8.438	13	13	0.998	8.178	0.73
B-8006*pH5.75*a	7.41	8.412	13	14	1.002	15.019	1.34
B-8006*pH5.75*b	7.312	8.307	13	15	0.995	15.614	1.4
B-8006*pH6.00*a	7.341	8.338	13	16	0.997	7.851	0.7
B-8006*pH6.00*b	7.439	8.433	13	17	0.994	7.079	0.63
B-8006*pH6.25*a	7.373	8.369	13	18	0.996	8.428	0.76
B-8006*pH6.25*b	7.38	8.376	14	1	0.996	8.016	0.72
B-8006*pH6.50*a	7.359	8.356	14	2	0.997	22.984	2.06
B-8006*pH6.50*b	7.382	8.38	14	3	0.998	22.261	1.99
B-8006*pH6.75*a	7.343	8.341	14	4	0.998	24.005	2.15
B-8006*pH6.75*b	7.451	8.447	14	5	0.996	22.894	2.05
B-8006*pH7.00*a	7.382	8.378	14	6	0.996	24.488	2.19
B-8006*pH7.00*b	7.35	8.348	14	7	0.998	23.547	2.1
B-8006*pH7.25*a	7.406	8.402	14	8	0.996	17.729	1.6
B-8006*pH7.25*b	7.417	8.413	14	9	0.996	17.091	1.53

B8006 LSA sample weight

Identification	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U PPB
B-8006*pH7.50*a	7.37	8.367	14	10	0.997	32.062	2.88
B-8006*pH7.50*b	7.437	8.434	14	11	0.997	30.59	2.74
B-8006*pH7.75*a	7.425	8.423	14	12	0.998	33.491	3
B-8006*pH7.75*b	7.399	8.398	14	13	0.999	32.686	2.92
B-8006*pH8.00*a	7.344	8.342	14	14	0.998	49.132	4.38
B-8006*pH8.00*b	7.373	8.37	14	15	0.997	50.019	4.46
B-8006*pH8.25*a	7.317	8.311	14	16	0.994	49.404	4.44
B-8006*pH8.25*b	7.385	8.384	14	17	0.999	50.232	4.47
B-8006*pH8.50*a	7.415	8.417	14	18	1.002	56.021	4.98
B-8006*pH8.50*b	7.459	8.461	15	1	1.002	54.501	4.85
B-8006*pH8.75*a	7.422	8.422	15	2	1.000	56.44	5.06
B-8006*pH8.75*b	7.417	8.416	15	3	0.999	55.329	4.95
B-8006*pH9.00*a	7.443	8.441	15	4	0.998	52.646	4.71
B-8006*pH9.00*b	7.388	8.386	15	5	0.998	50.411	4.49
B-8006-C*pH2*a1	7.382	8.381	15	6	0.999	56.656	5.03
B-8006-C*pH2*a2	7.474	8.471	15	7	0.997	54.423	4.85
B-8006-C*pH2*b1	7.355	8.354	15	8	0.999	57.001	5.07
B-8006-C*pH2*b2	7.349	8.345	15	9	0.996	54.548	4.86
B-8006-C*pH4*a1	7.365	8.363	15	10	0.998	51.656	4.62
B-8006-C*pH4*a2	7.312	8.308	15	11	0.996	46.456	4.16
B-8006-C*pH4*b1	7.384	8.381	15	12	0.997	49.39	4.41
B-8006-C*pH4*b2	7.334	8.333	15	13	0.999	49.494	4.42
B-8006-C*pH6*a1	7.315	8.314	15	14	0.999	4.464	0.4
B-8006-C*pH6*a2	7.352	8.349	15	15	0.997	4.21	0.38
B-8006-C*pH6*b1	7.402	8.396	15	16	0.994	11.477	1.02
B-8006-C*pH6*b2	7.42	8.418	15	17	0.998	11.898	1.06
B-8006-C*pH8*a1	7.369	8.365	15	18	0.996	51.754	4.61
B-8006-C*pH8*a2	7.363	8.359	16	1	0.996	53.686	4.78
B-8006-C*pH8*b1	7.386	8.383	16	2	0.997	50.573	4.51
B-8006-C*pH8*b2	7.383	8.381	16	3	0.998	51.324	4.58
B-8006-C*pH9.5*a1	7.38	8.379	16	4	0.999	54.563	4.88
B-8006-C*pH9.5*a2	7.376	8.372	16	5	0.996	56.656	5.07
B-8006-C*pH9.5*b1	7.38	8.379	16	6	0.999	57.411	5.11
B-8006-C*pH9.5*b2	7.357	8.352	16	7	0.995	56.559	5.06
B-8006*IUa	7.358	8.355	16	8	0.997	39.133	3.5
B-8006*IUb	7.355	8.35	16	9	0.995	40.496	3.6
B-8006*pH5.25A*a	7.382	8.38	16	10	0.998	15.244	1.35
B-8006*pH5.25A*b	7.447	8.448	16	11	1.001	14.174	1.25
B-8006*pH5.50A*a	7.414	8.413	16	12	0.999	24.333	2.15
B-8006*pH5.50A*b	7.352	8.347	16	13	0.995	23.085	2.06
B-8006*pH6.25A*a	7.346	8.339	16	14	0.993	9.924	0.89
B-8006*pH6.25A*b	7.383	8.382	16	15	0.999	10.108	0.9
B-8006*pH6.50A*a	7.374	8.371	16	16	0.997	18.851	1.68
B-8006*pH6.50A*b	7.418	8.417	16	17	0.999	19.418	1.73

Plot of B-8006 Data -

Equilibrium pH vs % ^{233}U lost from solution



3/5/93 GP
1400hrs Experiment B-8007 samples were prepared for LSA analysis following analytical procedure on p 40.

pH's of initial B-8007 samples were remeasured. Samples in 125ml PP bottles and 1 ml samples were taken.

3/8/93 GP
1000hrs. LSA data collection was begun for B-8007.

Following pages contain information concerning LSA analysis for B-8007 samples.

B8007 LSA sample weight

Identification	Eq pH	Vial wt g	Sam+vial wt g	RACK	VIAL	Sample wt	CPMB	U ppb
B-8007*pH2.00*a	1.91	7.3307	8.3294	6	2	0.9987	125.356	5.62
B-8007*pH2.00*b	1.91	7.3093	8.3093	6	3	1	120.537	5.39
B-8007*pH2.25*a	2.12	7.3581	8.363	6	4	1.0049	130.117	5.79
B-8007*pH2.25*b	2.12	7.3191	8.3251	6	5	1.006	130.895	5.82
B-8007*pH2.50*a	2.35	7.4113	8.417	6	6	1.0057	123.179	5.48
B-8007*pH2.50*b	2.35	7.3696	8.3738	6	7	1.0042	121.785	5.43
B-8007*pH2.75*a	2.65	7.3855	8.3911	6	8	1.0056	123.237	5.48
B-8007*pH2.75*b	2.65	7.3995	8.4036	6	9	1.0041	125.837	5.61
B-8007*pH3.00*a	2.9	7.4644	8.4675	6	10	1.0031	116.847	5.21
B-8007*pH3.00*b	2.9	7.413	8.4154	6	11	1.0024	117.598	5.25
B-8007*pH3.25*a	3.26	7.3815	8.3839	6	12	1.0024	110.11	4.91
B-8007*pH3.25*b	3.26	7.3233	8.325	6	13	1.0017	111.392	4.97
B-8007*pH3.50*a	3.45	7.3811	8.3853	6	14	1.0042	107.974	4.81
B-8007*pH3.50*b	3.45	7.3843	8.3855	6	15	1.0012	106.102	4.74
B-8007*pH3.75*a	3.65	7.3902	8.3927	6	16	1.0025	121.128	5.41
B-8007*pH3.75*b	3.65	7.446	8.4479	6	17	1.0019	120.098	5.36
B-8007*pH4.00*a	3.9	7.4154	8.4169	6	18	1.0015	95.396	4.26
B-8007*pH4.00*b	3.9	7.3176	8.3196	7	1	1.002	97.999	4.38
B-8007*pH4.25*a	4.16	7.4397	8.4498	7	2	1.0101	73.379	3.25
B-8007*pH4.25*b	4.16	7.396	8.4014	7	3	1.0054	70.987	3.16
B-8007*pH4.50*a	4.56	7.4173	8.4281	7	4	1.0108	49.921	2.21
B-8007*pH4.50*b	4.56	7.3942	8.3993	7	5	1.0051	50.913	2.27
B-8007*pH4.75*a	4.79	7.3388	8.3513	7	6	1.0125	27.826	1.23
B-8007*pH4.75*b	4.79	7.3132	8.3176	7	7	1.0044	27.995	1.25
B-8007*pH5.00*a	5.32	7.396	8.4054	7	8	1.0094	26.928	1.19
B-8007*pH5.00*b	5.32	7.3937	8.3983	7	9	1.0046	26.431	1.18
B-8007*pH5.25*a	5.76	7.4015	8.4127	7	10	1.0112	28.427	1.26
B-8007*pH5.25*b	5.76	7.3846	8.3891	7	11	1.0045	28.022	1.25
B-8007*pH5.50*a	5.27	7.3858	8.3994	7	12	1.0136	13.419	0.59
B-8007*pH5.50*b	5.27	7.3476	8.3559	7	13	1.0083	13.791	0.61
B-8007*pH5.75*a	6.1	7.4456	8.4599	7	14	1.0143	18.761	0.83
B-8007*pH5.75*b	6.1	7.2909	8.2979	7	15	1.007	19.394	0.86
B-8007*pH6.00*a	7.17	7.3939	8.4059	7	16	1.012	16.452	0.73
B-8007*pH6.00*b	6.17	7.3955	8.3993	7	17	1.0038	16.612	0.74
B-8007*pH6.25*a	6.42	7.4626	8.4732	7	18	1.0106	25.495	1.13
B-8007*pH6.25*b	6.42	7.4301	8.4338	8	1	1.0037	25.836	1.15
B-8007*pH6.50*a	6.67	7.4094	8.4148	8	2	1.0054	22.63	1.01
B-8007*pH6.50*b	6.67	7.4263	8.4288	8	3	1.0025	22.798	1.02
B-8007*pH6.75*a	6.84	7.3551	8.3628	8	4	1.0077	25.859	1.15
B-8007*pH6.75*b	6.84	7.4754	8.4786	8	5	1.0032	26.345	1.17
B-8007*pH7.00*a	6.87	7.4148	8.4204	8	6	1.0056	39.221	1.74
B-8007*pH7.00*b	6.87	7.319	8.3217	8	7	1.0027	38.353	1.71
B-8007*pH7.25*a	7.42	7.3811	8.3984	8	8	1.0173	34.508	1.52
B-8007*pH7.25*b	7.42	7.3338	8.3416	8	9	1.0078	35.951	1.6
B-8007*pH7.50*a	7.29	7.377	8.3845	8	10	1.0075	64.371	2.86

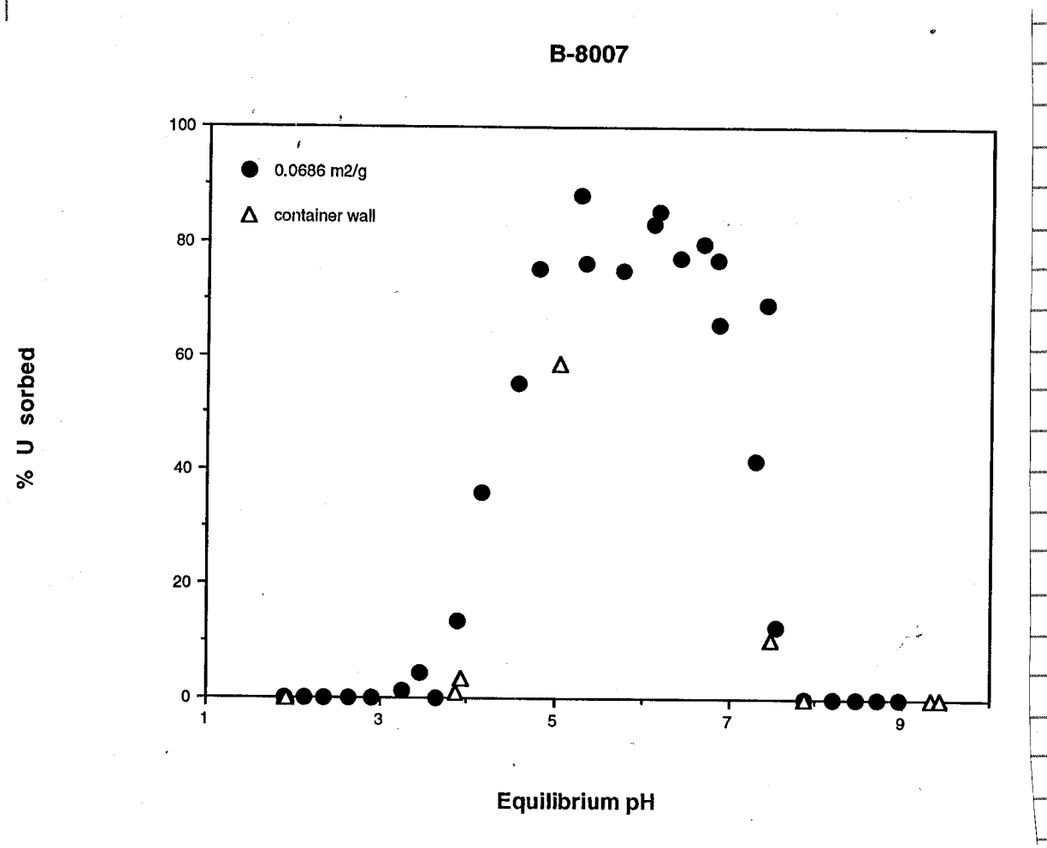
Calculations verified on pp. 143-144 of 6C-11 (controlled Copy #081)

B8007 LSA sample weight

B-8007*pH7.50*b	7.29	7.3661	8.3701	8	11	1.004	66.708	2.97
B-8007*pH7.75*a	7.54	7.3916	8.4031	8	12	1.0115	97.807	4.33
B-8007*pH7.75*b	7.54	7.3399	8.3443	8	13	1.0044	99.222	4.42
B-8007*pH8.00*a	7.86	7.4124	8.4177	8	14	1.0053	124.708	5.55
B-8007*pH8.00*b	7.86	7.4097	8.4119	8	15	1.0022	125.504	5.6
B-8007*pH8.25*a	8.19	7.428	8.4379	8	16	1.0099	127.81	5.66
B-8007*pH8.25*b	8.19	7.3151	8.3196	8	17	1.0045	140.28	6.25
B-8007*pH8.50*a	8.47	7.3585	8.3638	8	18	1.0053	142.968	6.36
B-8007*pH8.50*b	8.47	7.3483	8.3529	9	1	1.0046	136.092	6.06
B-8007*pH8.75*a	8.7	7.4787	8.4857	9	2	1.007	129.024	5.73
B-8007*pH8.75*b	8.7	7.4165	8.4195	9	3	1.003	136.732	6.1
B-8007*pH9.00*a	8.96	7.384	8.3914	9	4	1.0074	132.564	5.89
B-8007*pH9.00*b	8.96	7.3316	8.3354	9	5	1.0038	149.738	6.67
B-8007-C*pH2*a1	1.92	7.3705	8.3766	9	6	1.0061	130.895	5.82
B-8007-C*pH2*a2	1.92	7.3576	8.3581	9	7	1.0005	135.339	6.05
B-8007-C*pH2*b1	1.91	7.4232	8.4263	9	8	1.0031	131.935	5.88
B-8007-C*pH2*b2	1.91	7.3596	8.3614	9	9	1.0018	130.811	5.84
B-8007-C*pH4*a1	3.93	7.3635	8.3685	9	10	1.005	107.919	4.77
B-8007-C*pH4*a2	3.93	7.3884	8.3885	9	11	1.0001	109.239	4.89
B-8007-C*pH4*b1	3.87	7.3522	8.3559	9	12	1.0037	110.27	4.91
B-8007-C*pH4*b2	3.87	7.3281	8.3283	9	13	1.0002	111.555	4.99
B-8007-C*pH6*a1								
B-8007-C*pH6*a2								
B-8007-C*pH6*b1								
B-8007-C*pH6*b2								
B-8007-C*pH8*a1	7.87	7.3903	8.3959	9	14	1.0056	129.676	5.77
B-8007-C*pH8*a2	7.87	7.3341	8.3374	9	15	1.0033	130.867	5.84
B-8007-C*pH8*b1	7.48	7.3685	8.3757	9	16	1.0072	100.006	4.44
B-8007-C*pH8*b2	7.48	7.3572	8.359	9	17	1.0018	101.351	4.52
B-8007-C*pH9.5*a1	9.32	7.339	8.3459	9	18	1.0069	126.962	5.64
B-8007-C*pH9.5*a2	9.32	7.4004	8.4047	10	1	1.0043	131.175	5.84
B-8007-C*pH9.5*b1	9.43	7.346	8.3527	10	2	1.0067	142.175	6.32
B-8007-C*pH9.5*b2	9.43	7.3746	8.3809	10	3	1.0063	134.21	5.97
B-8007*IUa	5.03	7.3945	8.4021	10	4	1.0076	45.295	2.01
B-8007*IUb	5.03	7.4463	8.4468	10	5	1.0005	47.781	2.14
0.1 N NaNO3*a	5.61	7.3714	8.3833	10	6	1.0119	0.159	0.01
0.1 N NaNO3*b	5.61	7.3112	8.3165	10	7	1.0053	0.259	0.01

Hobi of B-8007 data.

Equilibrium pH vs % U lost from solution



3/12/93 JP Because many B-8007 samples had U concentrations above 5 ppb, some samples were rerun to check the LSA analysis.
B-8007 samples were not acidified before analysis by LSA. For selected B-8007 samples for this analysis, a 0.5 ml sample was taken and 0.5 ml of 0.02 N HNO_3 was added before analysis.

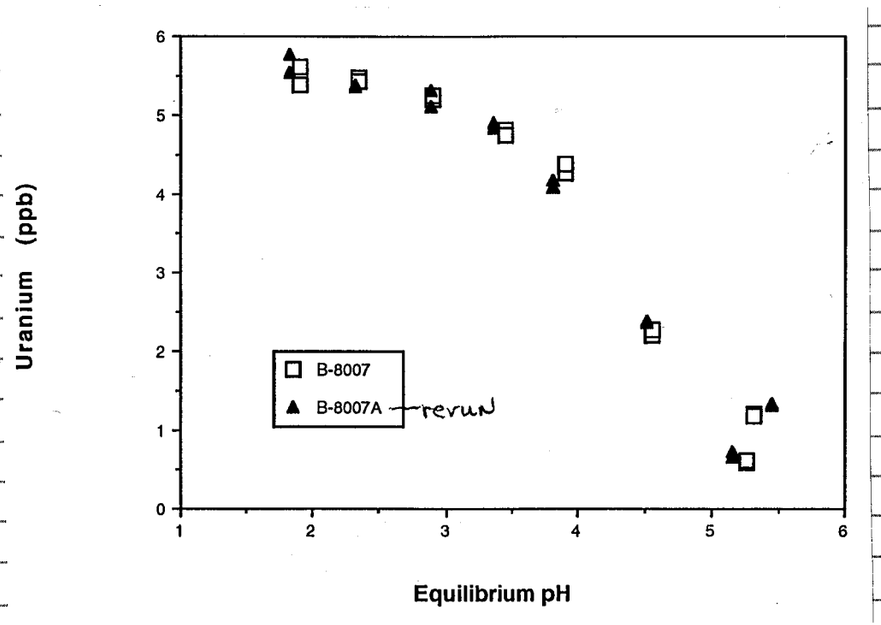
1400hrs pH's of rerun samples were remeasured and samples were prepared following procedure on p40 for LSA analysis.

1600hrs Samples were placed in LSA. For data collection.

Following pages contain data for LSA analysis of B-8007 rerun samples.

Identification	Eq pH	Vial wt g	Sam+vial wt g	RACK	VIAL	Sample wt	CPMB	U ppb
B-8007*pH2.00*a	1.83	7.4429	7.9499	2	2	0.507	65.481	5.78
B-8007*pH2.00*b	1.83	7.36674	7.8733	2	3	0.50656	62.802	5.55
B-8007*pH2.50*a	2.32	7.404	7.907	2	4	0.503	60.515	5.38
B-8007*pH2.50*b	2.32	7.3335	7.8384	2	5	0.5049	60.551	5.37
B-8007*pH3.00*a	2.89	7.3887	7.8928	2	6	0.5041	59.975	5.32
B-8007*pH3.00*b	2.89	7.3631	7.868	2	7	0.5049	57.787	5.12
B-8007*pH3.50*a	3.35	7.3561	7.8596	2	8	0.5035	55.289	4.91
B-8007*pH3.50*b	3.35	7.3415	7.8453	2	9	0.5038	54.533	4.84
B-8007*pH4.00*a	3.81	7.3757	7.8771	2	10	0.5014	45.754	4.08
B-8007*pH4.00*b	3.81	7.3314	7.835	2	11	0.5036	47.099	4.18
B-8007*pH4.50*a	4.51	7.3858	7.889	2	12	0.5032	26.588	2.36
B-8007*pH4.50*b	4.51	7.3675	7.8716	2	13	0.5041	26.956	2.39
B-8007*pH5.00*a	5.45	7.3936	7.8985	2	14	0.5049	14.772	1.31
B-8007*pH5.00*b	5.45	7.3627	7.8677	2	15	0.505	15.147	1.34
B-8007*pH5.50*a	5.16	7.3648	7.8678	2	16	0.503	7.396	0.66
B-8007*pH5.50*b	5.16	7.3891	7.8929	2	17	0.5038	8.082	0.72

Plot of Uranium concentration vs pH comparing original and rerun B-8007 data.



3/11/93
JP

1300 hrs

FILTRATION TEST OF U-BEARING SOLUTIONS

OBJECTIVE: to determine the amount of uranium lost to filter during sampling of U-bearing solutions for uranium sorption experiments

EQUIPMENT: Liquid Scintillation Analyzer
ORION ph/mV/ISE/ C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance

SUPPLIES:
- pH buffers (pH= 4,7)
- 2 125 ml PP bottles
- LSA vials 7ml
- NaHCO3 (lot 897186A)
- 1 Eppendorf pipet (for taking samples) and blue tips (23A)
- 500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike
- 0.1 m $NaNO_3$ stock solution
- 0.02 m HNO3 solution
- 2 Dynagard filters, 0.2micron, 3.9cm2 surface area
- 2 Dynagard filters, 0.2micron, 0.8cm2 surface area

PROCEDURE:

1) Into each of two 125 ml PP bottles labelled FL*pH6.00 and FL*pH7.00 tare 20 g of a 500 ppb U stock solution and 80 g of a 0.1 M NaNO3 stock solution.

2) Measure and record the pH of each sample (see FL DATA TABLE).

3) Add NaHCO3 to each sample until target pH (6.00 or 7.00) is achieved. Record this pH.

4) When target pH is reached immediately take from each bottle:

a) Two 0.5 ml aliqouts with an Eppendorf pipet and place into prelabeled and preweighed LSA vials (FL*pH6.00*a; FL*pH6.00*b; FL*pH7.00*a; FL*pH7.00*b).

b) Two 0.5 ml aliqouts with an Eppendorf pipet after filtration thru a 0.2 micron filter with a surface area of 3.9 cm2 and place into prelabeled and preweighed LSA vials (FL*pH6.00*c; FL*pH6.00*d; FL*pH7.00*c; FL*pH7.00*d).

c) Two 0.5 ml aliqouts with an Eppendorf pipet after filtration thru a 0.2 micron filter with a surface area of 0.8 cm2 and place into prelabeled and preweighed LSA vials (FL*pH6.00*e; FL*pH6.00*f; FL*pH7.00*e; FL*pH7.00*f).

5) Reweigh LSA sample vials + sample.

6) Add 0.5 ml of 0.02 M HNO3 to each LSA sample vial.

7) Analyze samples by LSA following procedure on p. 40.

3/11/93 1500hrs GP

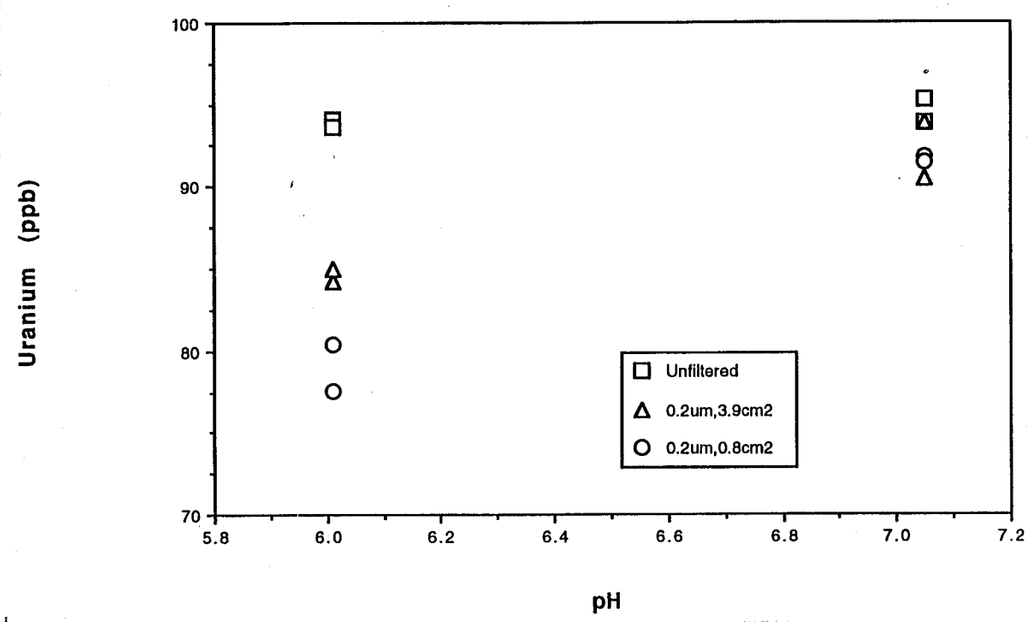
Experiment FL samples were prepared for LSA analysis following procedure on p 40.

Below is information on LSA analysis for experiment FL.

Identification	pH	Targ pH	Vial wt g	S+v wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
FL*pH6.00*a	3.84	6.01	7.391	7.9027	1	2	0.5117	1076.35	94.114
FL*pH6.00*b	3.84	6.01	7.3587	7.8639	1	3	0.5052	1056.98	93.609
FL*pH6.00*c	3.84	6.01	7.361	7.8653	1	4	0.5043	949.439	84.235
FL*pH6.00*d	3.84	6.01	7.428	7.9305	1	5	0.5025	957.968	84.992
FL*pH6.00*e	3.84	6.01	7.466	7.9702	1	6	0.5042	873.703	77.531
FL*pH6.00*f	3.84	6.01	7.3818	7.8856	1	7	0.5038	905.548	80.421
FL*pH7.00*a	3.88	7.05	7.3763	7.8792	1	8	0.5029	1070.65	95.254
FL*pH7.00*b	3.88	7.05	7.3655	7.8685	1	9	0.503	1055.79	93.913
FL*pH7.00*c	3.88	7.05	7.4299	7.9324	1	10	0.5025	1054.7	93.909
FL*pH7.00*d	3.88	7.05	7.346	7.8504	1	11	0.5044	1019.74	90.455
FL*pH7.00*e	3.88	7.05	7.4476	7.9526	1	12	0.505	1036.14	91.8
FL*pH7.00*f	3.88	7.05	7.3792	7.8836	1	13	0.5044	1030.47	91.406

Plot of FL Data

Uranium concentration vs pH



3/15/93 gp

3/15/93 gp

1355hrs

TIP LOSS TEST OF U-BEARING SOLUTIONS

OBJECTIVE: to determine the amount of uranium lost to Eppendorf tips during sampling of U-bearing solutions for uranium sorption experiments

EQUIPMENT: Liquid Scintillation Analyzer
ORION ph/mV/ISE/ C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance

SUPPLIES:
- pH buffers (pH= 2,4,7)
- 4 125 ml PP bottles
- LSA vials 7ml
- $NaHCO_3$ (lot 897186A)
- HNO_3 stock solutions (1.0, 0.1, 0.01, and 0.02 N)
- 1 Eppendorf pipet (for taking samples) and blue tips (23A)
- 500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike
- 0.1 m $NaNO_3$ stock solution

PROCEDURE:

1) Prepare 100 ppb ^{233}U solutions. Into each of four 125 ml PP bottles labelled TL*pH2.00, TL*pH3.00, TL*pH5.00, and TL*pH6.00 tare 20 g of a 500 ppb ^{233}U stock solution and 80 g of a 0.1 M $NaNO_3$ stock solution.

2) Measure and record the pH of each sample (see FL DATA TABLE).

3) Add HNO_3 or $NaHCO_3$ to each sample until target pH (2.00, 3.00, 5.00, or 6.00) is achieved. Record this pH.

4) When target pH is reached immediately take from each bottle:

   a) Two 0.5 ml aliqouts by pouring approximately 0.5 ml into prelabeled and preweighed LSA vials FL*pH*i*a and FL*pH*i*b (where *i* is the target pH of the sample).

   b) Two 0.5 ml aliqouts with an Eppendorf pipet and place into prelabeled and preweighed LSA vials FL*pH*i*c and FL*pH*i*d (where *i* is the target pH of the sample).

   c) Two 0.5 ml aliqouts with an Eppendorf pipet after rinsing the tip with 0.02 M HNO_3 and place into prelabeled and preweighed LSA vials FL*pH*i*e and FL*pH*i*f (where *i* is the target pH of the sample). Rinsing of Eppendorf tips is achieved by uptaking and discharging 1 ml aliqouts. This should be done 3 times before taking the 0.5 ml sample.

5) Reweigh LSA sample vials + sample.

6) Add 0.5 ml of 0.02 M HNO3 to each LSA sample vial.

7) Analyze samples by LSA following procedure on p. 40.

3/5/93 JP

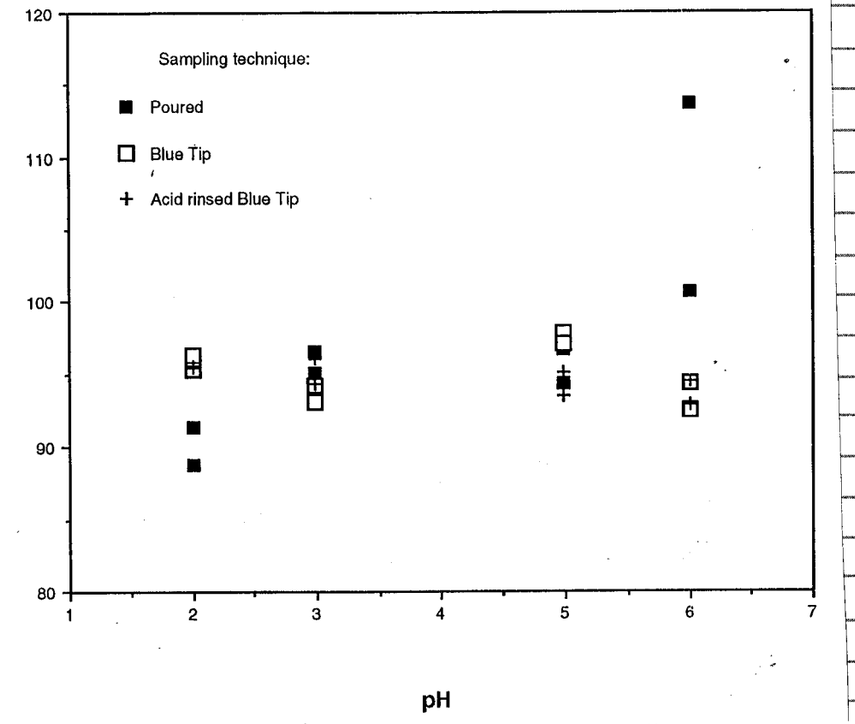
1600hrs Experimt TL samples were placed in LSA after adding cocktail.

Below is data on LSA analysis of TL samples.

Identification	pH	Targ pH	Vial wt g	S+v wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
TL*pH2.00*a	3.85	2.01	7.3925	7.8879	1	2	0.4954	1012.02	91.401
TL*pH2.00*b	3.85	2.01	7.4011	7.9397	1	3	0.5386	1069.24	88.823
TL*pH2.00*c	3.85	2.01	7.3806	7.8871	1	4	0.5065	1090.07	96.292
TL*pH2.00*d	3.85	2.01	7.3862	7.8915	1	5	0.5053	1076.57	95.325
TL*pH2.00*e	3.85	2.01	7.3326	7.8407	1	6	0.5081	1085.95	95.626
TL*pH2.00*f	3.85	2.01	7.368	7.8743	1	7	0.5063	1079.92	95.433
TL*pH3.00*a	3.86	2.99	7.4265	7.9773	1	8	0.5508	1170.83	95.108
TL*pH3.00*b	3.86	2.99	7.438	7.9886	1	9	0.5506	1188.66	96.591
TL*pH3.00*c	3.86	2.99	7.3323	7.84	1	10	0.5077	1056.24	93.083
TL*pH3.00*d	3.86	2.99	7.427	7.9323	1	11	0.5053	1064.58	94.264
TL*pH3.00*e	3.86	2.99	7.3239	7.8285	1	12	0.5046	1083.75	96.094
TL*pH3.00*f	3.86	2.99	7.4039	7.9095	1	13	0.5056	1065.7	94.307
TL*pH5.00*a	3.84	4.99	7.3455	7.7703	1	14	0.4248	895.741	94.344
TL*pH5.00*b	3.84	4.99	7.3588	7.9456	1	15	0.5868	1268.1	96.689
TL*pH5.00*c	3.84	4.99	7.33	7.8362	1	16	0.5062	1106.43	97.795
TL*pH5.00*d	3.84	4.99	7.4088	7.914	1	17	0.5052	1095.97	97.062
TL*pH5.00*e	3.84	4.99	7.3618	7.8699	1	18	0.5081	1080.65	95.159
TL*pH5.00*f	3.84	4.99	7.4586	7.9649	2	1	0.5063	1058.29	93.522
TL*pH6.00*a	3.82	6.01	7.3281	7.7525	2	2	0.4244	1078.46	113.696
TL*pH6.00*b	3.82	6.01	7.4151	7.9081	2	3	0.493	1109.2	100.665
TL*pH6.00*c	3.82	6.01	7.4088	7.9163	2	4	0.5075	1048.73	92.458
TL*pH6.00*d	3.82	6.01	7.4136	7.9186	2	5	0.505	1064.58	94.32
TL*pH6.00*e	3.82	6.01	7.3601	7.8662	2	6	0.5061	1050.14	92.838
TL*pH6.00*f	3.82	6.01	7.34	7.8449	2	7	0.5049	1066.91	94.545

Plot of TL data

U concentration vs pH for different sampling techniques.



3/17/93 JP

0915 hrs

EXPERIMENT SP - LSA ANALYSIS OF SPIKE 23A

OBJECTIVE: to determine the U concentration of spike 23A

EQUIPMENT: Liquid Scintillation Analyzer
Analytical balance

SUPPLIES: LSA vials 7ml
Eppendorf pipet (for taking samples) and blue tips
500 ppb stock solution prepared from 5 ppm ^{233}U commercial spike
0.02 m HNO_3 stock solution

PROCEDURE:

1. Take two 0.5 ml aliqouts of spike 23A by pouring approximately 0.5 ml into prelabeled and preweighed LSA vials SP-1 and SP-2

   Take two 0.5 ml aliqouts of spike 23A with an Eppendorf pipet and place into prelabeled and preweighed LSA vials SP-3 and SP-4.

   Take two 0.5 ml aliqouts of spike 23A with an Eppendorf pipet after rinsing tip with 0.02 M HNO_3 and place into prelabeled and preweighed LSA vials SP-5 and SP-6. Rinsing of Eppendorf tips is achieved by uptaking and discharging 1 ml aliqouts. This should be done 3 times before taking the 0.5 ml sample.

2) Reweigh LSA sample vials + sample.

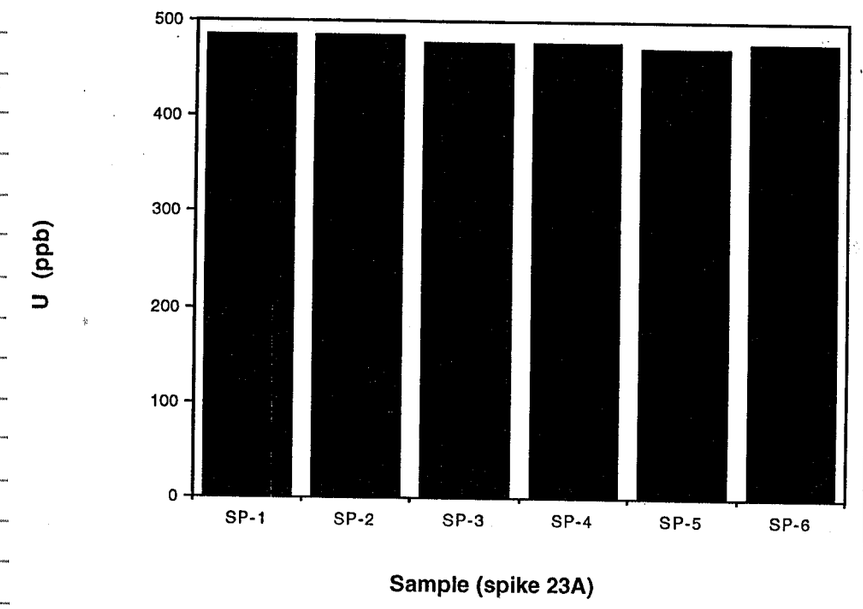
3) Add 0.5 ml of 0.02 M HNO3 to each LSA sample vial.

4) Analyze samples by LSA following procedure on p. 40.

Below is data for experiment SP

Identification	Vial wt g	S+v wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
SP-1	7.3459	7.8299	1	2	0.484	5237.66	484.18
SP-2	7.3278	7.8654	1	3	0.5376	5820.1	484.38
SP-3	7.3531	7.8486	1	4	0.4955	5285.33	477.29
SP-4	7.4149	7.918	1	5	0.5031	5364.52	477.08
SP-5	7.3655	7.8653	1	6	0.4998	5260.64	470.93
SP-6	7.3965	7.9016	1	7	0.5051	5393.59	477.77

Plot of U concentration for each sample.



5/13/93 JP

KINETICS EXPERIMENT Kα

OBJECTIVE: to determine the time it takes to reach sorption equilibrium between ^{233}U solutions and α-alumina powders

NOTE: prior experiments B-8005, B-8006, and B-8007 (initial ΣU = 5 ppb) indicated that maximum sorption of uranium onto α-alumina is obtained at equilibrium pH≈6.5. B-8005 and B-8006 solutions that had initial pH values of 3.5 to 6.5 ended up with equilibrium pH values around 6.75 to 7.00, whereas B-8007 solutions in this range had only slightly higher equilibrium pH values compared to initial pH's. Shift to more neutral equilibrium pH's in the pH range 3.5 to 6.5 is caused by α-alumina dissolution:

$$\alpha Al_2O_3 + H_2O + 3H^+ \rightarrow 2Al^{+++} + H_2O + 3OH^-$$

the larger shift for experiments B-8005 and B-8006 is caused by greater dissolution due to the α-alumina's higher surface area. Kinetics experiment Kα will have initial pH's of 4.00, 5.50, and 6.50. These initial pH's will be adjusted from that of a 100 ppb stock solution by addition of $NaHCO_3$, the amount of which is estimated using EQ3 calculations. The range of initial pH's should allow for the maximum shift of pH for solutions containing the highest surface area α-alumina.

EQUIPMENT: Liquid Scintillation Analyzer
ORION ph/mV/ISE/ C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance
Fisher Marathon 21K Centrifuge and 50 ml centrifuge tube adaptors

SUPPLIES:
- pH buffers (pH= 2,4,7)
- 50 ml FEP centrifuge tubes
- 1L FEP bottle
- LSA vials 7ml
- $NaHCO_3$ (**lot 897186A**)
- HNO_3 stock solutions (1.0, 0.1, 0.01, and 0.02 N)
- Eppendorf pipet (for taking samples) and blue tips
- 5 ppm ^{233}U commercial spike
- 0.1 m $NaNO_3$ stock solution (**lot 7808 KCCL**)
- alpha-alumina powders (NIST 8005, 8006, 8007)

PROCEDURE:

1330 hrs JP

In a precleaned 1L FEP bottle, prepare 650 g of 100 ppb U solution by diluting 130 g of a 500 ppb ^{233}U stock solution (**spike #23A**; in 0.1 m $NaNO_3$ matrix; prepared from a commercial 5 ppm ^{233}U spike) to a total of 650 g by taring 0.1 m $NaNO_3$ solution into the FEP bottle on a Mettler 4600 balance.

Kα - 4

- Initial ΣU = 100 ppb
- Initial ΣNa = 0.1+x (where 0.1 is from $NaNO_3$ and x is from $NaHCO_3$)

- Initial pH = 4.0
- Initial solution mass = 40 g
- 0.1 g of 8005, 8006, and 8007 α-alumina
- Equilibrium with atomspheric $CO_2(g)$ ($pCO_2 = 10^{-3.5}$ bar)

5/13/93 1400hrs gp

1. Transfer four 40 g aliqouts of the 100 ppb U solution into four 50 ml FEP centrifuge tubes labeled Kα-4*8005, Kα-4*8006, Kα-4*8007, and Kα-4*C.

2. Measure and record the initial pH of the solutions (≈3.85). Carefully add .0001 g of $NaHCO_3$ to each 50 ml solution. Cover tubes with a kimwipe and keep solution agitated to equilibrate with atmospheric $CO_2(g)$.

   Measure the pH of the solutions periodically until a constant value is reached (see Kœ pH Table), then proceed to the next step. This may take up to 10 days or longer.

3. Take two 0.5 ml aliquots from Kα-4*8005, Kα-4*8006, Kα-4*8007, and Kα-4*C using an Eppendorf pipet and transfer into prelabeled [e.g., Kα-4*8005*I1 (or I2)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

   Into tubes Kα-4*8005, Kα-4*8006, and Kα-4*8007, add 0.1 g of α-alumina powder 8005, 8006, and 8007, respectively. Record the time and room temperature. Solution Kα-4*C is a control to determine uranium losses to the container walls. Keep the solutions agitated.

4. At Δtime approximating the values listed below, centrifuge each tube at ~~15,000~~ 13,300 rpm for ~~30~~ 5 min, take one 0.5 ml sample from each solution with an Eppendorf pipet and transfer into prelabeled [e.g., Kα-4*8005*ia, where *i* is the sampling time number (1,2,3...)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA after all samples are taken. Measure and record the pH and temperature of each solution at each sampling time.

   5/31/93 gp

   *Take two 0.5 ml samples from the solutions <u>every third</u> sampling time. Label the second sample [e.g., Kα-4*8005*ib].*

   Use the following Δtime (hours): 2; 4; 22; 46; 70; 94; 142; 190; 238; 310; 382; 454. These may be changed as necessary.

Kα-5.5

- Initial ΣU = 100 ppb
- Initial ΣNa = 0.1+x (where 0.1 is from $NaNO_3$ and x is from $NaHCO_3$)
- Initial pH = 5.5
- Initial solution mass = 40 g
- 0.1 g of 8005, 8006, and 8007 α-alumina
- Equilibrium with atomspheric $CO_2(g)$ ($pCO_2 = 10^{-3.5}$ bar)

5/13/93 1400hrs gp

1. Transfer four 40 g aliqouts of the 100 ppb U solution into four 50 ml FEP centrifuge tubes labeled Kα-5.5*8005, Kα-5.5*8006, Kα-5.5*8007, and Kα-5.5*C.

2. Measure and record the initial pH of the solutions (≈3.85). Carefully add .0005 g of $NaHCO_3$ to each 50 ml solution. Cover tubes with a kimwipe and keep solution agitated to equilibrate with atmospheric $CO_2(g)$.

   Measure the pH of the solutions periodically until a constant value is reached (see Kœ pH Table), then proceed to the next step.

3. Take two 0.5 ml aliquots from Kα-5.5*8005, Kα-5.5*8006, Kα-5.5*8007, and Kα-5.5*C using an Eppendorf pipet and transfer into prelabeled [e.g., Kα-5.5*8005*I1 (or I2)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

   Into tubes Kα-5.5*8005, Kα-5.5*8006, and Kα-5.5*8007, add 0.1 g of α-alumina powder 8005, 8006, and 8007, respectively. Record the time and room temperature. Solution Kα-5.5*C is a control to determine uranium losses to the container walls. Keep the solutions agitated.

4. At Δtime approximating the values listed below, centrifuge each tube at ~~15,000~~ 13,300 rpm for ~~30~~ 5 min, take one 0.5 ml sample from each solution with an Eppendorf pipet and transfer into prelabeled [e.g., Kα-5.5*8005*ia, where *i* is the sampling time number (1,2,3...)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA after all samples are taken. Measure and record the pH and temperature of each solution at each sampling time.

   5/31/93 gp

   *Take two 0.5 ml samples from the solutions <u>every third</u> sampling time. Label the second sample [e.g., Kα-5.5*8005*ib].*

   Use the following Δtime (hours): 2; 4; 22; 46; 70; 94; 142; 190; 238; 310; 382; 454. These may be changed as necessary.

Kα-6.5

- Initial ΣU = 100 ppb
- Initial ΣNa = 0.1+x (where 0.1 is from $NaNO_3$ and x is from $NaHCO_3$)
- Initial pH = 6.5
- Initial solution mass = 40 g
- 0.1 g of 8005, 8006, and 8007 α-alumina
- Equilibrium with atomspheric $CO_2(g)$ ($pCO_2 = 10^{-3.5}$ bar)

5/13/93 gp 1400hrs

1. Transfer four 40 g aliqouts of the 100 ppb U solution into four 50 ml FEP centrifuge tubes labeled Kα-6.5*8005, Kα-6.5*8006, Kα-6.5*8007, and Kα-6.5*C.

2. Measure and record the initial pH of the solutions (≈3.85). Carefully add .0007 g of $NaHCO_3$ to each 50 ml solution. Cover tubes with a kimwipe and keep solution agitated to equilibrate with atmospheric $CO_2(g)$.

Measure the pH of the solutions periodically until a constant value is reached (see Kœ pH Table), then proceed to the next step.

3. Take two 0.5 ml aliquots from Kα-6.5*8005, Kα-6.5*8006, Kα-6.5*8007, and Kα-6.5*C using an Eppendorf pipet and transfer into prelabeled [e.g., Kα-6.5*8005*I1 (or I2)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

Into tubes Kα-6.5*8005, Kα-6.5*8006, and Kα-6.5*8007, add 0.1 g of α-alumina powder 8005, 8006, and 8007, respectively. Record the time and room temperature. Solution Kα-6.5*C is a control to determine uranium losses to the container walls. Keep the solutions agitated.

5/31/93 JP

4. At Δtime ~~approximating~~ JP 5/31/93 the values listed below, centrifuge each tube at ~~15,000~~ 13,300 JP 5/31/93 rpm for ~~5~~ 30 min, take one 0.5 ml sample from each solution with an Eppendorf pipet and transfer into prelabeled [e.g., Kα-6.5*8005*ia, where *i* is the sampling time number (1,2,3...)] and preweighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA after all samples are taken. Measure and record the pH and temperature of each solution at each sampling time.

*Take two 0.5 ml samples from the solutions every third sampling time. Label the second sample [e.g., Kα-6.5*8005*ib].*

Use the following Δtime (hours): 2; 4; 22; 46; 70; 94; 142; 190; 238; 310; 382; 454. These may be changed as necessary.

5/13/93 1430hr JP

Transfer remaining 100 ppb solution in the 1L bottle into two 50 ml FEP centrifuge tube labeled Kα*IU1 and Kα*IU2. Take two 0.5 ml aliqouts of solution Kα*IU1 and Kα*IU2 using an Eppendorf pipet and transfer into a prelabeled (Kα*IU1 or 2*a (or b)) and preweighed LSA vial. Reweigh vial after addition of sample, add 0.5 ml 0.02 m HNO_3, and analyze by LSA within the same day as sampling.

After the last samples have been taken, take two 0.5 ml aliqouts of solution Kα*IU1 and Kα*IU2 using an Eppendorf pipet and transfer into a prelabeled (Kα*FU1 or 2*a (or b)) and preweighed LSA vial. Reweigh vial after addition of sample, add 0.5 ml 0.02 m HNO_3, and analyze by LSA within the same day as sampling.

Kœ pH table

Identification	Initial pH	> pHs after	addition of	NaHCO3	>	>	>
Date/Time	5/13/93 1445	5/14/93 1510	5/18/93 1430	5/24/93 0830	5/26/93 0950	6/1/93 0815	
Kœ-4*8005	3.86 23.1°C	4.94 23.9°C	4.89 22.6°C	4.03 20.7°C	4.04 21.2°C	4.03 22.1°C	
Kœ-4*8006	3.86 23.1°C	4.02 24.0°C	3.99 22.6°C	4.00 20.7°C	4.02 21.2°C	4.02 22.1°C	
Kœ-4*8007	3.85 23.1°C	4.57 24.1°C	4.56 22.8°C	3.99 20.7°C	3.99 21.2°C	3.99 22.1°C	
Kœ-4*C	3.87 23.2°C	4.51 24.2°C	4.47 22.9°C	3.99 20.7°C	3.99 21.2°C	3.98 22.1°C	
Kœ-5.5*8005	3.86 23.2°C	6.41 23.9°C	6.42 22.6°C	5.46 20.7°C	5.46 21.3°C	5.45 22.1°C	
Kœ-5.5*8006	3.86 23.3°C	4.88 24.0°C	4.89 22.5°C	5.53 20.7°C	5.65 21.3°C	[illegible] 22.1°C	
Kœ-5.5*8007	3.85 23.3°C	5.74 24.1°C	5.51 22.8°C	5.43 20.6°C	5.44 21.2°C	5.44 22.1°C	
Kœ-5.5*C	3.85 23.3°C	4.45 24.2°C	4.47 22.7°C	5.36 20.7°C	5.39 21.3°C	5.39 22.1°C	
Kœ-6.5*8005	3.87 23.4°C	6.91 23.8°C	6.89 22.5°C	6.52 20.7°C	6.53 21.3°C	6.55 22.1°C	
Kœ-6.5*8006	3.89 23.4°C	7.04 24.3°C	7.09 22.7°C	6.58 20.6°C	6.58 21.4°C	6.62 22.1°C	
Kœ-6.5*8007	3.90 23.5°C	6.72 24.3°C	6.72 22.8°C	6.46 20.6°C	6.58 21.4°C	6.58 22.1°C	
Kœ-6.5*C	3.89 23.5°C	6.84 24.4°C	6.80 22.6°C	6.53 20.6°C	6.55 21.4°C	6.54 22.1°C	

5/19/93 gp 0900hrs.

pH's of Kα samples were adjusted to target values by addition of HNO_3 or $NaHCO_3$. Listed below are the amounts of HNO_3 or $NaHCO_3$ added to each sample.

	0.1M HNO_3(ml)	0.01M HNO_3(ml)	$NaHCO_3$(g)
Kα-4*8005	0.03	0.04	
Kα-4*8006	—	—	
Kα-4*8007	0.02	0.12	
Kα-4*C	0.02	0.11	
Kα-5.5*8005	0.01		
Kα-5.5*8006			.00002
Kα-5.5*8007			.000015
Kα-5.5*C			.00004
Kα-6.5*8005		0.13	
Kα-6.5*8006		0.225	
Kα-6.5*8007		0.04	
Kα-6.5*C		0.12	

6/1/93 gp Time & room temp that alpha-alumina was added to samples.

	Time		Temp	wt (g)
Kα-4*8005	1045	6/1/93	71.8°F	.1000
Kα-4*8006	1055	6/1/93	72.1°F	.1005
Kα-4*8007	1103	6/1/93	72.3°F	.0999
Kα-5.5*8005	1049	6/1/93	72.0°F	.1002
Kα-5.5*8006	1058	6/1/93	72.2°F	.1001
Kα-5.5*8007	1105	6/1/93	72.4°F	.1001
Kα-6.5*8005	1052	6/1/93	72.0°F	.1005
Kα-6.5*8006	1100	6/1/93	72.3°F	.1005
Kα-6.5*8007	1108	6/1/93	72.5°F	.1005

6/2/93 Prepared additional reducing agent for spectrophotometric analysis of SiO_2. Dissolved 500 mg 1-amino-2-Naphthol-4-sulfonic acid (lot 4807 KEKS) and 1 g Na_2SO_3 (lot 904672) in 50 ml H_2O. Dissolved 30 g $NaHSO_3$ (lot 915268) in 150 ml H_2O. Mixed the two solutions. Filtered into a 500 ml PP bottle & labeled "Reducing Agent".

6/3/93 JJF 1115hrs

Prepared a 10 ppm silica standard for spectrophotometric analysis of SiO_2. Diluted 10 ml silica standard (lot No. 3131-3, JohnsonINC) to 1L with H_2O in a 1L volumetric flask. Transferred solution to a 1L PP bottle & labeled "Standard Silica Solution".

Kæ experiment results.

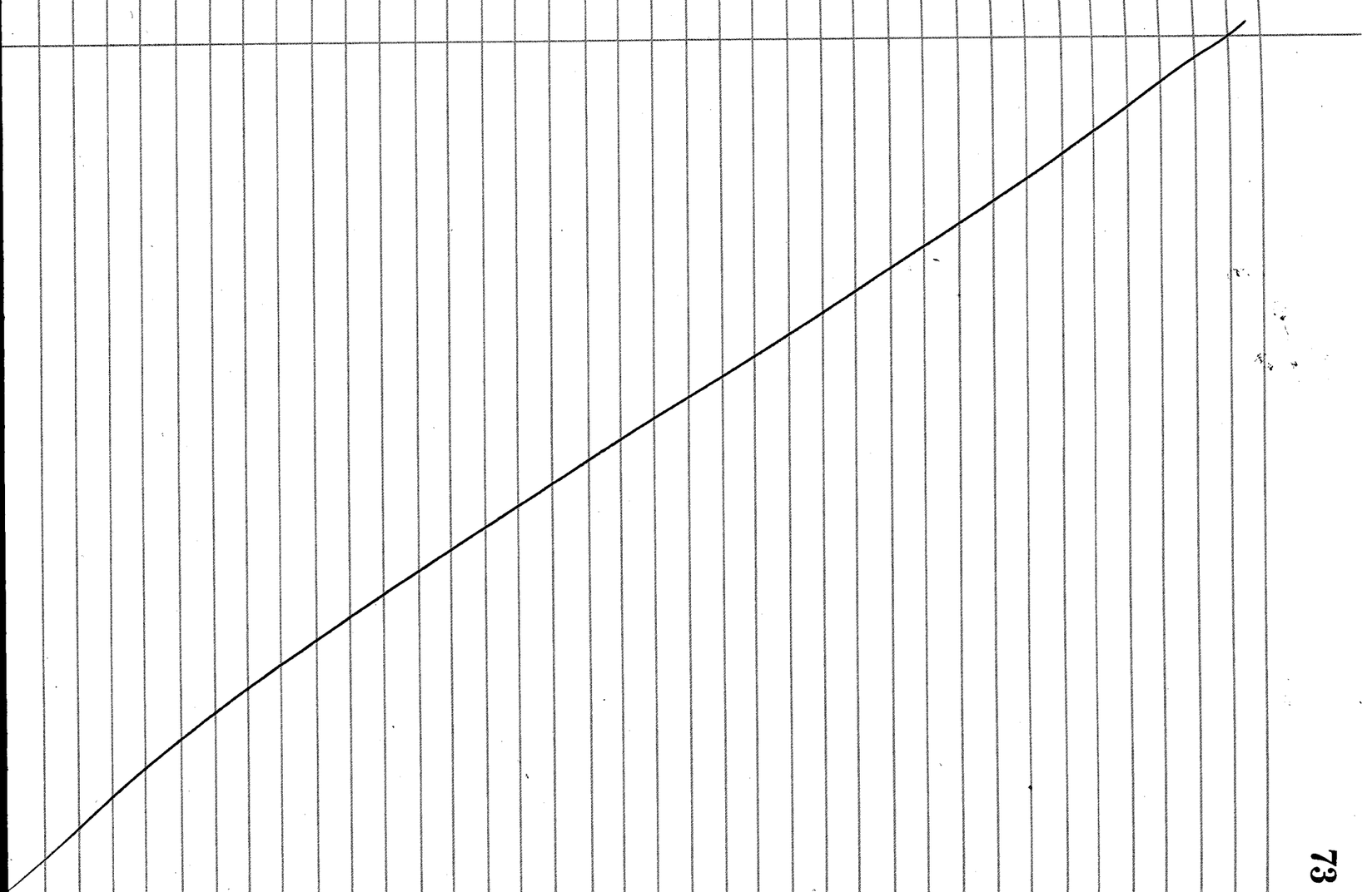
Kæ Initial Conc Sample Wt

Identification	Date/Time	pH	Vial wt g	Sam wt	CPMB	U (ppb)
Kæ*IU1*a	5/13/93 1430	3.82 23.0C	7.2749	0.4966	1059.03	95.42
Kæ*IU1*b	5/13/93 1430	3.82 23.0C	7.3007	0.4943	1065.49	96.44
Kæ*IU2*a	5/13/93 1430	3.81 23.1C	7.3191	0.499	1084.06	97.2
Kæ*IU2*b	5/13/93 1430	3.81 23.1C	7.3184	0.498	1078.54	96.9
Kæ*IU1*c	6/3/93 1314		7.2656	0.4986	1143.79	102.64
Kæ*IU2*c	6/3/93 1314		7.2655	0.4974	1174.13	105.61
Kæ*FU1*a	6/21/93 1035	3.78 23.2C	7.2423	0.5017	1245.64	111.09
Kæ*FU1*b	6/21/93 1035	3.78 23.2C	7.2572	0.501	1233.15	110.13
Kæ*FU2*a	6/21/93 1037	3.77 23.2C	7.2423	0.5019	1238.66	110.42
Kæ*FU2*b	6/21/93 1037	3.77 23.2C	7.2063	0.5037	1242.14	110.34
Kæ-4*8005*I1	6/1/93 0945	4.03 22.1C	7.2972	*8.2956		
Kæ-4*8005*I2	6/1/93 0945	4.03 22.1C	7.319	7.7715	1179.11	105.51
Kæ-4*8006*I1	6/1/93 0945	4.02 22.1C	7.2064	7.795	1218.86	108.35
Kæ-4*8006*I2	6/1/93 0945	4.02 22.1C	7.2587	7.8181	1172.87	104.81
Kæ-4*8007*I1	6/1/93 0945	3.99 22.1C	7.2621	7.8164	1174.66	105.01
Kæ-4*8007*I2	6/1/93 0945	3.99 22.1C	7.2694	7.7642	1164.86	104.19
Kæ-4*C*I1	6/1/93 0945	3.98 22.1C	7.2431	7.7629	1162.85	104.1
Kæ-4*C*I2	6/1/93 0945	3.98 22.1C	7.2072	7.744	1157.2	103.59
				7.7582	1170.76	104.85
				7.7442		
Kæ-5.5*8005*I1	6/1/93 0947	5.45 22.1C	7.259	7.71		
Kæ-5.5*8005*I2	6/1/93 0947	5.45 22.1C	7.2888			
Kæ-5.5*8006*I1	6/1/93 0947	5.64 22.1C	7.3037	7.7566	1013.32	91.11
Kæ-5.5*8006*I2	6/1/93 0947	5.64 22.1C	7.2618	7.7868	1048.42	94.19
Kæ-5.5*8007*I1	6/1/93 0947	5.44 22.1C	7.2551	7.8009	1075.77	96.81
Kæ-5.5*8007*I2	6/1/93 0947	5.44 22.1C	7.2752	7.7598	1125.06	101.08
				7.753	1043.24	93.75
				7.7728	1014.51	91.22

Sam +vial wt g	RACK	VIAL
	17	8
	17	9
	17	10
	17	11
	20	14
	20	15
	7	10
	7	11
	7	12
	7	13
7.2956	8	2
7.8223	8	3
7.7071	8	4
7.7592	8	5
7.7623	8	6
7.7692	8	7
7.7429	8	8
7.7068	8	9
7.7566	8	10
7.7868	8	11
7.8009	8	12
7.7598	8	13
7.753	8	14
7.7728	8	15

Kæ Initial Conc Sample Wt

Kæ-5.5*C*I1	6/1/93 0947	5.39 22.1C	7.1795	7.6778	8	16	0.4983	1097.13	98.51
Kæ-5.5*C*I2	6/1/93 0947	5.39 22.1C	7.2753	7.7733	8	17	0.498	1099.61	98.79
Kæ-6.5*8005*I1	6/1/93 0949	6.55 22.1C	7.3232	7.8238	8	18	0.5006	913.963	81.69
Kæ-6.5*8005*I2	6/1/93 0949	6.55 22.1C	7.2796	7.7784	9	1	0.4988	937.688	84.11
Kæ-6.5*8006*I1	6/1/93 0949	6.62 22.1C	7.2012	7.6988	9	2	0.4976	916.968	82.45
Kæ-6.5*8006*I2	6/1/93 0949	6.62 22.1C	7.2849	7.7844	9	3	0.4995	938.834	84.09
Kæ-6.5*8007*I1	6/1/93 0949	6.58 22.1C	7.3126	7.8148	9	4	0.5022	814.9	72.6
Kæ-6.5*8007*I2	6/1/93 0949	6.58 22.1C	7.2362	7.7358	9	5	0.4996	842.512	75.45
Kæ-6.5*C*I1	6/1/93 0949	6.54 22.1C	7.2558	7.758	9	6	0.5022	911.053	81.17
Kæ-6.5*C*I2	6/1/93 0949	6.54 22.1C	7.2373	7.7371	9	7	0.4998	940.409	84.19

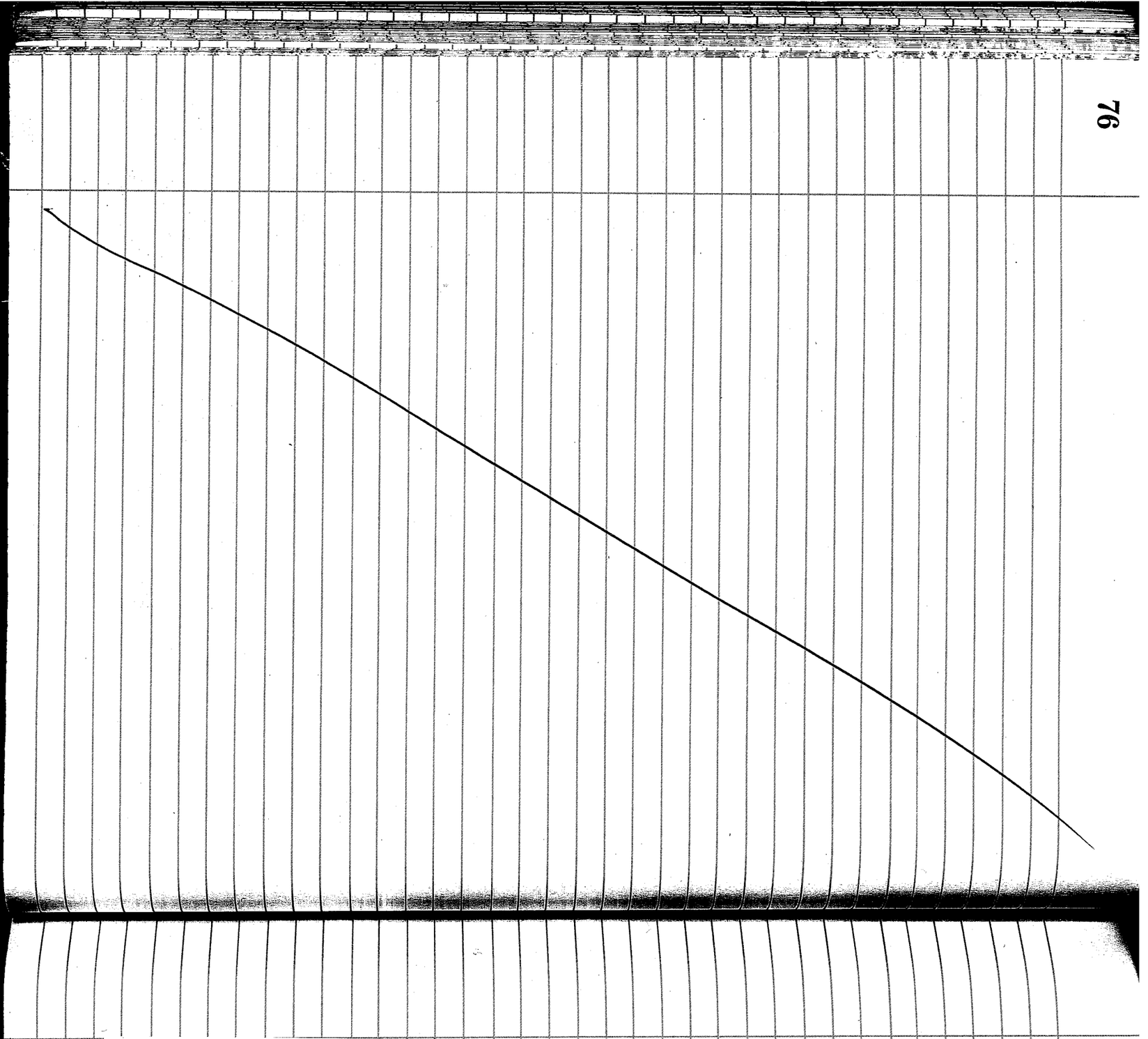


Kæ-4 Sample Wt

Identification	Date/Time	pH	Vial wt g	Sam +vial wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
Kæ-4*8005*1a	6/1/93 1440	4.61 24.5C	7.1184	7.6085	17	2	0.4901	1043.47	95.26
Kæ-4*8005*2a	6/1/93 1628	4.84 24.8C	7.1654	7.6542	17	3	0.4888	1078.49	98.72
Kæ-4*8005*3a	6/2/93 1035	5.71 21.8C	7.269	7.7649	17	4	0.4959	639.37	57.69
Kæ-4*8005*3b	6/2/93 1035	5.71 21.8C	7.319	7.8399	17	5	0.5209	694.22	59.63
Kæ-4*8005*4a	6/3/93 0930	5.76 20.5C	7.2708	7.7658	5	1	0.495	564.171	50.99
Kæ-4*8005*5a	6/4/93 0937	5.80 20.6C	7.291	7.8166	5	2	0.5256	539.894	45.96
Kæ-4*8005*6a	6/5/93 0943	5.91 20.8C	7.2819	7.7799	5	3	0.498	502.264	45.12
Kæ-4*8005*6b	6/5/93 0943	5.91 20.8C	7.2759	7.7998	5	4	0.5239	504.865	43.1
Kæ-4*8005*7a	6/7/93 0950	5.92 20.9C	7.2757	7.7699	5	2	0.4942	405.329	36.7
Kæ-4*8005*8a	6/9/93 0925	5.92 21.3C	7.3247	7.8211	5	3	0.4964	386.706	34.85
Kæ-4*8005*9a	6/11/93 0917	6.02 20.9C	7.3384	7.8354	5	4	0.497	360.198	32.43
Kæ-4*8005*9b	6/11/93 0917	6.02 20.9C	7.3712	7.8683	5	5	0.4971	349.659	31.47
Kæ-4*8005*10a	6/14/93 1007	6.13 20.0C	7.3359	7.8394	19	2	0.5035	335.049	29.77
Kæ-4*8005*11a	6/17/93 0925	6.18 21.4C	7.8409	8.3439	4	2	0.503	320.626	28.52
Kæ-4*8005*12a	6/21/93 1033	6.16 23.2C	7.3019	7.7989	6	2	0.497	287.484	25.88
Kæ-4*8005*12b	6/21/93 1033	6.16 23.2C	7.3269	7.8246	6	3	0.4977	285.712	25.68
Kæ-4*8006*1a	6/1/93 1500	4.35 24.6C	7.1625	7.6535	17	6	0.491	1162.07	105.89
Kæ-4*8006*2a	6/1/93 1640	4.41 24.8C	7.2738	7.7671	17	7	0.4933	1109.14	100.6
Kæ-4*8006*3a	6/2/93 1001	4.78 21.8C	7.2916	7.7967	17	8	0.5051	1151.55	102
Kæ-4*8006*3b	6/2/93 1001	4.78 21.8C	7.2715	7.7816	17	9	0.5101	1193.12	104.65
Kæ-4*8006*4a	6/3/93 0858	4.85 20.5C	7.2625	7.7661	5	5	0.5036	1154.59	102.58
Kæ-4*8006*5a	6/4/93 0904	4.84 20.6C	7.2971	7.7989	5	6	0.5018	1122.61	100.1
Kæ-4*8006*6a	6/5/93 0915	4.90 20.8C	7.2625	7.7645	5	7	0.502	1124.38	100.21
Kæ-4*8006*6b	6/5/93 0915	4.90 20.8C	7.2709	7.7788	5	8	0.5079	1182.41	104.16
Kæ-4*8006*7a	6/7/93 0830	4.90 20.9C	7.2967	7.7969	5	6	0.5002	1162.06	103.94
Kæ-4*8006*8a	6/9/93 0906	4.86 21.3C	7.3967	7.8983	5	7	0.5016	1152.47	102.8
Kæ-4*8006*9a	6/11/93 0858	4.91 20.9C	7.3414	7.8426	5	8	0.5012	1120.62	100.04
Kæ-4*8006*9b	6/11/93 0858	4.91 20.9C	7.2222	7.7247	5	9	0.5025	1147.01	102.13
Kæ-4*8006*10a	6/14/93 1008	5.00 20.0C	7.2925	7.7965	19	3	0.504	1116.45	99.11
Kæ-4*8006*11a	6/17/93 0926	4.99 21.4C	7.8139	8.315	4	7	0.5011	1098.85	98.11
Kæ-4*8006*12a	6/21/93 1032	5.03 23.2C	7.3003	7.8007	6	4	0.5004	1042.31	93.19
Kæ-4*8006*12b	6/21/93 1032	5.03 23.2C	7.2469	7.7469	6	5	0.5	1080.91	96.72

Kæ-4 Sample Wt

Kæ-4*8007*1a	6/1/93 1514	3.99 24.7C	7.2668	7.7571	17	10	0.4903	1149.48	104.89
Kæ-4*8007*2a	6/1/93 1650	3.96 24.9C	7.3044	7.7996	17	11	0.4952	1204.77	108.85
Kæ-4*8007*3a	6/2/93 1005	4.01 21.8C	7.2317	7.7315	17	12	0.4998	1216.06	108.86
Kæ-4*8007*3b	6/2/93 1005	4.01 21.8C	7.3016	7.8137	17	13	0.5121	1245.76	108.84
Kæ-4*8007*4a	6/3/93 0900	4.01 20.5C	7.2436	7.7436	5	9	0.5	1190.87	106.56
Kæ-4*8007*5a	6/4/93 0907	4.02 20.6C	7.2582	7.7569	5	10	0.49787	1251.69	112.3
Kæ-4*8007*6a	6/5/93 0914	4.05 20.8C	7.2803	7.7801	5	11	0.4998	1214.25	108.7
Kæ-4*8007*6b	6/5/93 0914	4.05 20.8C	7.1824	7.6998	5	12	0.5174	1276.06	110.35
Kæ-4*8007*7a	6/7/93 0827	3.99 20.9C	7.3248	7.8232	5	10	0.4984	1192.31	107.04
Kæ-4*8007*8a	6/9/93 0903	3.99 21.3C	7.3319	7.8308	5	11	0.4989	1231.51	110.44
Kæ-4*8007*9a	6/11/93 0854	4.00 20.9C	7.3359	7.8336	5	12	0.4977	1253.66	112.7
Kæ-4*8007*9b	6/11/93 0854	4.00 20.9C	7.3682	7.8694	5	13	0.5012	1289.06	115.07
Kæ-4*8007*10a	6/14/93 1009	4.04 20.0C	7.3441	7.8432	19	4	0.4991	1242.48	111.38
Kæ-4*8007*11a	6/17/93 0925	3.96 21.1C	7.8202	8.3231	4	4	0.5029	1289.56	114.73
Kæ-4*8007*12a	6/21/93 1030	4.03 23.2C	7.2575	7.7581	6	6	0.5006	1315.4	117.57
Kæ-4*8007*12b	6/21/93 1030	4.03 23.2C	7.2373	7.7369	6	7	0.4996	1243.54	111.37
Kæ-4*C*1a	6/1/93 1445	3.93 24.5C	7.2689	7.7577	17	14	0.4888	1145.98	104.9
Kæ-4*C*2a	6/1/93 1630	3.94 24.8C	7.2328	7.7235	17	15	0.4907	1122.2	102.32
Kæ-4*C*3a	6/2/93 1033	3.91 21.8C	7.3062	7.8047	17	16	0.4985	991.516	89
Kæ-4*C*3b	6/2/93 1032	3.91 21.8C	7.2517	7.7815	17	17	0.5298	1174.93	99.22
Kæ-4*C*4a	6/3/93 0901	4.00 20.6C	7.1365	7.6358	5	13	0.4993	1157.61	103.73
Kæ-4*C*5a	6/4/93 0908	3.99 20.6C	7.2776	7.776	5	14	0.4984	1141.51	102.47
Kæ-4*C*6a	6/5/93 0912	3.99 20.8C	7.2863	7.7846	5	15	0.4983	1175.87	105.58
Kæ-4*C*6b	6/5/93 0912	3.99 20.8C	7.2402	7.7488	5	16	0.5086	1199.2	105.49
Kæ-4*C*7a	6/7/93 0826	3.96 20.8C	7.2798	7.778	5	14	0.4982	1170.5	105.12
Kæ-4*C*8a	6/9/93 0900	3.98 21.3C	7.3573	7.8579	5	15	0.5006	1163.81	104.02
Kæ-4*C*9a	6/11/93 0852	3.99 20.9C	7.3383	7.8404	5	16	0.5021	1183.01	105.42
Kæ-4*C*9b	6/11/93 0852	3.99 20.9C	7.3843	7.886	5	17	0.5017	1189.11	106.05
Kæ-4*C*10a	6/14/93 1010	4.03 20.1C	7.3393	7.8419	19	5	0.5026	1193.61	106.26
Kæ-4*C*11a	6/17/93 0927	3.96 21.4C	7.8975	8.4043	4	5	0.5068	1243.62	109.8
Kæ-4*C*12a	6/21/93 1028	4.01 23.2C	7.2285	7.7261	6	8	0.4976	1194.35	107.39
Kæ-4*C*12b	6/21/93 1028	4.01 23.2C	7.2304	7.7286	6	9	0.4982	1242.98	111.63

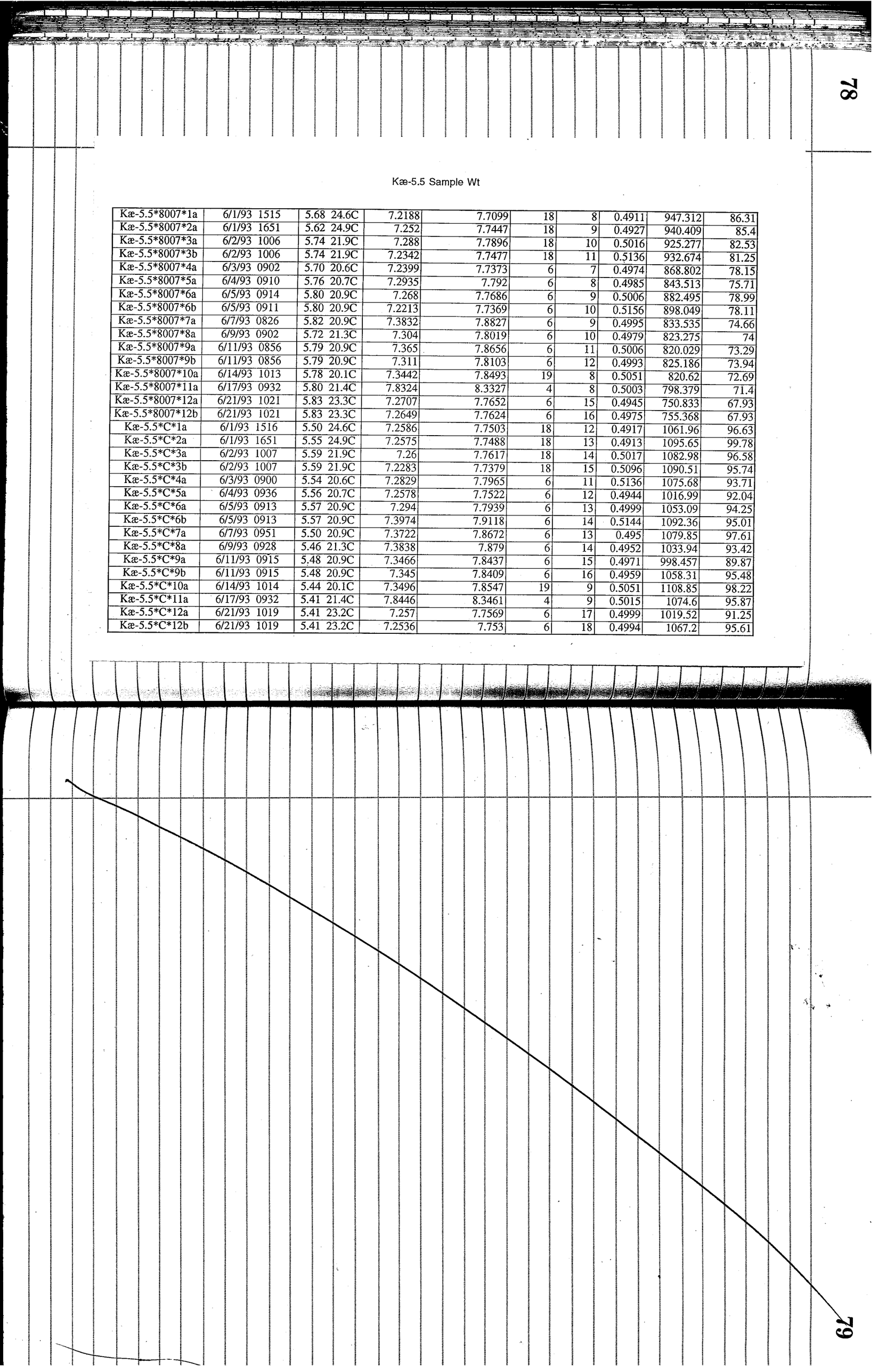


Kæ-5.5 Sample Wt

Identification	Date/Time	pH	Vial wt g	Sam +vial wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
Kæ-5.5*8005*1a	6/1/93 1442	7.13 24.5C	7.2364	7.725	17	18	0.4886	777.938	71.24
Kæ-5.5*8005*2a	6/1/93 1630	7.04 24.9C	7.2444	7.7315	18	1	0.4871	729.175	66.98
Kæ-5.5*8005*3a	6/2/93 1034	6.98 22.1C	7.2861	7.7804	18	2	0.4943	520.442	47.11
Kæ-5.5*8005*3b	6/2/93 1034	6.98 22.1C	7.228	7.7507	18	3	0.5227	544.85	46.64
Kæ-5.5*8005*4a	6/3/93 0930	6.97 20.6C	7.2702	7.7628	5	17	0.4926	463.075	42.06
Kæ-5.5*8005*5a	6/4/93 0933	6.99 20.6C	7.2207	7.7159	5	18	0.4952	445.122	40.22
Kæ-5.5*8005*6a	6/5/93 0942	7.03 20.9C	7.2291	7.7274	6	1	0.4983	418.459	37.57
Kæ-5.5*8005*6b	6/5/93 0942	7.03 20.9C	7.2628	7.7894	6	2	0.5266	442.582	37.6
Kæ-5.5*8005*7a	6/7/93 0950	7.04 20.9C	7.3027	7.7971	6	1	0.4944	395.715	35.81
Kæ-5.5*8005*8a	6/9/93 0927	6.95 21.4C	7.3689	7.8663	6	2	0.4874	399.78	35.96
Kæ-5.5*8005*9a	6/11/93 0914	7.05 21.0C	7.3368	7.8317	6	3	0.4949	411.14	37.17
Kæ-5.5*8005*9b	6/11/93 0914	7.05 21.0C	7.2915	7.7877	6	4	0.4962	395.893	35.7
Kæ-5.5*8005*10a	6/14/93 1011	6.97 20.1C	7.3536	7.8576	19	6	0.504	413.005	36.66
Kæ-5.5*8005*11a	6/17/93 0930	6.93 21.4C	7.8427	8.3459	4	6	0.5032	390.392	34.71
Kæ-5.5*8005*12a	6/21/93 1026	6.96 23.3C	7.261	7.7595	6	11	0.4985	370.546	33.26
Kæ-5.5*8005*12b	6/21/93 1026	6.96 23.3C	7.2405	7.7383	6	12	0.4978	363.377	32.66
Kæ-5.5*8006*1a	6/1/93 1501	6.93 24.6C	7.2723	7.7607	18	4	0.4884	961.093	88.05
Kæ-5.5*8006*2a	6/1/93 1641	6.88 24.9C	7.3058	7.7981	18	5	0.4923	964.494	87.66
Kæ-5.5*8006*3a	6/2/93 1002	6.90 21.9C	7.1786	7.6818	18	6	0.5023	852.27	75.92
Kæ-5.5*8006*3b	6/2/93 1002	6.90 21.9C	7.2724	7.7826	18	7	0.5102	874.597	76.7
Kæ-5.5*8006*4a	6/3/93 0901	6.88 20.6C	7.2689	7.7688	6	3	0.4999	754.618	67.54
Kæ-5.5*8006*5a	6/4/93 0903	6.86 20.7C	7.2626	7.7633	6	4	0.5007	756.593	67.61
Kæ-5.5*8006*6a	6/5/93 0914	6.89 20.9C	7.2641	7.765	6	5	0.5009	743.01	66.37
Kæ-5.5*8006*6b	6/5/93 0914	6.89 20.9C	7.2571	7.7646	6	6	0.5075	766.414	67.57
Kæ-5.5*8006*7a	6/7/93 0829	6.85 20.9C	7.3495	7.85	6	5	0.5005	722.656	64.6
Kæ-5.5*8006*8a	6/9/93 0905	6.82 21.3C	7.3557	7.855	6	6	0.4993	713.145	63.9
Kæ-5.5*8006*9a	6/11/93 0850	6.88 20.9C	7.366	7.8675	6	7	0.5015	710.525	63.39
Kæ-5.5*8006*9b	6/11/93 0850	6.88 20.9C	7.3297	7.8307	6	8	0.501	727.423	64.96
Kæ-5.5*8006*10a	6/14/93 1012	6.84 20.1C	7.3342	7.837	19	7	0.5028	717.167	63.82
Kæ-5.5*8006*11a	6/17/93 0931	6.86 21.4C	7.8246	8.3305	4	7	0.5059	724.777	64.1
Kæ-5.5*8006*12a	6/21/93 1023	6.85 23.3C	7.2578	7.7566	6	13	0.4988	675.238	60.57
Kæ-5.5*8006*12b	6/21/93 1023	6.85 23.3C	7.2363	7.7363	6	14	0.5	688.069	61.57

Kæ-5.5 Sample Wt

Kæ-5.5*8007*1a	6/1/93 1515	5.68 24.6C	7.2188	7.7099	18	8	0.4911	947.312	86.31
Kæ-5.5*8007*2a	6/1/93 1651	5.62 24.9C	7.252	7.7447	18	9	0.4927	940.409	85.4
Kæ-5.5*8007*3a	6/2/93 1006	5.74 21.9C	7.288	7.7896	18	10	0.5016	925.277	82.53
Kæ-5.5*8007*3b	6/2/93 1006	5.74 21.9C	7.2342	7.7477	18	11	0.5136	932.674	81.25
Kæ-5.5*8007*4a	6/3/93 0902	5.70 20.6C	7.2399	7.7373	6	7	0.4974	868.802	78.15
Kæ-5.5*8007*5a	6/4/93 0910	5.76 20.7C	7.2935	7.792	6	8	0.4985	843.513	75.71
Kæ-5.5*8007*6a	6/5/93 0914	5.80 20.9C	7.268	7.7686	6	9	0.5006	882.495	78.99
Kæ-5.5*8007*6b	6/5/93 0911	5.80 20.9C	7.2213	7.7369	6	10	0.5156	898.049	78.11
Kæ-5.5*8007*7a	6/7/93 0826	5.82 20.9C	7.3832	7.8827	6	9	0.4995	833.535	74.66
Kæ-5.5*8007*8a	6/9/93 0902	5.72 21.3C	7.304	7.8019	6	10	0.4979	823.275	74
Kæ-5.5*8007*9a	6/11/93 0856	5.79 20.9C	7.365	7.8656	6	11	0.5006	820.029	73.29
Kæ-5.5*8007*9b	6/11/93 0856	5.79 20.9C	7.311	7.8103	6	12	0.4993	825.186	73.94
Kæ-5.5*8007*10a	6/14/93 1013	5.78 20.1C	7.3442	7.8493	19	8	0.5051	820.62	72.69
Kæ-5.5*8007*11a	6/17/93 0932	5.80 21.4C	7.8324	8.3327	4	8	0.5003	798.379	71.4
Kæ-5.5*8007*12a	6/21/93 1021	5.83 23.3C	7.2707	7.7652	6	15	0.4945	750.833	67.93
Kæ-5.5*8007*12b	6/21/93 1021	5.83 23.3C	7.2649	7.7624	6	16	0.4975	755.368	67.93
Kæ-5.5*C*1a	6/1/93 1516	5.50 24.6C	7.2586	7.7503	18	12	0.4917	1061.96	96.63
Kæ-5.5*C*2a	6/1/93 1651	5.55 24.9C	7.2575	7.7488	18	13	0.4913	1095.65	99.78
Kæ-5.5*C*3a	6/2/93 1007	5.59 21.9C	7.26	7.7617	18	14	0.5017	1082.98	96.58
Kæ-5.5*C*3b	6/2/93 1007	5.59 21.9C	7.2283	7.7379	18	15	0.5096	1090.51	95.74
Kæ-5.5*C*4a	6/3/93 0900	5.54 20.6C	7.2829	7.7965	6	11	0.5136	1075.68	93.71
Kæ-5.5*C*5a	6/4/93 0936	5.56 20.7C	7.2578	7.7522	6	12	0.4944	1016.99	92.04
Kæ-5.5*C*6a	6/5/93 0913	5.57 20.9C	7.294	7.7939	6	13	0.4999	1053.09	94.25
Kæ-5.5*C*6b	6/5/93 0913	5.57 20.9C	7.3974	7.9118	6	14	0.5144	1092.36	95.01
Kæ-5.5*C*7a	6/7/93 0951	5.50 20.9C	7.3722	7.8672	6	13	0.495	1079.85	97.61
Kæ-5.5*C*8a	6/9/93 0928	5.46 21.3C	7.3838	7.879	6	14	0.4952	1033.94	93.42
Kæ-5.5*C*9a	6/11/93 0915	5.48 20.9C	7.3466	7.8437	6	15	0.4971	998.457	89.87
Kæ-5.5*C*9b	6/11/93 0915	5.48 20.9C	7.345	7.8409	6	16	0.4959	1058.31	95.48
Kæ-5.5*C*10a	6/14/93 1014	5.44 20.1C	7.3496	7.8547	19	9	0.5051	1108.85	98.22
Kæ-5.5*C*11a	6/17/93 0932	5.41 21.4C	7.8446	8.3461	4	9	0.5015	1074.6	95.87
Kæ-5.5*C*12a	6/21/93 1019	5.41 23.2C	7.257	7.7569	6	17	0.4999	1019.52	91.25
Kæ-5.5*C*12b	6/21/93 1019	5.41 23.2C	7.2536	7.753	6	18	0.4994	1067.2	95.61



Kæ-6.5 Sample Wt

Identification	Date/Time	pH	Vial wt g	Sam +vial wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
Kæ-6.5*8005*1a	6/1/93 1444	7.40 24.5C	7.2366	7.7242	18	16	0.4876	732.812	67.24
Kæ-6.5*8005*2a	6/1/93 1630	7.22 24.9C	7.2703	7.7584	18	17	0.4881	705.976	64.71
Kæ-6.5*8005*3a	6/2/93 1033	7.18 22.0C	7.2625	7.7623	18	18	0.4998	529.1	47.36
Kæ-6.5*8005*3b	6/2/93 1033	7.18 22.0C	7.2637	7.7929	20	1	0.5292	571.788	48.34
Kæ-6.5*8005*4a	6/3/93 0931	7.17 20.7C	7.284	7.7773	6	15	0.4933	453.769	41.16
Kæ-6.5*8005*5a	6/4/93 0935	7.16 20.7C	7.2088	7.7024	6	16	0.4936	429.204	38.9
Kæ-6.5*8005*6a	6/5/93 0941	7.21 20.9C	7.3284	7.9582	6	17	0.6298	538.693	38.27
Kæ-6.5*8005*6b	6/5/93 0941	7.21 20.9C	7.3945	7.9268	6	18	0.5323	453.55	38.12
Kæ-6.5*8005*7a	6/7/93 0952	7.19 21.0C	7.3642	7.8594	7	1	0.4952	408.074	36.87
Kæ-6.5*8005*8a	6/9/93 0926	7.07 21.4C	7.3099	7.807	7	2	0.4971	395.178	35.57
Kæ-6.5*8005*9a	6/11/93 0912	7.15 21.0C	7.36	7.8567	7	3	0.4967	414.55	37.34
Kæ-6.5*8005*9b	6/11/93 0912	7.15 21.0C	7.2795	7.7741	7	4	0.4946	402.077	36.37
Kæ-6.5*8005*10a	6/14/93 1015	7.06 20.2C	7.3646	7.8686	19	10	0.504	426.474	37.86
Kæ-6.5*8005*11a	6/17/93 0933	7.06 21.4C	7.8582	8.3594	4	10	0.5012	426.433	38.07
Kæ-6.5*8005*12a	6/21/93 1015	7.16 23.4C	7.3247	7.822	7	1	0.4973	395.689	35.6
Kæ-6.5*8005*12b	6/21/93 1015	7.16 23.4C	7.2707	7.7666	7	2	0.4959	393.908	35.54
Kæ-6.5*8006*1a	6/1/93 1502	7.31 24.6C	7.3097	7.8011	20	2	0.4914	882.542	80.36
Kæ-6.5*8006*2a	6/1/93 1642	7.14 24.9C	7.2563	7.7738	20	3	0.5175	898.305	77.67
Kæ-6.5*8006*3a	6/2/93 1004	7.12 22.1C	7.2541	7.7573	20	4	0.5032	783.281	69.64
Kæ-6.5*8006*3b	6/2/93 1004	7.12 22.1C	7.2515	7.7598	20	5	0.5083	803.769	70.75
Kæ-6.5*8006*4a	6/3/93 0903	7.08 20.7C	7.2465	7.7458	7	1	0.4993	733.957	65.77
Kæ-6.5*8006*5a	6/4/93 0902	7.09 20.7C	7.2499	7.7516	7	2	0.5017	732.244	65.3
Kæ-6.5*8006*6a	6/5/93 0916	7.10 21.0C	7.3025	7.806	7	3	0.5035	721.102	64.08
Kæ-6.5*8006*6b	6/5/93 0916	7.10 21.0C	7.3436	7.8528	7	4	0.5092	726.381	63.83
Kæ-6.5*8006*7a	6/7/93 0828	7.08 21.0C	7.2868	7.7857	7	5	0.4989	686.493	61.57
Kæ-6.5*8006*8a	6/9/93 0908	7.02 21.4C	7.3505	7.8524	7	6	0.5019	684.207	60.99
Kæ-6.5*8006*9a	6/11/93 0900	7.05 21.0C	7.1923	7.6959	7	7	0.5036	709.221	63.01
Kæ-6.5*8006*9b	6/11/93 0900	7.05 21.0C	7.3049	7.8078	7	8	0.5029	701.153	62.38
Kæ-6.5*8006*10a	6/14/93 1010	7.03 20.2C	7.3636	7.8668	19	11	0.5032	710.746	63.2
Kæ-6.5*8006*11a	6/17/93 0933	7.03 21.4C	7.8769	8.3798	4	11	0.5029	704.431	62.67
Kæ-6.5*8006*12a	6/21/93 1013	7.03 23.3C	7.2836	7.7827	7	3	0.4991	697.237	62.5
Kæ-6.5*8006*12b	6/21/93 1013	7.03 23.3C	7.2652	7.7634	7	4	0.4982	685.155	61.53

Kæ-6.5 Sample Wt

Kæ-6.5*8007*1a	6/1/93 1516	6.54 24.6C	7.2769	7.7694	20	6	0.4925	764.761	69.48
Kæ-6.5*8007*2a	6/1/93 1652	6.56 24.9C	7.2746	7.7679	20	7	0.4933	743.865	67.46
Kæ-6.5*8007*3a	6/2/93 1007	6.55 22.0C	7.2591	7.7638	20	8	0.5047	743.944	65.95
Kæ-6.5*8007*3b	6/2/93 1007	6.55 22.0C	7.2572	7.7702	20	9	0.513	756.271	65.96
Kæ-6.5*8007*4a	6/3/93 0903	6.57 20.6C	7.2512	7.7519	7	5	0.5007	691.724	61.81
Kæ-6.5*8007*5a	6/4/93 0908	6.59 20.8C	7.263	7.7606	7	6	0.4976	711.102	63.94
Kæ-6.5*8007*6a	6/5/93 0912	6.60 21.0C	7.3817	7.8815	7	7	0.4998	701.309	62.78
Kæ-6.5*8007*6b	6/5/93 0912	6.60 21.0C	7.3212	7.8342	7	8	0.513	680.447	59.35
Kæ-6.5*8007*7a	6/7/93 0825	6.60 21.0C	7.3882	7.8858	7	9	0.4976	656.436	59.02
Kæ-6.5*8007*8a	6/9/93 0904	6.58 21.4C	7.3673	7.8663	7	10	0.499	671.729	60.23
Kæ-6.5*8007*9a	6/11/93 0902	6.57 21.0C	7.347	7.8456	7	11	0.4986	632.024	56.71
Kæ-6.5*8007*9b	6/11/93 0902	6.57 21.0C	7.3847	7.8843	7	12	0.4996	646.954	57.94
Kæ-6.5*8007*10a	6/14/93 1017	6.57 20.2C	7.3878	7.884	19	12	0.4962	639.526	57.67
Kæ-6.5*8007*11a	6/17/93 0934	6.59 21.4C	7.8324	8.3313	4	12	0.4989	644.134	57.77
Kæ-6.5*8007*12a	6/21/93 1011	6.59 23.3C	7.2403	7.7394	7	5	0.4991	622.68	55.82
Kæ-6.5*8007*12b	6/21/93 1011	6.59 23.3C	7.2612	7.7601	7	6	0.4989	617.854	55.41
Kæ-6.5*C*1a	6/1/93 1459	6.51 24.5C	7.179	7.669	20	10	0.49	889.254	81.2
Kæ-6.5*C*2a	6/1/93 1642	6.53 24.9C	7.2798	7.7736	20	11	0.4938	858.9	77.82
Kæ-6.5*C*3a	6/2/93 1008	6.54 21.9C	7.2686	7.7679	20	12	0.4993	835.375	74.86
Kæ-6.5*C*3b	6/2/93 1008	6.54 21.9C	7.2547	7.7646	20	13	0.5099	864.854	75.89
Kæ-6.5*C*4a	6/3/93 0929	6.52 20.6C	7.2144	7.7109	7	9	0.4965	809.468	72.94
Kæ-6.5*C*5a	6/4/93 0905	6.53 20.8C	7.2386	7.7393	7	10	0.5007	802.834	71.74
Kæ-6.5*C*6a	6/5/93 0940	6.60 21.0C	7.3941	7.893	7	11	0.4989	849.527	76.19
Kæ-6.5*C*6b	6/5/93 0940	6.60 21.0C	7.3104	7.8388	7	12	0.5284	799.564	67.7
Kæ-6.5*C*7a	6/7/93 0827	6.52 20.9C	7.3159	7.8161	7	13	0.5002	792.766	70.91
Kæ-6.5*C*8a	6/9/93 0907	6.46 21.4C	7.3327	7.8335	7	14	0.5008	796.161	71.13
Kæ-6.5*C*9a	6/11/93 0904	6.53 21.0C	7.3368	7.8366	7	15	0.4998	785.356	70.3
Kæ-6.5*C*9b	6/11/93 0904	6.53 21.0C	7.3503	7.8481	7	16	0.4978	763.088	68.59
Kæ-6.5*C*10a	6/14/93 1018	6.50 20.2C	7.2284	7.778	19	13	0.4996	744.665	66.69
Kæ-6.5*C*11a	6/17/93 0935	6.51 21.4C	7.7686	8.2708	4	13	0.5022	753.24	67.11
Kæ-6.5*C*12a	6/21/93 1009	6.52 23.3C	7.2394	7.7386	7	7	0.4992	745.586	66.82
Kæ-6.5*C*12b	6/21/93 1009	6.52 23.3C	7.2895	7.7862	7	8	0.4967	749.895	67.55

Identification	Date/Time	pH	Vial wt g	Sam +vial wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
Kæ-4*8005*13a	7/1/93 0907	6.18 21.3C	7.7292	8.2288	2	2	0.4996	304.647	27.28
Kæ-4*8005*13b	7/1/93 0907	6.18 21.3C	7.8154	8.3165	2	3	0.5011	302.469	27.01
Kæ-4*8006*13a	7/1/93 0908	6.05 21.3C	7.7518	8.2534	2	4	0.5016	1111.01	99.1
Kæ-4*8006*13b	7/1/93 0908	6.05 21.3C	7.7545	8.2553	2	5	0.5008	1102.18	98.47
Kæ-4*8007*13a	7/1/93 0909	4.04 21.3C	7.7287	8.2291	2	6	0.5004	1325.93	118.55
Kæ-4*8007*13b	7/1/93 0909	4.04 21.3C	7.7361	8.2383	2	7	0.5022	1336.73	119.09
Kæ-4*C*13a	7/1/93 0911	3.98 21.3C	7.7668	8.2665	2	8	0.4997	1233.36	110.43
Kæ-4*C*13b	7/1/93 0911	3.98 21.3C	7.7342	8.2376	2	9	0.5034	1237.08	109.95
Kæ-5.5*8005*13a	7/1/93 0856	6.97 21.4C	7.8017	8.3059	2	11	0.5042	387.824	34.41
Kæ-5.5*8005*13b	7/1/93 0856	6.97 21.4C	7.8007	8.3055	2	12	0.5048	379.415	33.63
Kæ-5.5*8006*13a	7/1/93 0852	6.87 21.4C	7.7282	8.2391	2	13	0.5109	720.548	63.1
Kæ-5.5*8006*13b	7/1/93 0852	6.87 21.4C	7.8189	8.3222	2	14	0.5033	720.233	64.03
Kæ-5.5*8007*13a	7/1/93 0858	5.74 21.3C	7.7549	8.2616	2	15	0.5067	779.045	68.79
Kæ-5.5*8007*13b	7/1/93 0858	5.74 21.3C	7.8032	8.3081	2	16	0.5049	789.484	69.96
Kæ-5.5*C*13a	7/1/93 0910	5.40 21.3C	7.7578	8.2589	2	17	0.5011	1122.29	100.21
Kæ-5.5*C*13b	7/1/93 0910	5.40 21.3C	7.7865	8.2912	2	18	0.5047	1097.22	97.27
Kæ-6.5*8005*13a	7/1/93 0850	7.16 21.4C	7.7995	8.3029	3	2	0.5034	410.54	36.49
Kæ-6.5*8005*13b	7/1/93 0850	7.16 21.4C	7.7737	8.2765	3	3	0.5028	411.91	36.65
Kæ-6.5*8006*13a	7/1/93 0855	7.05 21.4C	7.7657	8.2659	3	4	0.5002	709.15	63.43
Kæ-6.5*8006*13b	7/1/93 0855	7.05 21.4C	7.7899	8.3004	3	5	0.5105	740.449	64.89
Kæ-6.5*8007*13a	7/1/93 0854	6.65 21.4C	7.7508	8.256	3	6	0.5052	658.876	58.35
Kæ-6.5*8007*13b	7/1/93 0854	6.65 21.4C	7.7879	8.2891	3	7	0.5012	656.615	58.62
Kæ-6.5*C*13a	7/1/93 0906	6.53 21.4C	7.7867	8.2882	3	8	0.5015	735.177	65.59
Kæ-6.5*C*13b	7/1/93 0906	6.53 21.4C	7.7767	8.2852	3	9	0.5085	773.934	68.1

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6/10/93

URANIUM SORPTION EXPERIMENT B-8005B

Kd vs pH; Equilibrium with atmospheric pCO_2; Initial ΣU=100 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: May 31, 1993

OBJECTIVE: to investigate the importance of solid surface area and pH on uranium sorption on alpha-alumina. Experimental data will also be correlated with uranium aqueous speciation.

EQUIPMENT:
- Gyratory shaker
- Liquid scintillation analyzer (Packard 1900 TR)
- ORION ph/mV/ISE/°C meter
- Combination ph electrode
- Automatic termperature compensator probe
- Analytical balance
- Fisher Marathon 21K Centrifuge and 50 ml centrifuge tube adaptors

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
2	50 ml FEP centrifuge tubes (to contain B-8005B*IU1 and B-8005B*IU2)
30	50 ml FEP centrifuge tubes (to contain experimental mixtures and control solutions)
1	2000 ml FEP bottle (for preparation of 100 ppb U solution)
	weighing paper
	Eppendorf pipets and tips (for adjusting pH and taking samples)
	8005 Alpha Alumina (surface area 2.09 m^2/g; from NIST)
	reagent grade $NaHCO_3$ (Lot 897186A)
	500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike (spike #23A)
	7 ml LSA vials
2L	0.1 m $NaNO_3$ stock solution (lot 7808 KCCL)
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3
.1L	stock solution of 3.0 m $NaHCO_3$
.1L	stock solution of 2.0 m $NaHCO_3$
.5L	stock solution of 1.0 m $NaHCO_3$
.5L	stock solution of 0.5 m $NaHCO_3$
.5L	stock solution of 0.1 m $NaHCO_3$
.5L	stock solution of 0.05 m $NaHCO_3$
.5L	stock solution of 0.01 m $NaHCO_3$
.5L	stock solution of 0.005 m $NaHCO_3$
	ultrapure water

PREPARATION:

1. Preclean:
   32 50 ml FEP centrifuge tubes
   1 2L FEP bottle

PROCEDURE:

Note: In transferrring uranium solutions, avoid using glass or polypropylene labware.

Solution B-8005B (1 centrifuge tube for each pH value)

- Initial ΣU = 100 ppb
- Initial pH = 2.0 to 9.0; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 40 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8005 alpha alumina to use = 0.100±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

6/10/93 a) Into a pre-cleaned 2L FEP bottle, prepare 1280 g of 100 ppb U solution by diluting 256 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 1280 g by carefully taring 0.1 m $NaNO_3$ solution into the plastic bottles on a Mettler 4600 balance.

1340 b) Into each of thirty 50 ml FEP centrifuge tubes labeled B-8005B*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 40 g of the 100 ppb U solution.

Transfer the remaining solution into two 50 ml FEP centrifuge tubes labeled B-8005B*IU1 and B-8005B*IU2. Take two 0.5 ml samples from B-8005B*IU1 and B-8005B*IU2 with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8005B-IU1*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of the sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

1440 c) For each solution B-8005B*pH*i*, where *i*≤3.85:

Adjust the pH of each solution to the approximate value *i* by adding HNO_3 solution or $NaHCO_3$ solution with an Eppendorf micropipet. The concentration and approximate amount to be added is given in Table B-8005B-1. Swril the solutions by hand. Record the micropipet volume and concentration of solution added. *Do not measure the pH at this time.* Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to about 120 rpm. Leave the tubes on the shaker for about ten days to allow the solutions to reach equilibrium with atomospheric $CO_2(g)$.

6/24/93 d) 0825 hrs Measure and record the pH of each solution B-8005B*pH*i*. *Minimize the amount of time the glass electrode is in contact with the uranium solution. Make sure to rinse the electrode well before transferring into another solution.*

6/24/93 1300 hrs From each solution B-8005B*pH*i*, take two 0.5 ml sample with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8005B*IU-pH*i**a (or b)] and pre-weighed scintillation vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. The measured concentrations are the initial values to be used in the calculation of sorption data.

1355 hrs e) Tare 0.100±0.001 g of 8005 alpha alumina onto weighing paper, and carefully transfer into each of the B-8005B*pH*i* centrifuge tubes. Replace the porous cover and replace on the shaker.

f) 7/15/93 0900 hrs After equilibrium is reached (at least 10 days), centrifuge each tube at 15,000 rpm for 30 min to separate the aqueous phase from the solid. Take two 0.5 ml samples from each centrifuge tube B-8005B*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8005B*pH*i**a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling.

Measure and record the pH and temperature of solutions B-8005B*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

g) After all samples are taken, take two 0.5 ml samples from B-8005B*IU1 and B-8005B*IU2 using an Eppendorf pipet and transfer into prelabled (B-8005B*FU1*a (or b)) and preweighed LSA vials. Reweigh vial after addition of sample, add 0.5 ml 0.02 m HNO_3, and analyze by LSA within the same day as sampling.

h) If the analytical results are good, reversibility and reproducibility tests can be done by changing the pH of the solutions and re-equilibrating them at the new pH values.

Procedure for reversibility and reproducibility experiments will be written later.

Table B-8005B-1. Amount of reagent grade HNO_3 or $NaHCO_3$ to add to 40 ml 0.1 m $NaNO_3$ solution containing 100 ppb U to result in pH values given in first column. The amount of reagent added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atomospheric $CO_2(g)$.

Solution pH	Volume HNO_3 added (ml)	Molarity of HNO_3
2.00	0.42	1.0
2.25	0.26	1.0
2.50	0.14	1.0
2.75	0.07	1.0
2.90	0.47	0.1
3.00	0.37	0.1
3.05	0.32	0.1
3.10	0.27	0.1
3.20	0.22	0.1
3.30	0.16	0.1
3.40	0.12	0.1
3.50	0.07	0.1
3.70	0.02	0.1
Solution pH	**Vol of $NaHCO_3$ added (ml)**	**Molarity of $NaHCO_3$**
3.90	0.20	0.005
4.00	0.40	0.005
4.05	0.25	0.01
4.15	0.32	0.01
4.50	0.10	0.05
5.00	0.12	0.05
5.50	0.15	0.05
6.00	0.22	0.05
6.50	0.20	0.1
7.00	0.50	0.1
7.25	0.08	1.0
7.50	0.12	1.0
7.75	0.19	1.0
8.00	0.34	1.0
8.25	0.07	2.0
8.62	0.11	2.0
9.00	0.12	3.0

Table B-8005B

Sample	Target pH	Wt. 233U soln added (g)	pH date/time		pH date/time	
			6/24/93	0825	7/15/93	1400
B-8005B*pH2.00	2.00	40.01	1.96	21.1°C	1.99	23.3°C
B-8005B*pH2.25	2.25	40.01	2.15	21.1°C	2.18	23.3°C
B-8005B*pH2.50	2.50	40.00	2.41	21.1°C	2.46	23.3°C
B-8005B*pH2.75	2.75	40.00	2.69	21.2°C	2.77	23.3°C
B-8005B*pH2.90	2.90	40.01	2.84	21.2°C	2.94	23.4°C
B-8005B*pH3.00	3.00	40.02	2.90	21.2°C	2.99	23.4°C
B-8005B*pH3.05	3.05	40.01	3.03	21.2°C	3.14	23.4°C
B-8005B*pH3.10	3.10	40.00	3.07	21.2°C	3.24	23.4°C
B-8005B*pH3.20	3.20	40.01	3.14	21.2°C	3.37	23.4°C
B-8005B*pH3.30	3.30	40.01	3.23	21.3°C	3.55	23.5°C
B-8005B*pH3.40	3.40	40.00	3.32	21.3°C	3.68	23.5°C
B-8005B*pH3.50	3.50	40.01	3.45	21.3°C	3.92	23.5°C
B-8005B*pH3.70	3.70	40.01	3.67	21.3°C	4.42	23.6°C
B-8005B*pH3.90	3.90	40.01	3.86	21.3°C	5.11	23.8°C
B-8005B*pH4.00	4.00	40.02	3.97	21.3°C	5.58	23.8°C
B-8005B*pH4.05	4.05	40.01	4.03	21.5°C	5.87	23.7°C
B-8005B*pH4.15	4.15	40.02	4.11	21.5°C	6.30	23.9°C
B-8005B*pH4.50	4.50	40.02	4.46	21.5°C	6.75	24.0°C
B-8005B*pH5.00	5.00	40.03	4.92	21.5°C	6.89	24.0°C
B-8005B*pH5.50	5.50	40.03	6.34	21.6°C	7.14	24.1°C
B-8005B*pH6.00	6.00	40.01	6.97	21.6°C	7.47	24.2°C
B-8005B*pH6.50	6.50	40.02	7.52	21.6°C	7.71	24.3°C
B-8005B*pH7.00	7.00	40.02	8.03	21.7°C	8.18	24.3°C
B-8005B*pH7.25	7.25	40.03	8.26	21.7°C	8.34	24.3°C
B-8005B*pH7.50	7.50	40.01	8.46	21.7°C	8.46	24.2°C
B-8005B*ph7.75	7.75	40.02	8.65	21.7°C	8.67	24.2°C
B-8005B*pH8.00	8.00	40.02	8.89	21.7°C	8.86	24.2°C
B-8005B*ph8.25	8.25	40.02	8.48	21.7°C	8.51	24.2°C
B-8005B*pH8.62	8.62	40.02	8.69	21.7°C	8.71	24.1°C
B-8005B*pH9.00	9.00	40.02	8.82	21.7°C	8.83	24.1°C
B-8005B*IU1		39.98	3.77	21.8°C	3.77	23.2°C
B-8005B*IU2		39.24	3.76	21.8°C	3.80	23.2°C

B-8005B LSA sample weight

Identification	Date/Time	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U (ppb)
B-8005B*IU1*a	6/10/93 1420	7.3676	7.8674	8	6	.4998	1067.07	95.52
B-8005B*IU1*b	6/10/90 1420	7.2974	7.7972	8	7	.4998	1094.41	97.97
B-8005B*IU2*a	6/10/93 1423	7.3869	7.884	8	8	.4971	1041.25	93.72
B-8005B*IU2*b	6/10/93 1423	7.3582	7.8582	8	9	.5000	1052.70	94.20
B-8005B*IU*pH2.00*a	6/24/93 1304	7.6823	8.1852	11	2	.5029	1093.60	97.29
B-8005B*IU*pH2.00*b	1304	7.7543	8.2559	11	3	.5016	1135.67	101.30
B-8005B*IU*pH2.25*a	1306	7.7747	8.2767	11	4	.502	1119.27	99.86
B-8005B*IU*pH2.25*b	1306	7.7381	8.2396	11	5	.5015	1133.62	101.14
B-8005B*IU*pH2.50*a	1308	7.7955	8.2987	11	6	.5032	1118.46	99.45
B-8005B*IU*pH2.50*b	1308	7.7709	8.2716	11	7	.5007	1113.09	99.46
B-8005B*IU*pH2.75*a	1310	7.8143	8.3151	11	8	.5008	1118.46	99.92
B-8005B*IU*pH2.75*b	1310	7.7715	8.2697	11	9	.4982	1117.70	100.38
B-8005B*IU*pH2.90*a	1311	7.8401	8.3405	11	10	.5004	1132.25	101.24
B-8005B*IU*pH2.90*b	1311	7.7417	8.2461	11	11	.5044	1114.38	98.85
B-8005B*IU*pH3.00*a	1312	7.7404	8.2417	11	12	.5013	1097.30	97.94
B-8005B*IU*pH3.00*b	1312	7.7063	8.2696	11	13	.5033	1136.79	101.06
B-8005B*IU*pH3.05*a	1313	7.7572	8.2567	11	14	.4995	1089.92	97.63
B-8005B*IU*pH3.05*b	1313	7.7057	8.2050	11	15	.4993	1079.29	96.71
B-8005B*IU*pH3.10*a	1314	7.7747	8.2774	11	16	.5027	1128.85	100.47
B-8005B*IU*pH3.10*b	1314	7.7583	8.2615	11	17	.5032	1117.20	99.34
B-8005B*IU*pH3.20*a	1315	7.6880	8.1929	11	18	.5049	1127.50	99.91
B-8005B*IU*pH3.20*b	1315	7.7816	8.2835	12	1	.5019	1130.13	100.75
B-8005B*IU*pH3.30*a	1316	7.7408	8.2457	12	2	.5049	1130.72	100.20
B-8005B*IU*pH3.30*b	1316	7.7396	8.2420	12	3	.5024	1083.39	96.48
B-8005B*IU*pH3.40*a	1317	7.7157	8.2170	12	4	.5013	1073.08	95.77
B-8005B*IU*pH3.40*b	1317	7.7657	8.2691	12	5	.5034	1137.82	101.13
B-8005B*IU*pH3.50*a	1318	7.7479	8.2486	12	6	.5007	1103.02	98.56
B-8005B*IU*pH3.50*b	1318	7.7961	8.2978	12	7	.5017	1134.29	101.16
B-8005B*IU*pH3.70*a	1319	7.7968	8.3002	12	8	.5034	1145.37	101.80
B-8005B*IU*pH3.70*b	1319	7.8197	8.3198	12	9	.5001	1134.90	101.54
B-8005B*IU*pH3.90*a	1320	7.7695	8.2705	12	10	.5010	1097.05	97.97

B-8005B LSA sample weight

B-8005B*IU*pH3.90*b	6/24/93 1320	7.7865	8.2870	12	11	.5005	1113.34	99.53
B-8005B*IU*pH4.00*a	1321	7.7210	8.2252	12	12	.5042	1110.30	98.53
B-8005B*IU*pH4.00*b	1321	7.8065	8.3118	12	13	.5053	1120.28	99.20
B-8005B*IU*pH4.05*a	1322	7.7661	8.2710	12	14	.5049	1100.27	97.50
B-8005B*IU*pH4.05*b	1322	7.7838	8.2854	12	15	.5016	1084.82	96.76
B-8005B*IU*pH4.15*a	1323	7.8100	8.3142	12	16	.5042	1145.88	101.68
B-8005B*IU*pH4.15*b	1323	7.7779	8.2814	12	17	.5035	1089.43	96.81
B-8005B*IU*pH4.50*a	1324	7.7487	8.2507	12	18	.5020	1125.73	100.33
B-8005B*IU*pH4.50*b	1324	7.7990	8.3014	13	1	.5024	1091.88	97.24
B-8005B*IU*pH5.00*a	1325	7.8095	8.3127	13	2	.5032	1081.19	96.13
B-8005B*IU*pH5.00*b	1325	7.7453	8.2493	13	3	.5040	1103.27	97.94
B-8005B*IU*pH5.50*a	1326	7.7085	8.2110	13	4	.5025	1048.35	93.34
B-8005B*IU*pH5.50*b	1326	7.7055	8.2122	13	5	.5067	1042.12	92.02
B-8005B*IU*pH6.00*a	1327	7.7624	8.2652	13	6	.5028	1090.41	97.03
B-8005B*IU*pH6.00*b	1327	7.7609	8.2627	13	7	.5018	1080.02	96.30
B-8005B*IU*pH6.50*a	1328	7.7773	8.2791	13	8	.5018	1087.49	96.96
B-8005B*IU*pH6.50*b	1328	7.7659	8.2672	13	9	.5013	1098.04	98.00
B-8005B*IU*pH7.00*a	1329	7.7118	8.2137	13	10	.5019	1119.27	99.78
B-8005B*IU*pH7.00*b	1329	7.7428	8.2440	13	11	.5012	1091.88	97.47
B-8005B*IU*pH7.25*a	1330	7.7604	8.2602	13	12	.4998	1119.27	100.20
B-8005B*IU*pH7.25*b	1330	7.7359	8.2360	13	13	.5001	1106.27	98.97
B-8005B*IU*pH7.50*a	1331	7.7775	8.2816	13	14	.5041	1122.87	99.66
B-8005B*IU*pH7.50*b	1331	7.8206	8.3235	13	15	.5029	1111.83	98.92
B-8005B*IU*pH7.75*a	1332	7.7749	8.2757	13	16	.5008	1104.51	98.68
B-8005B*IU*pH7.75*b	1332	7.7716	8.2730	13	17	.5014	1117.20	99.69
B-8005B*IU*pH8.00*a	1333	7.7292	8.2315	13	18	.5023	1120.22	99.78
B-8005B*IU*pH8.00*b	1333	7.7791	8.2805	14	1	.5014	1127.84	100.64
B-8005B*IU*pH8.25*a	1334	7.7598	8.2620	14	2	.5022	1122.30	99.99
B-8005B*IU*pH8.25*b	1334	7.7226	8.2285	14	3	.5059	1157.88	102.40
B-8005B*IU*pH8.62*a	1335	7.7556	8.2576	14	4	.5020	1098.53	97.91
B-8005B*IU*pH8.62*b	1335	7.8083	8.3148	14	5	.5065	1131.23	99.93
B-8005B*IU*pH9.00*a	1336	7.7511	8.2552	14	6	.5041	1148.60	101.95
B-8005B*IU*pH9.00*b	1336	7.7753	8.2796	14	7	.5043	1076.66	95.52

B-8005B LSA sample weight

B-8005B*pH2.00*a	7/15/93 0928	7.7758	8.2748	1	2	0.499	1158.97	103.92
B-8005B*pH2.00*b	0928	7.7895	8.2888	1	3	0.4993	1184.03	106.10
B-8005B*pH2.25*a	0930	7.7666	8.2660	1	4	0.4994	1201.17	107.61
B-8005B*pH2.25*b	0930	7.8119	8.3102	1	5	0.4983	1166.91	104.78
B-8005B*pH2.50*a	0932	7.8080	8.3079	1	6	0.4999	1167.36	104.48
B-8005B*pH2.50*b	0932	7.7631	8.2626	1	7	0.4995	1170.71	104.86
B-8005B*pH2.75*a	0933	7.7430	8.2419	1	8	0.4999	1179.06	105.74
B-8005B*pH2.75*b	0933	7.7654	8.2660	1	9	0.5006	1180.08	105.47
B-8005B*pH2.90*a	0935	7.8033	8.3031	1	10	0.4998	1174.61	105.15
B-8005B*pH2.90*b	0935	7.7869	8.2865	1	11	0.4996	1167.89	104.59
B-8005B*pH3.00*a	0937	7.8085	8.3071	1	12	0.4986	1179.55	105.85
B-8005B*pH3.00*b	0937	7.7837	8.2816	1	13	0.4979	1183.50	106.35
B-8005B*pH3.05*a	0950	7.8148	8.3110	1	14	0.4962	1149.03	103.61
B-8005B*pH3.05*b	0950	7.7659	8.2607	1	15	0.4948	1146.70	103.69
B-8005B*pH3.10*a	0951	7.8264	8.3218	1	16	0.4954	1175.61	106.17
B-8005B*pH3.10*b	0951	7.7761	8.2710	1	17	0.4949	1151.38	104.09
B-8005B*pH3.20*a	0952	7.7707	8.2648	1	18	0.4941	1166.65	105.64
B-8005B*pH3.20*b	0952	7.7846	8.2793	4	1	0.4947	1170.71	105.88
B-8005B*pH3.30*a	0954	7.7801	8.2765	4	2	0.4964	1153.46	103.96
B-8005B*pH3.30*b	0954	7.7632	8.2586	4	3	0.4954	1179.55	106.53
B-8005B*pH3.40*a	0955	7.7573	8.2532	4	4	0.4959	1151.38	103.88
B-8005B*pH3.40*b	0955	7.7713	8.2668	4	5	0.4955	1176.94	106.27
B-8005B*pH3.50*a	0957	7.7496	8.2455	4	6	0.4959	1122.240	101.27
B-8005B*pH3.50*b	0957	7.8258	8.3213	4	7	0.4955	1141.56	103.08
B-8005B*pH3.70*a	1016	7.8048	8.3021	4	8	0.4973	1058.41	95.22
B-8005B*pH3.70*b	1016	7.7481	8.2450	4	9	0.4969	1094.86	98.58
B-8005B*pH3.90*a	1018	7.8059	8.3028	4	10	0.4969	874.939	78.78
B-8005B*pH3.90*b	1018	7.7991	8.2946	4	11	0.4955	889.805	80.35
B-8005B*pH4.00*a	1019	7.7521	8.2487	4	12	0.4966	663.700	59.80
B-8005B*pH4.00*b	1019	7.7966	8.2933	4	13	0.4967	693.541	62.47
B-8005B*pH4.05*a	1020	7.7977	8.2934	4	14	0.4957	533.621	48.11
B-8005B*pH4.05*b	1020	7.7566	8.2532	4	15	0.4966	539.568	48.61

B-8005B LSA sample weight

B-8005B*pH4.15*a	1023	7.8055	8.3012	4	16	0.4957	416.871	37.63
B-8005B*pH4.15*b	1023	7.7654	8.2607	4	17	0.4953	416.965	37.66
B-8005B*pH4.50*a	1025	7.7871	8.2830	4	18	0.4959	454.652	41.02
B-8005B*pH4.50*b	1025	7.8250	8.3227	6	1	0.4977	486.591	43.74
B-8005B*pH5.00*a	1045	7.7757	8.2693	6	2	0.4936	487.341	44.17
B-8005B*pH5.00*b	1045	7.8406	8.3343	6	3	0.4937	509.866	46.21
B-8005B*pH5.50*a	1046	7.8450	8.3387	6	4	0.4937	513.693	46.55
B-8005B*pH5.50*b	1046	7.7480	8.2413	6	5	0.4933	529.687	48.04
B-8005B*pH6.00*a	1047	7.7738	8.2694	6	6	0.4956	652.880	58.94
B-8005B*pH6.00*b	1047	7.7774	8.2718	6	7	0.4944	642.116	58.11
B-8005B*pH6.50*a	1048	7.8097	8.3042	6	8	0.4945	857.375	77.57
B-8005B*pH6.50*b	1048	7.8085	8.3037	6	9	0.4952	848.366	76.65
B-8005B*pH7.00*a	1049	7.8555	8.3502	6	10	0.4947	1095.11	99.04
B-8005B*pH7.00*b	1049	7.7615	8.2564	6	11	0.4949	1108.58	100.22
B-8005B*pH7.25*a	1050	7.7752	8.2716	6	12	0.4964	1120.07	100.95
B-8005B*pH7.25*b	1050	7.8153	8.3106	6	13	0.4953	1132.55	102.31
B-8005B*pH7.50*a	1110	7.8265	8.3217	6	14	0.4952	1212.95	109.95
B-8005B*pH7.50*b	1110	7.8688	8.3627	6	15	0.4939	1173.02	106.26
B-8005B*pH7.75*a	1111	7.8066	8.3010	6	16	0.4944	1189.06	107.61
B-8005B*pH7.75*b	1111	7.8043	8.2983	6	17	0.4940	1188.79	107.67
B-8005B*pH8.00*a	1112	7.8181	8.3133	6	18	0.4952	1173.29	106.01
B-8005B*pH8.00*b	1112	7.8084	8.3034	7	1	0.4950	1165.86	105.38
B-8005B*pH8.25*a	1113	7.8398	8.3331	7	2	0.4933	1168.94	106.02
B-8005B*pH8.25*b	1113	7.7525	8.2458	7	3	0.4933	1173.29	106.42
B-8005B*pH8.62*a	1114	7.7319	8.2268	7	4	0.4949	1173.82	106.12
B-8005B*pH8.62*b	1114	7.7680	8.2627	7	5	0.4947	1143.74	103.44
B-8005B*pH9.00*a	1115	7.7921	8.2865	7	6	0.4944	1190.67	107.75
B-8005B*pH9.00*b	1115	7.8271	8.3220	7	7	0.4949	1167.10	105.51
B-8005B*FU1*a	1030	7.7441	8.2477	7	9	0.5036	1171.24	104.06
B-8005B*FU1*b	1030	7.7227	8.2251	7	10	0.5024	1173.55	104.51
B-8005B*FU2*a	1031	7.8217	8.3249	7	11	0.5032	1182.17	105.11
B-8005B*FU2*b	1031	7.8018	8.3040	7	12	0.5022	1173.55	104.55

7/6/93 SP

URANIUM SORPTION EXPERIMENT B-8006B

Kd vs pH: Equilibrium with atmospheric pCO_2: Initial ΣU=100 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: May 31, 1993

OBJECTIVE: to investigate the importance of solid surface area and pH on uranium sorption on alpha-alumina. Experimental data will also be correlated with uranium aqueous speciation.

EQUIPMENT:
Gyratory shaker
Liquid scintillation analyzer (Packard 1900 TR)
ORION ph/mV/ISE/°C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance
Fisher Marathon 21K Centrifuge and 50 ml centrifuge tube adaptors

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
2	50 ml FEP centrifuge tubes (to contain B-8006B*IU1 and B-8006B*IU2)
30	50 ml FEP centrifuge tubes (to contain experimental mixtures and control solutions)
1	2000 ml FEP bottle (for preparation of 100 ppb U solution)
	weighing paper
	Eppendorf pipets and tips (for adjusting pH and taking samples)
	8006 Alpha Alumina (surface area 0.229 m^2/g; from NIST)
	reagent grade $NaHCO_3$ (Lot 897186A)
	500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike (spike #23A)
	7 ml LSA vials
2L	0.1 m $NaNO_3$ stock solution (lot 7808 KCCL)
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3
.1L	stock solution of 3.0 m $NaHCO_3$
.1L	stock solution of 2.0 m $NaHCO_3$
.5L	stock solution of 1.0 m $NaHCO_3$
.5L	stock solution of 0.5 m $NaHCO_3$
.5L	stock solution of 0.1 m $NaHCO_3$
.5L	stock solution of 0.05 m $NaHCO_3$
.5L	stock solution of 0.01 m $NaHCO_3$
.5L	stock solution of 0.005 m $NaHCO_3$
	ultrapure water

PREPARATION:

1. Preclean:
   32 50 ml FEP centrifuge tubes
   1 2L FEP bottle

PROCEDURE:

Note: In transferrring uranium solutions, avoid using glass or polypropylene labware.

Solution B-8006B (1 centrifuge tube for each pH value)

- Initial ΣU = 100 ppb
- Initial pH = 2.0 to 9.0; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 40 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8006 alpha alumina to use = 0.100±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

7/6/93 1330hrs JP

a) Into a pre-cleaned 2L FEP bottle, prepare 1280 g of 100 ppb U solution by diluting 256 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 1280 g by carefully taring 0.1 m $NaNO_3$ solution into the plastic bottles on a Mettler 4600 balance.

b) Into each of thirty 50 ml FEP centrifuge tubes labeled B-8006B*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 40 g of the 100 ppb U solution.

Transfer the remaining solution into two 50 ml FEP centrifuge tubes labeled B-8006B*IU1 and B-8006B*IU2. Take two 0.5 ml samples from B-8006B*IU1 and B-8006B*IU2 with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8006B-IU1*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of the sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

c) For each solution B-8006B*pH*i*, where $i \leq 3.85$:

Adjust the pH of each solution to the approximate value *i* by adding HNO_3 solution or $NaHCO_3$ solution with an Eppendorf micropipet. The concentration and approximate amount to be added is given in Table B-8006B-1. Swril the solutions by hand. Record the micropipet volume and concentration of solution added. *Do not measure the pH at this time.* Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to about 120 rpm. Leave the tubes on the shaker for about ten days to allow the solutions to reach equilibrium with atomospheric $CO_2(g)$.

7/20/93 0900hrs JP

d) Measure and record the pH of each solution B-8006B*pH*i*. *Minimize the amount of time the glass electrode is in contact with the uranium solution. Make sure to rinse the electrode well before transferring into another solution.*

From each solution B-8006B*pH*i*, take two 0.5 ml sample with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8006B*IU-pH*i*a (or b)] and pre-weighed scintillation vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. The measured concentrations are the initial values to be used in the calculation of sorption data.

7/20/93 1730hrs JP

e) Tare 0.100±0.001 g of 8006 alpha alumina onto weighing paper, and carefully transfer into each of the B-8006B*pH*i* centrifuge tubes. Replace the porous cover and replace on the shaker.

8/10/93 0800 hrs JP

After equilibrium is reached (at least 10 days), centrifuge each tube at 15,000 rpm for 30 min to separate the aqueous phase from the solid. Take two 0.5 ml samples from each centrifuge tube B-8006B*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8006B*pH*i*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling.

Measure and record the pH and temperature of solutions B-8006B*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

g) After all samples are taken, take two 0.5 ml samples from B-8006B*IU1 and B-8006B*IU2 using an Eppendorf pipet and transfer into prelabled (B-8006B*FU1*a (or b)) and preweighed LSA vials. Reweigh vial after addition of sample, add 0.5 ml 0.02 m HNO_3, and analyze by LSA within the same day as sampling.

h) If the analytical results are good, reversibility and reproducibility tests can be done by changing the pH of the solutions and re-equilibrating them at the new pH values.

Procedure for reversibility and reproducibility experiments will be written later.

Table B-8006B-1. Amount of reagent grade HNO_3 or $NaHCO_3$ to add to 40 ml 0.1 m $NaNO_3$ solution containing ~100 ppb U to result in pH values given in first column. The amount of reagent added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atomospheric $CO_2(g)$.

Solution pH	Volume HNO_3 added (ml)	Molarity of HNO_3
2.00	0.40	1.0
2.37	0.16	1.0
2.75	0.07	1.0
3.00	0.35	0.1
3.10	0.27	0.1
3.25	0.16	0.1
3.40	0.10	0.1
3.55	0.05	0.1
3.70	0.02	0.1
3.80	0.00	

Solution pH	Vol of $NaHCO_3$ added (ml)	Molarity of $NaHCO_3$
3.90	0.20	0.005
4.00	0.40	0.005
4.05	0.25	0.01
4.10	0.32	0.01
4.15	0.34	0.01
4.20	0.37	0.01
4.30	0.40	0.01
4.50	0.11	0.05
5.00	0.12	0.05
5.50	0.13	0.05
6.00	0.14	0.05
6.50	0.15	0.05
7.00	0.19	0.05
7.25	0.23	0.05
7.50	0.31	0.05
7.75	0.23	0.1
8.00	0.36	0.1
8.25	0.12	0.5
8.62	0.13	1.0
9.00	0.35	1.0

Table B-8006B

Sample	Target pH	Wt. 233U soln added (g)	pH date/time		pH date/time	
			7/20/93	1110	8/10/93	1400
B-8006B*pH2.00	2.00	40.01	2.03	21.7°C	1.98	25.6°C
B-8006B*pH2.37	2.37	40.02	2.35	21.7°C	2.35	25.6°C
B-8006B*pH2.75	2.75	40.00	2.69	21.8°C	2.69	25.7°C
B-8006B*pH3.00	3.00	40.03	2.94	21.8°C	2.97	25.7°C
B-8006B*pH3.10	3.10	39.99	3.04	21.8°C	3.07	25.7°C
B-8006B*pH3.25	3.25	39.99	3.23	21.9°C	3.30	25.6°C
B-8006B*pH3.40	3.40	40.01	3.36	21.9°C	3.46	25.6°C
B-8006B*pH3.55	3,55	40.00	3.54	21.9°C	3.68	25.6°C
B-8006B*pH3.70	3.70	39.99	3.65	21.9°C	3.88	25.6°C
B-8006B*pH3.80	3.80	40.01	3.74	22.0°C	4.14	24.9°C
B-8006B*pH3.90	3.90	40.01	3.85	22.0°C	4.34	24.8°C
B-8006B*pH4.00	4.00	40.00	3.99	23.3°C	4.61	24.8°C
B-8006B*pH4.05	4.05	40.01	4.01	23.4°C	4.95	24.8°C
B-8006B*pH4.10	4.10	39.99	4.09	23.4°C	5.55	25.0°C
B-8006B*pH4.15	4.15	40.02	4.12	23.4°C	5.78	25.0°C
B-8006B*pH4.20	4.20	40.02	4.16	23.4°C	6.04	25.1°C
B-8006B*pH4.30	4.30	40.03	4.21	23.4°C	6.19	25.1°C
B-8006B*pH4.50	4.50	40.01	4.57	23.4°C	6.73	25.1°C
B-8006B*pH5.00	5.00	40.00	4.81	23.5°C	6.85	25.1°C
B-8006B*pH5.50	5.50	40.00	5.72	23.5°C	6.92	25.1°C
B-8006B*pH6.00	6.00	40.00	6.07	23.6°C	6.97	25.2°C
B-8006B*pH6.50	6.50	39.99	6.44	24.7°C	7.04	25.2°C
B-8006B*pH7.00	7.00	39.99	6.85	24.6°C	7.17	23.5°C
B-8006B*pH7.25	7.25	40.00	7.09	24.6°C	7.26	23.4°C
B-8006B*pH7.50	7.50	40.00	7.40	24.5°C	7.53	23.4°C
B-8006B*ph7.75	7.75	39.99	7.68	24.5°C	7.73	23.3°C
B-8006B*pH8.00	8.00	39.99	7.92	24.5°C	7.97	23.3°C
B-8006B*ph8.25	8.25	39.99	8.21	24.4°C	8.22	23.2°C
B-8006B*pH8.62	8.62	40.01	8.55	24.4°C	8.56	23.2°C
B-8006B*pH9.00	9.00	40.02	8.95	24.5°C	8.98	23.2°C
B-8006B*IU1		39.96	3.77	23.2°C	3.74	25.6°C
B-8006B*IU2		39.50	3.78	23.2°C	3.73	25.6°C

B-8006B LSA sample weight

Identification	Date/Time	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U (ppb)
B-8006B*IU1*a	7/6/93 1400	7.8089	8.3071	2	2	.4982	1079.10	96.91
B-8006B*IU1*b	7/6/93 1400	7.8210	8.3202	2	3	.4992	1041.93	93.39
B-8006B*IU2*a	7/6/93 1400	7.7727	8.2722	2	4	.4995	1047.10	93.79
B-8006B*IU2*b	7/6/93 1400	7.7767	8.2754	2	5	.4987	1065.85	95.63
B-8006B*IU*pH2.00*a	7/20/93 0900	7.7806	8.2839	11	2	.5033	1114.22	99.05
B-8006B*IU*pH2.00*b		7.8056	8.3074		3	.5018	1141.61	101.79
B-8006B*IU*pH2.37*a		7.8213	8.3238		4	.5025	1126.32	100.29
B-8006B*IU*pH2.37*b		7.7804	8.2830		5	.5026	1122.70	99.94
B-8006B*IU*pH2.75*a		7.8130	8.3127		6	.4997	1114.22	99.76
B-8006B*IU*pH2.75*b		7.7812	8.2810		7	.4998	1141.35	102.17
B-8006B*IU*pH3.00*a		7.8870	8.3866		8	.4996	1123.71	100.63
B-8006B*IU*pH3.00*b		7.8145	8.3162		9	.5017	1122.70	100.12
B-8006B*IU*pH3.10*a		7.8246	8.3255		10	.5009	1129.19	100.86
B-8006B*IU*pH3.10*b		7.8006	8.3015		11	.5009	1128.69	100.81
B-8006B*IU*pH3.25*a		7.8401	8.3413		12	.5012	1136.01	101.41
B-8006B*IU*pH3.25*b		7.7623	8.2631		13	.5008	1170.49	104.57
B-8006B*IU*pH3.40*a		7.7644	8.2644		14	.5000	1112.42	99.54
B-8006B*IU*pH3.40*b		7.7882	8.2876		15	.4994	1181.19	105.82
B-8006B*IU*pH3.55*a		7.7918	8.2933		16	.5015	1188.30	106.01
B-8006B*IU*pH3.55*b		7.7346	8.2367		17	.5021	1155.73	102.99
B-8006B*IU*pH3.70*a		7.7285	8.2293		18	.5008	1130.81	101.03
B-8006B*IU*pH3.70*b		7.7996	8.3012	12	1	.5016	1151.42	102.70
B-8006B*IU*pH3.80*a		7.7645	8.2661		2	.5016	1168.72	104.25
B-8006B*IU*pH3.80*b		7.7354	8.2366		3	.5012	1151.80	102.82
B-8006B*IU*pH3.90*a		7.8099	8.3129		4	.5030	1125.31	100.10
B-8006B*IU*pH3.90*b		7.7467	8.2488		5	.5021	1144.04	101.94
B-8006B*IU*pH4.00*a		7.8774	8.3796		6	.5022	1139.19	101.49
B-8006B*IU*pH4.00*b		7.8087	8.3118		7	.5031	1128.43	100.35
B-8006B*IU*pH4.05*a		7.7857	8.2886		8	.5029	1060.52	94.35
B-8006B*IU*pH4.05*b		7.8145	8.3155		9	.5010	1037.30	92.64
B-8006B*IU*pH4.10*a		7.8127	8.3156		10	.5029	1151.28	102.43

B-8006B LSA sample weight

B-8006B*IU*pH4.10*b		7.8015	8.3039		11	.5024	1182.21	105.28
B-8006B*IU*pH4.15*a		7.7662	8.2686		12	.5024	1155.06	102.86
B-8006B*IU*pH4.15*b		7.7532	8.2553		13	.5021	1150.77	102.54
B-8006B*IU*pH4.20*a		7.7441	8.2465		14	.5024	1134.99	101.08
B-8006B*IU*pH4.20*b		7.7571	8.2590		15	.5019	1098.61	97.93
B-8006B*IU*pH4.30*a		7.7587	8.2609		16	.5022	1134.48	101.07
B-8006B*IU*pH4.30*b		7.7180	8.2211		17	.5031	1125.31	100.08
B-8006B*IU*pH4.50*a		7.8110	8.3130		18	.5020	1129.80	100.25
B-8006B*IU*pH4.50*b		7.7849	8.2864	13	1	.5015	1134.22	101.19
B-8006B*IU*pH5.00*a		7.7435	8.2958		2	.5023	1109.13	98.79
B-8006B*IU*pH5.00*b		7.7481	8.2513		3	.5032	1129.45	100.42
B-8006B*IU*pH5.50*a		7.7850	8.2871		4	.5021	1095.15	97.59
B-8006B*IU*pH5.50*b		7.8437	8.3451		5	.5014	1106.11	98.70
B-8006B*IU*pH6.00*a		7.8096	8.3107		6	.5011	1074.36	95.93
B-8006B*IU*pH6.00*b		7.7596	8.2635		7	.5039	1082.98	96.16
B-8006B*IU*pH6.50*a		7.7467	8.2484		8	.5017	1100.10	98.11
B-8006B*IU*pH6.50*b		7.8233	8.3250		9	.5017	1101.59	98.24
B-8006B*IU*pH7.00*a		7.7488	8.2489		10	.5001	1111.42	99.43
B-8006B*IU*pH7.00*b		7.7793	8.2799		11	.5006	1100.60	~~98.37~~ 98.37
B-8006B*IU*pH7.25*a		7.8404	8.3486		12	.5082	1129.96	100.67
B-8006B*IU*pH7.25*b		7.8636	8.3658		13	.5022	1111.42	99.02
B-8006B*IU*pH7.50*a		7.8019	8.3035		14	.5016	1144.04	102.05
B-8006B*IU*pH7.50*b		7.7883	8.2895		15	.5012	1165.38	104.03
B-8006B*IU*pH7.75*a		7.7578	8.2587		16	.5009	1117.03	99.78
B-8006B*IU*pH7.75*b		7.8082	8.3096		17	.5014	1105.83	98.68
B-8006B*IU*pH8.00*a		7.7904	8.2911		18	.5007	1127.01	100.71
B-8006B*IU*pH8.00*b		7.7909	8.2911	14	1	.5002	1142.24	102.17
B-8006B*IU*pH8.25*a		7.7883	8.2691		2	.5008	1146.49	102.43
B-8006B*IU*pH8.25*b		7.8334	8.3306		3	.4972	1146.23	103.15
B-8006B*IU*pH8.62*a		7.7633	8.2615		4	.4982	1139.55	102.34
B-8006B*IU*pH8.62*b		7.7866	8.2861		5	.4995	1127.92	101.03
B-8006B*IU*pH9.00*a		7.7715	8.2713		6	.4998	1155.99	103.48
B-8006B*IU*pH9.00*b		7.7625	8.2623		7	.4998	1131.06	101.25
B-8006B*IU*pH9.00*a		7.7955	8.2960		9	.5005	1115.72	99.73
B-8006B*IU*pH9.00*b		7.8186	8.3183		10	.4997	1169.70	~~101.69~~ 104.73
B-8006B*IU*pH9.00*a		7.9160	8.4189		11	.5029	1143.01	101.69
B-8006B*IU*pH9.00*b		7.8996	8.4024		12	.5028	1146.49	102.02

B-8006B LSA sample weight

	8/10/93 0930	~~7.8620~~ DB 8/10/93						
B-8006B*pH2.00*a	7.8620	~~7.8709~~	8.3659	11	2	.5039	1231.16	109.32
B-8006B*pH2.00*b	7.8709	~~7.8781~~	8.3748		3	.5039	1253.90	111.34
B-8006B*pH2.37*a	7.8781	~~7.8967~~	8.3812		4	.5031	1263.10	112.33
B-8006B*pH2.37*b	7.8967	~~7.9073~~	8.3982		5	.5015	1302.29	116.19
B-8006B*pH2.75*a	7.9073	~~7.8590~~	8.4084		6	.5011	1259.78	112.48
B-8006B*pH2.75*b	7.8590	~~7.8630~~	8.3609		7	.5019	1241.81	110.70
B-8006B*pH3.00*a	7.8630	~~7.8576~~	8.3648		8	.5018	1247.28	111.21
B-8006B*pH3.00*b		7.8576	8.3610		9	.5034	1281.69	113.92
B-8006B*pH3.10*a		7.8787	8.3821		10	.5034	1241.53	110.35
B-8006B*pH3.10*b		7.8718	8.3760		11	.5042	1295.55	114.96
B-8006B*pH3.25*a		7.8098	8.3139		12	.5041	1273.22	113.01
B-8006B*pH3.25*b		7.8764	8.3815		13	.5051	1259.21	111.54
B-8006B*pH3.40*a		7.8856	8.3873		14	.5017	1286.57	114.74
B-8006B*pH3.40*b		7.8551	8.3564		15	.5013	1250.52	111.61
B-8006B*pH3.55*a		7.8846	8.3855		16	.5009	1237.38	110.53
B-8006B*pH3.55*b		7.9080	8.4094		17	.5014	1252.36	111.75
B-8006B*pH3.70*a		7.8641	8.3660		18	.5019	1278.85	114.00
B-8006B*pH3.70*b		7.8446	8.3462	12	1	.5016	1270.07	113.29
B-8006B*pH3.80*a		7.8980	8.4007		2	.5027	1270.93	113.12
B-8006B*pH3.80*b		7.8641	8.3675		3	.5034	1270.93	112.96
B-8006B*pH3.90*a		7.8616	8.3641		4	.5025	1231.16	109.62
B-8006B*pH3.90*b		7.9197	8.4230		5	.5033	1229.96	109.34
B-8006B*pH4.00*a		7.8838	8.3881		6	.5043	1202.13	106.65
B-8006B*pH4.00*b		7.8449	8.3483		7	.5034	1267.86	112.69
B-8006B*pH4.05*a		7.9095	8.4119		8	.5024	913.003	81.31
B-8006B*pH4.05*b		7.8697	8.3718		9	.5021	892.287	79.51
B-8006B*pH4.10*a		7.9134	8.4160		10	.5026	1049.43	93.42
B-8006B*pH4.10*b		7.8510	8.3532		11	.5022	1016.90	90.60
B-8006B*pH4.15*a		7.8638	8.3661		12	.5023	980.785	87.36
B-8006B*pH4.15*b		7.8529	8.3558		13	.5029	1027.64	91.37
B-8006B*pH4.20*a		7.8546	8.3575		14	.5029	933.135	83.02
B-8006B*pH4.20*b		7.8885	8.3906		15	.5021	926.421	82.55

?

B-8006B LSA sample weight

B-8006B*pH4.30*a		7.8653	8.3691		16	.5038	905.916	80.45
B-8006B*pH4.30*b		7.8772	8.3819		17	.5047	904.265	80.16
B-8006B*pH4.50*a		7.9256	8.4282		18	.5026	893.085	79.50
B-8006B*pH4.50*b		7.9176	8.4209	13	1	.5033	924.214	82.16
B-8006B*pH5.00*a		7.8928	8.3946		2	.5018	941.088	83.91
B-8006B*pH5.00*b		~~7.8~~ 7.8900	8.3919		3	.5019	963.236	85.87
B-8006B*pH5.50*a		7.8409	8.3431		4	.5022	951.650	84.78
B-8006B*pH5.50*b	8/10/93	7.8776	8.3797		5	.5021	952.843	84.91
B-8006B*pH6.00*a		7.8650	8.3680		6	.5030	938.663	83.49
B-8006B*pH6.00*b		7.8234	8.3258		7	.5024	924.839	82.36
B-8006B*pH6.50*a		7.8967	8.3974		8	.5007	994.920	88.91
B-8006B*pH6.50*b		7.8631	8.3666		9	.5035	977.950	86.90
B-8006B*pH7.00*a		7.8791	8.3818		10	.5027	1020.62	90.84
B-8006B*pH7.00*b		7.8397	8.3426		11	.5029	1029.66	91.61
B-8006B*pH7.25*a		7.8938	8.3965		12	.5027	1062.66	94.58
B-8006B*pH7.25*b		7.8222	8.3247		13	.5025	1043.07	92.87
B-8006B*pH7.50*a		7.8721	8.3742		14	.5021	1125.39	100.28
B-8006B*pH7.50*b		7.8577	8.3613		15	.5036	1125.65	100.01
B-8006B*pH7.75*a		7.8343	8.3367		16	.5024	1182.05	105.27
B-8006B*pH7.75*b		7.9023	8.4059		17	.5036	1188.14	105.56
B-8006B*pH8.00*a	7.8756	~~7.8873~~ DB 8/10/93	8.3768		18	.5012	1244.20	111.07
B-8006B*pH8.00*b	7.8847	~~7.8307~~	8.3894	14	1	.5047	1255.75	111.32
B-8006B*pH8.25*a		7.8710	8.3740		2	.5030	1293.51	115.06
B-8006B*pH8.25*b		7.8471	8.3495		3	.5024	1274.01	113.46
B-8006B*pH8.62*a		7.8812	8.3831		4	.5019	1312.39	116.99
B-8006B*pH8.62*b		7.8537	8.3549		5	.5012	1311.80	117.10
B-8006B*pH9.00*a		7.8990	8.4015		6	.5025	1277.41	113.74
B-8006B*pH9.00*b		7.8765	8.3794		7	.5029	1262.62	112.33
B-8006B*FU1*a		7.8467	8.3487		9	.5020	1283.42	114.39
B-8006B*FU1*b		7.8911	8.3933		10	.5022	1263.95	112.61
B-8006B*FU2*a		7.8603	8.3611		11	.5008	1300.53	116.19
B-8006B*FU2*b		7.8769	8.3775		12	.5006	1255.19	112.18

7/6/93 gp

URANIUM SORPTION EXPERIMENT B-8007B

Kd vs pH; Equilibrium with atmospheric pCO_2; Initial ΣU=100 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: May 31, 1993

OBJECTIVE: to investigate the importance of solid surface area and pH on uranium sorption on alpha-alumina. Experimental data will also be correlated with uranium aqueous speciation.

EQUIPMENT:
Gyratory shaker
Liquid scintillation analyzer (Packard 1900 TR)
ORION ph/mV/ISE/^{0}C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance
Fisher Marathon 21K Centrifuge and 50 ml centrifuge tube adaptors

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
2	50 ml FEP centrifuge tubes (to contain B-8007B*IU1 and B-8007B*IU2)
30	50 ml FEP centrifuge tubes (to contain experimental mixtures and control solutions)
1	2000 ml FEP bottle (for preparation of 100 ppb U solution)
	weighing paper
	Eppendorf pipets and tips (for adjusting pH and taking samples)
	8007 Alpha Alumina (surface area 0.0686 m^2/g; from NIST)
	reagent grade $NaHCO_3$ (Lot 897186A)
	500 ppb stock solution prepared from 50 ppm ^{233}U commercial spike (spike #23A)
	7 ml LSA vials
2L	0.1 m $NaNO_3$ stock solution (lot 7808 KCCL)
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.02 m HNO_3
1L	stock solution of 0.01 m HNO_3
.1L	stock solution of 3.0 m $NaHCO_3$
.1L	stock solution of 2.0 m $NaHCO_3$
.5L	stock solution of 1.0 m $NaHCO_3$
.5L	stock solution of 0.5 m $NaHCO_3$
.5L	stock solution of 0.1 m $NaHCO_3$
.5L	stock solution of 0.05 m $NaHCO_3$
.5L	stock solution of 0.01 m $NaHCO_3$
.5L	stock solution of 0.005 m $NaHCO_3$
	ultrapure water

PREPARATION:

1. Preclean:
   32 50 ml FEP centrifuge tubes
   1 2L FEP bottle

PROCEDURE:

Note: In transferrring uranium solutions, avoid using glass or polypropylene labware.

Solution B-8007B (1 centrifuge tube for each pH value)

- Initial ΣU = 100 ppb
- Initial pH = 2.0 to 9.0; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 40 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. 8007 alpha alumina to use = 0.100±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

7/6/93 1300hrs SP

a) Into a pre-cleaned 2L FEP bottle, prepare 1280 g of 100 ppb U solution by diluting 256 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 1280 g by carefully taring 0.1 m $NaNO_3$ solution into the plastic bottles on a Mettler 4600 balance.

b) Into each of thirty 50 ml FEP centrifuge tubes labeled B-8007B*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 40 g of the 100 ppb U solution.

Transfer the remaining solution into two 50 ml FEP centrifuge tubes labeled B-8007B*IU1 and B-8007B*IU2. Take two 0.5 ml samples from B-8007B*IU1 and B-8007B*IU2 with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8007B-IU1*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of the sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. This is the initial ^{233}U concentration.

c) For each solution B-8007B*pH*i*, where *i*≤3.85:

Adjust the pH of each solution to the approximate value *i* by adding HNO_3 solution or $NaHCO_3$ solution with an Eppendorf micropipet. The concentration and approximate amount to be added is given in Table B-8007B-1. Swril the solutions by hand. Record the micropipet volume and concentration of solution added. *Do not measure the pH at this time.* Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set to about 120 rpm. Leave the tubes on the shaker for about ten days to allow the solutions to reach equilibrium with atomospheric $CO_2(g)$.

7/22/93 SP 1000hrs

d) Measure and record the pH of each solution B-8007B*pH*i*. *Minimize the amount of time the glass electrode is in contact with the uranium solution. Make sure to rinse the electrode well before transferring into another solution.*

From each solution B-8007B*pH*i*, take two 0.5 ml sample with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8007B*IU-pH*i*a (or b)] and pre-weighed scintillation vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling. The measured concentrations are the initial values to be used in the calculation of sorption data.

7/20/93 1800hrs SP

e) Tare 0.100±0.001 g of 8007 alpha alumina onto weighing paper, and carefully transfer into each of the B-8007B*pH*i* centrifuge tubes. Replace the porous cover and replace on the shaker.

8/10/93 0845 SP

f) After equilibrium is reached (at least 10 days), centrifuge each tube at 15,000 rpm for 30 min to separate the aqueous phase from the solid. Take two 0.5 ml samples from each centrifuge tube B-8007B*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., B-8007B*pH*i*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA within the same day as sampling.

Measure and record the pH and temperature of solutions B-8007B*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

g) After all samples are taken, take two 0.5 ml samples from B-8007B*IU1 and B-8007B*IU2 using an Eppendorf pipet and transfer into prelabled (B-8007B*FU1*a (or b)) and preweighed LSA vials. Reweigh vial after addition of sample, add 0.5 ml 0.02 m HNO_3, and analyze by LSA within the same day as sampling.

h) If the analytical results are good, reversibility and reproducibility tests can be done by changing the pH of the solutions and re-equilibrating them at the new pH values.

Procedure for reversibility and reproducibility experiments will be written later.

Table B-8007B-1. Amount of reagent grade HNO_3 or $NaHCO_3$ to add to 40 ml 0.1 m $NaNO_3$ solution containing 100 ppb U to result in pH values given in first column. The amount of reagent added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atomospheric $CO_2(g)$.

Solution pH	Volume HNO_3 added (ml)	Molarity of HNO_3
2.00	0.40	1.0
2.37	0.16	1.0
2.75	0.07	1.0
3.00	0.35	0.1
3.25	0.16	0.1
3.50	0.06	0.1
3.75	0.01	0.1
Solution pH	**Vol of $NaHCO_3$ added (ml)**	**Molarity of $NaHCO_3$**
4.00	0.40	0.005
4.25	0.38	0.01
4.40	0.08	0.05
4.50	0.11	0.05
4.75	0.115	0.05
5.00	0.12	0.05
5.15	0.124	0.05
5.40	0.127	0.05
5.50	0.13	0.05
5.60	0.133	0.05
5.80	0.136	0.05
6.00	0.14	0.05
6.25	0.145	0.05
6.50	0.15	0.05
6.75	0.17	0.05
7.00	0.19	0.05
7.25	0.23	0.05
7.50	0.31	0.05
7.75	0.23	0.1
8.00	0.36	0.1
8.25	0.12	0.5
8.62	0.13	1.0
9.00	0.35	1.0

Table B-8007B

Sample	Target pH	Wt. 233U soln added (g)	pH date/time		pH date/time	
			7/20/93 1350			
B-8007B*pH2.00	2.00	39.99	1.98	22.9°C	1.95	25.4°C
B-8007B*pH2.37	2.37	40.02	2.38	23.0°C	2.34	25.4°C
B-8007B*pH2.75	2.75	40.02	2.75	23.1°C	2.73	25.5°C
B-8007B*pH3.00	3.00	40.02	3.00	23.1°C	3.00	25.5°C
B-8007B*pH3.25	3.25	40.00	3.25	23.1°C	3.24	25.5°C
B-8007B*pH3.50	3.50	39.98	3.50	23.1°C	3.51	25.5°C
B-8007B*pH3.75	3.75	39.98	3.69	23.2°C	3.75	25.5°C
B-8007B*pH4.00	4.00	40.02	3.96	23.1°C	4.05	25.5°C
B-8007B*pH4.25	4.25	40.00	4.22	23.1°C	4.25	24.7°C
B-8007B*pH4.40	4.40	40.00	4.43	23.1°C	4.50	24.7°C
B-8007B*pH4.50	4.50	39.99	4.68	23.1°C	4.80	24.7°C
B-8007B*pH4.75	4.75	40.02	4.71	23.1°C	4.99	24.7°C
B-8007B*pH5.00	5.00	40.00	4.89	23.7°C	5.30	24.6°C
B-8007B*pH5.15	5.15	40.01	5.23	23.7°C	5.60	24.6°C
B-8007B*pH5.40	5.40	40.02	5.64	23.7°C		
B-8007B*pH5.50	5.50	40.00	5.52	23.7°C	5.76	24.5°C
B-8007B*pH5.60	5.60	40.00	5.96	23.8°C	~~6.07~~	~~24.4°C~~
B-8007B*pH5.80	5.80	40.00	6.02	23.8°C	6.04	24.4°C
B-8007B*pH6.00	6.00	40.01	6.24	23.8°C	6.18	24.3°C
B-8007B*pH6.25	6.25	40.02	6.30	23.9°C	5.92	24.4°C
B-8007B*pH6.50	6.50	40.01	6.41	23.9°C	6.39	24.3°C
B-8007B*pH6.75	6.75	39.99	6.70	23.9°C	6.73	24.3°C
B-8007B*pH7.00	7.00	40.01	6.86	24.0°C	7.02	23.9°C
B-8007B*pH7.25	7.25	39.98	7.10	24.0°C	7.36	23.8°C
B-8007B*pH7.50	7.50	40.02	7.49	24.9°C	7.54	23.7°C
B-8007B*ph7.75	7.75	40.01	7.70	24.8°C	7.69	23.7°C
B-8007B*pH8.00	8.00	39.98	8.00	24.8°C	7.98	23.7°C
B-8007B*ph8.25	8.25	40.00	8.23	24.8°C	8.24	23.6°C
B-8007B*pH8.62	8.62	40.00	8.59	24.8°C	8.63	23.6°C
B-8007B*pH9.00	9.00	39.97	8.94	24.8°C	8.94	23.6°C
B-8007B*IU1		40.01	3.76	23.2°C	3.75	25.3°C
B-8007B*IU2		39.57	3.78	23.2°C	3.76	25.6°C

7/21/93
5.99 25.3°C

B-8007B LSA sample weight

Identification	Date/Time	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U (ppb)
B-8007B*IU1*a	7/16/93 1430	7.7709	8.2712	2	6	.5003	1078.61	96.46
B-8007B*IU1*b	7/16/93 1430	7.8003	8.2996	2	7	.4993	1052.55	94.32
B-8007B*IU2*a	7/16/93 1430	7.7306	8.2307	2	8	.5001	1067.94	95.54
B-8007B*IU2*b	7/16/93 1430	7.8237	8.3241	2	9	.5004	1083.66	96.89
B-8007B*IU*pH2.00*a	7/20/93 1000	7.7970	8.2985	15	1	.5015	1136.88	101.43
B-8007B*IU*pH2.00*b		7.7952	8.2980		2	.5028	1136.37	101.12
B-8007B*IU*pH2.37*a		7.8077	8.3033		3	.4956	1151.68	103.97
B-8007B*IU*pH2.37*b		7.7850	8.2850		4	.5000	1139.81	101.99
B-8007B*IU*pH2.75*a		7.7956	8.2967		5	.5011	1128.18	100.73
B-8007B*IU*pH2.75*b		7.7348	8.2361		6	.5013	1122.38	100.17
B-8007B*IU*pH3.00*a		7.7896	8.2900		7	.5004	1120.87	100.22
B-8007B*IU*pH3.00*b		7.7620	8.2623		8	.5003	1178.31	105.38
B-8007B*IU*pH3.25*a		7.8244	8.3238		9	.4994	1160.75	103.99
B-8007B*IU*pH3.25*b		7.8588	8.3603		10	.5015	1116.27	99.59
B-8007B*IU*pH3.50*a		7.8766	8.3764		11	.4998	1166.88	104.46
B-8007B*IU*pH3.50*b		7.8772	8.3781		12	.5009	1166.35	104.18
B-8007B*IU*pH3.75*a		7.9011	8.4005		13	.4994	1146.75	102.74
B-8007B*IU*pH3.75*b		7.8847	8.3843		14	.4996	1149.73	102.96
B-8007B*IU*pH4.00*a		7.8861	8.3856		15	.4995	1093.70	97.97
B-8007B*IU*pH4.00*b		7.8183	8.3177		16	.4994	1139.70	102.11
B-8007B*IU*pH4.25*a		7.7834	8.2824		17	.4990	1135.24	101.79
B-8007B*IU*pH4.25*b		7.8583	8.3566		18	.4983	1124.73	100.99
B-8007B*IU*pH4.40*a		7.8641	8.3648	18	1	.5007	1139.44	101.82
B-8007B*IU*pH4.40*b		7.8701	8.3696		2	.4995	1131.06	101.31
B-8007B*IU*pH4.50*a		7.9287	8.4271		3	.4984	1135.24	101.91
B-8007B*IU*pH4.50*b		7.8832	8.3821		4	.4989	1145.20	102.70
B-8007B*IU*pH4.75*a		7.8682	8.3643		5	.4961	1120.62	101.06
B-8007B*IU*pH4.75*b		7.8405	8.3385		6	.4980	1162.59	104.45
B-8007B*IU*pH5.00*a		7.8406	8.3410		7	.5004	1141.35	102.05
B-8007B*IU*pH5.00*b		7.9082	8.4088		8	.5006	1153.50	103.10
B-8007B*IU*pH5.15*a		7.8798	8.3797		9	.4999	561.011	50.21

B-8007B LSA sample weight

B-8007B*IU*pH5.15*b		7.8391	8.3401		10	.5010	547.684	48.91
B-8007B*IU*pH5.40*a		7.8522	8.3530		11	.5008	1093.18	97.67
B-8007B*IU*pH5.40*b		7.8536	8.3558		12	.5022	1093.43	97.42
B-8007B*IU*pH5.50*a		7.8560	8.3571		13	.5011	1145.72	102.30
B-8007B*IU*pH5.50*b		7.8694	8.3706		14	.5012	1115.72	99.60
B-8007B*IU*pH5.60*a		7.8969	8.3975		15	.5006	1097.13	98.06
B-8007B*IU*pH5.60*b		7.8293	8.3312		16	.5019	1148.30	102.36
B-8007B*IU*pH5.80*a		7.8866	8.3876		17	.5010	1119.10	99.94
B-8007B*IU*pH5.80*b		7.8941	8.3957		18	.5016	1124.22	100.28
B-8007B*IU*pH6.00*a		7.9072	8.4076	19	1	.5004	1119.55	100.10
B-8007B*IU*pH6.00*b		7.8729	8.3729		2	.5000	1115.47	99.82
B-8007B*IU*pH6.25*a		7.8628	8.3636		3	.5008	1084.65	96.90
B-8007B*IU*pH6.25*b		7.8428	8.3448		4	.5020	1114.22	99.31
B-8007B*IU*pH6.50*a		7.9243	8.4240		5	.4997	1125.05	100.73
B-8007B*IU*pH6.50*b		7.8963	8.3976		6	.5013	1116.27	99.63
B-8007B*IU*pH6.75*a		7.8699	8.3712		7	.5013	1102.35	98.39
B-8007B*IU*pH6.57*b		7.8550	8.3564		8	.5014	1130.81	100.91
B-8007B*IU*pH7.00*a		7.8689	8.3703		9	.5014	1101.10	98.25
B-8007B*IU*pH7.00*b		7.8457	8.3473		10	.5016	1160.75	103.54
B-8007B*IU*pH7.25*a		7.8757	8.3784		11	.5027	1114.17	99.16
B-8007B*IU*pH7.25*b		7.8965	8.3990		12	.5025	1124.80	100.15
B-8007B*IU*pH7.50*a		7.8554	8.3569		13	.5015	1149.21	102.53
B-8007B*IU*pH7.50*b		7.9039	8.4070		14	.5031	1122.45	99.82
B-8007B*IU*pH7.75*a		7.8977	8.4002		15	.5025	1092.20	97.25
B-8007B*IU*pH7.75*b		7.8829	8.3858		16	.5029	1127.34	100.30
B-8007B*IU*pH8.00*a		7.8246	8.3260		17	.5014	1148.43	102.48
B-8007B*IU*pH8.00*b		7.8683	8.3721		18	.5038	1130.98	100.44
B-8007B*IU*pH8.25*a		7.9092	8.4113	20	1	.5021	1155.47	102.96
B-8007B*IU*pH8.25*b		7.8682	8.3687		2	.5005	1179.33	105.42
B-8007B*IU*pH8.62*a		7.8632	8.3638		3	.5006	1139.30	101.83
B-8007B*IU*pH8.62*b		7.8845	8.3854		4	.5009	1140.32	101.86
B-8007B*IU*pH9.00*a		7.8666	8.3683		5	.5017	1140.07	101.67
B-8007B*IU*pH9.00*b		7.8527	8.3542		6	.5015	1133.71	101.15

B-8007B*MU1*a		7.8510	8.3528		8	.5018	1144.56	102.05
B-8007B*MU1*b		7.8388	8.3414		9	.5026	1126.32	100.27
B-8007B*MU2*a		7.8093	8.3106		10	.5013	1134.48	101.25
B-8007B*MU2*b		7.8203	8.3217		11	.5014	1142.50	101.95

B-8007B LSA sample weight

B-8007B*pH2.00*a	8/10/93 1030	7.9165	8.4178	3	1	.5013	1286.28	114.80
B-8007B*pH2.00*b		7.8268	8.3278		2	.5010	1303.17	116.38
B-8007B*pH2.37*a		7.8575	8.3593		3	.5018	1320.23	117.72
B-8007B*pH2.37*b		7.8307	8.3321		4	.5014	1299.35	115.95
B-8007B*pH2.75*a		7.9075	8.4089		5	.5014	1342.64	119.81
B-8007B*pH2.75*b		7.8298	8.3309		6	.5011	1316.00	117.50
B-8007B*pH3.00*a		7.8073	8.3101		7	.5028	1292.92	115.05
B-8007B*pH3.00*b		7.9056	8.4058		8	.5002	1263.95	113.06
B-8007B*pH3.25*a		7.8419	8.3430		9	.5011	1298.18	115.91
B-8007B*pH3.25*b		7.8743	8.3758		10	.5015	1305.83	116.50
B-8007B*pH3.50*a		7.8321	8.3334		11	.5019	1299.05	115.80
B-8007B*pH3.50*b		7.8293	8.3315		12	.5022 8/9	1284.83	114.47
B-8007B*pH3.75*a		7.8883	8.3910		13	.5080,5027	1282.26	114.13
B-8007B*pH3.75*b		7.8789	8.3811		14	.5022 8/9	1289.16	114.85
B-8007B*pH4.00*a		7.8917	8.3939		15	.5022	1260.92	112.34
B-8007B*pH4.00*b		7.8815	8.3835		16	.5020	1245.03	110.97
B-8007B*pH4.25*a		7.9423	8.4454		17	.5031	1260.35	112.09
B-8007B*pH4.25*b		7.8704	8.3712		18	.5008	1297.88	115.95
B-8007B*pH4.40*a		7.8732	8.3736	8	1	.5004	1264.81	113.09
B-8007B*pH4.40*b		7.8719	8.3747		2	.5028	1320.52	117.51
B-8007B*pH4.50*a		7.8658	8.3664		3	.5006	1232.55	110.16
B-8007B*pH4.50*b		7.9089	8.4062		4	.4973	1227.74	110.46
B-8007B*pH4.75*a		7.8523	8.3539		5	.5016	1242.93	110.87
B-8007B*pH4.75*b		7.8579	8.3581		6	.5002	1255.92	112.34
B-8007B*pH5.00*a		7.8493	8.3520		7	.5027	1187.09	105.65
B-8007B*pH5.00*b		7.9063	8.4085		8	.5022	1188.14	105.85
B-8007B*pH5.15*a		7.8573	8.3573		9	.5000	519.890	46.52
B-8007B*pH5.15*b		7.8483	8.3514		10	.5031	542.660	48.26
B-8007B*pH5.40*a		7.8502	8.3514		11	.5012	1152.04	102.84
B-8007B*pH5.40*b		7.8781	8.3795		12	.5014	1101.68	98.31
B-8007B*pH5.50*a		7.8811	8.3826		13	.5015	1129.03	100.73
B-8007B*pH5.50*b		7.9088	8.4114		14	.5026	1167.24	103.91

B-8007B LSA sample weight

B-8007B*pH5.60*a		7.8587	8.3600		15	.5013	1119.44	99.91
B-8007B*pH5.60*b		7.8686	8.3702		16	.5016	1111.00	99.10
B-8007B*pH5.80*a		7.8614	8.3620		17	.5006	1145.81	102.41
B-8007B*pH5.80*b		7.9012	8.4014		18	.5002	1129.54	101.04
B-8007B*pH6.00*a		7.8833	8.3852	9	1	.5019	1138.00	101.45
B-8007B*pH6.00*b		7.8796	8.3811		2	.5015	1116.06	99.57
B-8007B*pH6.25*a		7.8877	8.3895		3	.5018	1062.11	94.70
B-8007B*pH6.25*b		7.8643	8.3681		4	.5038	1102.14	97.88
B-8007B*pH6.50*a		7.8875	8.3906		5	.5031	1093.04	97.21
B-8007B*pH6.50*b		7.8634	8.3649		6	.5015	1071.60	95.60
B-8007B*pH6.75*a		7.8675	8.3683		7	.5008	1096.73	97.98
B-8007B*pH6.57*b		7.8875	8.3889		8	.5014	1068.01	95.30
B-8007B*pH7.00*a		7.9367	8.4334		9	.4967	1130.87	101.87
B-8007B*pH7.00*b		7.8776	8.3759		10	.4983	1101.49	98.90
B-8007B*pH7.25*a		7.8812	8.3830		11	.5018	1141.81	101.81
B-8007B*pH7.25*b		7.8676	8.3674		12	.4998	1134.20	101.53
B-8007B*pH7.50*a		7.8812	8.3831		13	.5019	1148.44	102.38
B-8007B*pH7.50*b		7.8913	8.3933		14	.5020	1170.07	104.29
B-8007B*pH7.75*a		7.8529	8.3594		15	.5065	1226.37	108.33
B-8007B*pH7.75*b		7.9056	8.4067		16	.5011	1212.42	108.25
B-8007B*pH8.00*a		7.8828	8.3833		17	.5005	1297.36	115.98
B-8007B*pH8.00*b		7.8728	8.3742		18	.5014	1252.47	111.76
B-8007B*pH8.25*a		7.8935	8.3951	15	1	.5016	1305.04	116.41
B-8007B*pH8.25*b		7.8529	8.3556		2	.5027	1308.47	116.46
B-8007B*pH8.62*a		7.9135	8.4144		3	.5009	1294.31	115.61
B-8007B*pH8.62*b		7.8382	8.3393		4	.5011	1292.92	115.44
B-8007B*pH9.00*a		7.8677	8.3687		5	.5010	1321.56	118.02
B-8007B*pH9.00*b		7.8977	8.3990		6	.5013	1311.71	117.07
B-8007B*FU1*a		7.8418	8.3440		8	.5022	1274.26	113.53
B-8007B*FU1*b		7.8752	8.3759		9	.5007	1251.96	111.87
B-8007B*FU2*a		7.8500	8.3510		10	.5010	1290.19	115.22
B-8007B*FU2*b		7.8611	8.3629		11	.5018	1292.28	115.22

7/8/93 JP

^{233}U waste Disposal -
LSA samples for experiments B-8006 and B-8007 were disposed of following the procedure outlined below.

MEMORANDUM

To: Todd D., Paul B., Jim P., Paula M. and Roberto P.

From: Bret W. Leslie, Radiation Safety POC
BWL

Date: June 15, 1993

Subject: Liquid Scintillation Cocktail ^{233}U Waste Disposal

After some extended deliberations by both the Safety Office and Radiation Safety Office, they have approved the disposal procedure for ^{233}U/scintillation cocktail solution via the sanitary sewer. From Todd's experiments the maximum activity of ^{233}U in the cocktail would be 2×10^{-10} Ci/ml (*i.e.* 200 pCi/ml cocktail solution). Note that soluble daily release limit for ^{233}U is the larger of (1) the quantity which, if diluted by the average quantity of sewage released to the sewer by the license, will result in average concentration not greater than the limits specified in Appendix 21-A, Table I, Column 2 (**900 pCi/ml**) or (2) ten times the quantity of such material specified in Appendix 21-B (**10 nCi, ie no more than 100 nCi**). It has been required by Frank Iddings (RSO) that we limit daily disposal to less than 100 nCi of ^{233}U.

Note that disposal procedure will entail dilution of sample (total volume 6 ml) by a factor of 25 prior to disposal in running water. Perhaps the easiest way to accomplish this is to empty the contents of 6 vials at time and dilute to 1000 ml in plastic beaker and then pour down the sink with liberal simultaneous flushing of water. The sink should not have any other glassware in it during disposal operations to minimize contamination and cleaning. The vials will be rinsed thoroughly and checked for residual activity. This does not mean that all sample vials have to be loaded with cocktail solution and counted again, however, you should test a couple of your highest activity vials for residual contamination, prior to disposing of your vials in the trash. The vials, once clean, can be disposed in the regular trash provided lids are not attached. Samples should be disposed of on a regular basis or when an experiment is over (check with BL or RP).

I have also attached a sample sanitary sewer disposal log sheet (compliments of Todd) for the ^{233}U disposal. Except for U wasted bret, all information on the sheet should be on any sheet you use (design) to record disposal. Note that the activity remaining column (in units of nCi or Ci) can be totaled (*i.e.* only one entry) for each disposal operation. The activity remaining should reflect the activity and spike # and isotope that was disposed. The completed disposal sheet for each disposal operation should be placed in the radiation control log binder under the waste disposal section, and a copy of it can be used to replace the existing page in the sample binder in the counting room.

7/9/93 JP

^{233}U waste disposal -
samples for the filtration loss experiment (p 53), the tip loss experiment (p 57), and the SP experiment (p 61) were disposed of following the procedure outlined on p 121.

5 LSA vials were selected at random to determine residual activity before disposal of the vials.

The vials were labeled as below. 0.5 ml of 0.02 m HNO_3 and 0.5 ml of 0.1 m $NaNO_3$ and 5 ml of scintillation cocktail were added to each vial and analyzed by LSA.

Results are shown below.

Identification	Vial wt g	S+v wt g	RACK	VIAL	Sam wt	CPMB	U (ppb)
WLSA1	7.9247	8.4265	7	14	0.5018	0.0	0.0
WLSA2	7.8914	8.3951	7	15	0.5037	0.0	0.0
WLSA3	7.9586	8.4611	7	16	0.5025	0.033	0.0029
WLSA4	8.0171	8.5188	7	17	0.5017	0.933	0.0832
WLSA5	7.9447	8.4479	7	18	0.5032	0.633	0.0563

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7/19/93 JP

URANIUM SORPTION EXPERIMENT CL-100
Kd vs pH; Equilibrium with atmospheric pCO_2; Initial ΣU=100 ppb

WRITTEN BY: R.T. PABALAN
REVISION NO.: 2
REVISED BY: J.D. PRIKRYL

DATE WRITTEN: Aug 5, 1992
DATE REVISED: March 10, 1993

OBJECTIVE: to investigate the importance of evaporation and uranium loss to walls of 50 ml FEP centrifuge tubes used in sorption experiments as a function of solution pH

EQUIPMENT:
Gyratory shaker
Liquid Scintillation Analyzer
ORION ph/mV/ISE/ C meter
Combination ph electrode
Automatic termperature compensator probe
Analytical balance

SUPPLIES:

	pH buffers (pH= 2,4,7,9,10)
2	50 ml FEP centrifuge tubes (to contain CL-100*IU1 and IU2)
20	50 ml FEP centrifuge tubes (to contain experimental solutions)
1	1L FEP bottle (for preparation of 100 ppb U solution
	weighing paper
	Eppendorf pipets and tips (for adjusting pH and taking samples)
	500 ppb stock solution prepared from 5 ppm ^{233}U commercial spike (spike #23A)
	7 ml LSA vials
1L	0.1 m $NaNO_3$ stock solution (lot 7808 KCCL)
1L	stock solution of 1.0 m HNO_3
1L	stock solution of 0.1 m HNO_3
1L	stock solution of 0.01 m HNO_3
1L	stock solution of 0.02 m HNO_3
.5L	stock solution of 1.0 m $NaHCO_3$
.5L	stock solution of 0.5 m $NaHCO_3$
.5L	stock solution of 0.1 m $NaHCO_3$
.5L	stock solution of 0.05 m $NaHCO_3$
.5L	stock solution of 0.01 m $NaHCO_3$
.5L	stock solution of 0.005 m $NaHCO_3$
	ultrapure water

PREPARATION:

1. Preclean:
   22 50 ml FEP centrifuge tubes
   1 1L FEP bottle

PROCEDURE:

Solution CL-100 (1 tube for each pH value)

- Initial ΣU = 100 ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit from 5-8, every 0.5 pH unit from 2-5 and 8-9; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 40 ml
- Ionic strength = 0.1 m $NaNO_3$
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- pCO_2 = atmospheric = $10^{-3.48}$ bar

a) In a pre-cleaned 1L FEP bottle, prepare 900 g of 100 ppb U solution by diluting 180 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 5 ppm ^{233}U spike) to a total of 900 g by carefully taring 0.1 m $NaNO_3$ solution into the FEP bottle on a Mettler 4600 balance.

b) Into each of 20 50 ml FEP centrifuge tubes labeled CL-100*pH*i* [where *i* is the appropriate initial pH of the solution (see below)], tare 40 g of the 100 ppb U solution.

Into 2 50 ml FEP centrifuge tubes labelled CL-100*IU1 and CL-100*IU2, tare 41 g of the 100 ppb U solution. Take two 0.5 ml samples from CL-100*IU1 and CL-100*IU2 with an Eppendorf pipet, transfer into pre-labeled [i.e., CL-100*IU1*a (orb)] and pre-weighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA. This is the initial ^{233}U concentration. Record the total weight of each sample.

c) 1. For each solution CL-100*pH*i*, where *i*≤3.85:

Adjust the pH of each solution to the approximate value *i* by adding HNO_3 solution or $NaHCO_3$ solution with an Eppendorf micropipet. The concentration and approximate amount to be added is given in Table CL-100. Swril the solutions by hand. Record the micropipet volume and concentration of solution added. *Do not measure the pH at this time.*

Record the total weight of the samples. Cover the bottles with a porous material (e.g., kimwipe) and place on gyratory shaker set at about 120 rpm. Leave the tubes on the shaker for about 2 weeks to allow solutions to reach equilibrium with atomospheric CO_2 (g).

d) Record the total weight of each sample CL*100*pH*i* and CL-100*IU1 and CL-100*IU2. Measure and record the pH of each sample solution. *Minimize the amount of time the glass electrode is in contact with the uranium solution. Make sure to rinse the electrode well before transferring into another solution.*

From each solution take two 0.5 ml samples with an Eppendorf pipet, transfer into pre-labeled [i.e., CL-100*IU-pH/*a (or b) or CL-100*MI*a (or b)] and pre-weighed scintillation vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA. The measured concentrations are the initial values to be used in the calculation of sorption data.

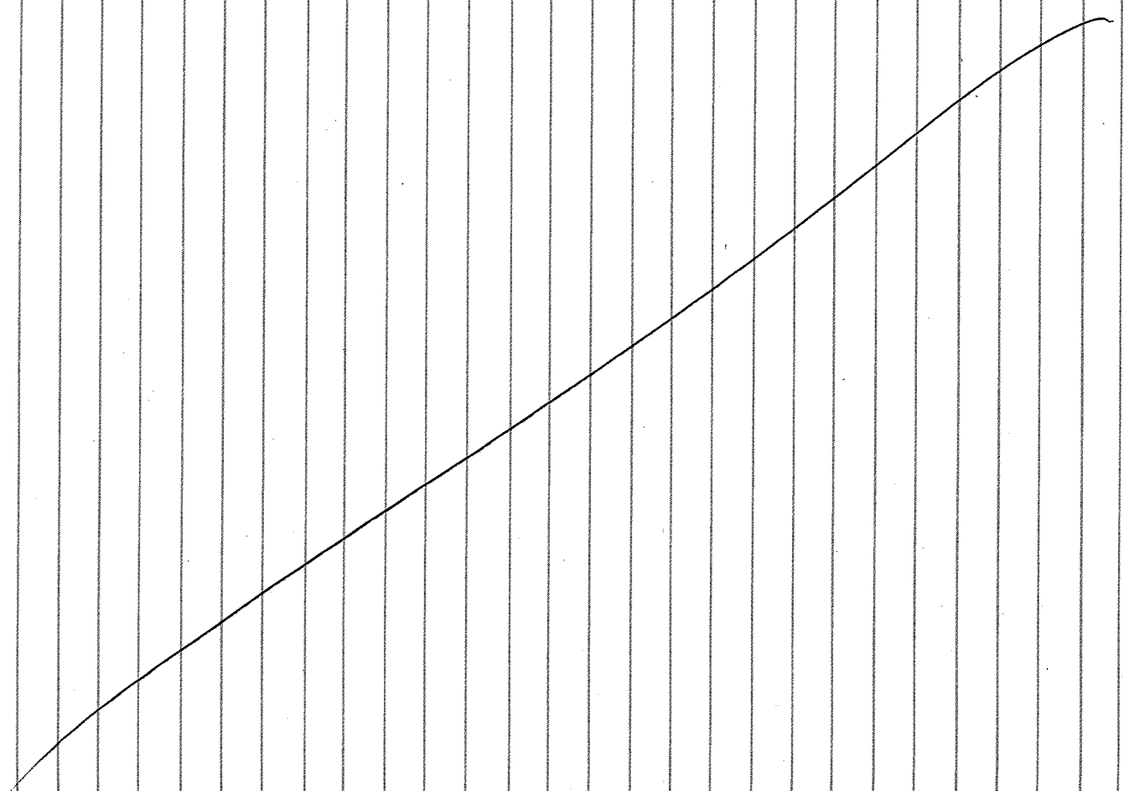
Record the total weight of each sample.

e) JP 8/23/93 0830hrs After three weeks again record the total weight of each sample to determine evaporation loss. Take two 0.5 ml samples from each centrifuge tube with an Eppendorf pipet, transfer into pre-labedled [e.g., CL-100*pH/*a (or b) or CL-100*FI*a (or b)] and pre-weighed LSA vials. Reweigh vials after addition of sample, add 0.5 ml 0.02 m HNO_3 to each sample, and analyze by LSA.

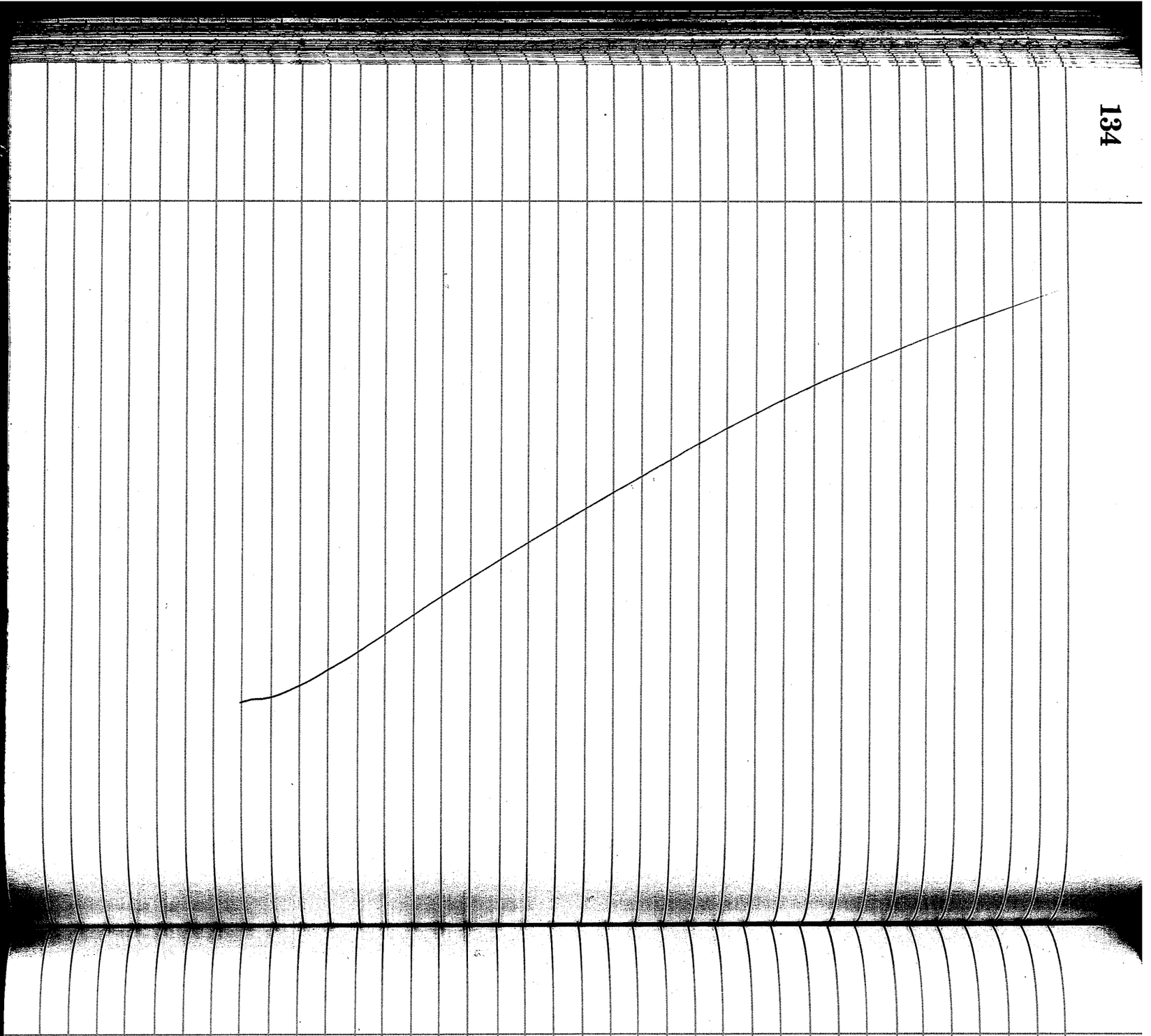
Measure and record the pH and temperature of each solution.

Table CL-100. Amount of reagent grade HNO_3 or $NaHCO_3$ to add to 40 ml 0.1 m $NaNO_3$ solution containing 100 ppb U to result in pH values given in first column. The amount of reagent added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atomospheric $CO_2(g)$.

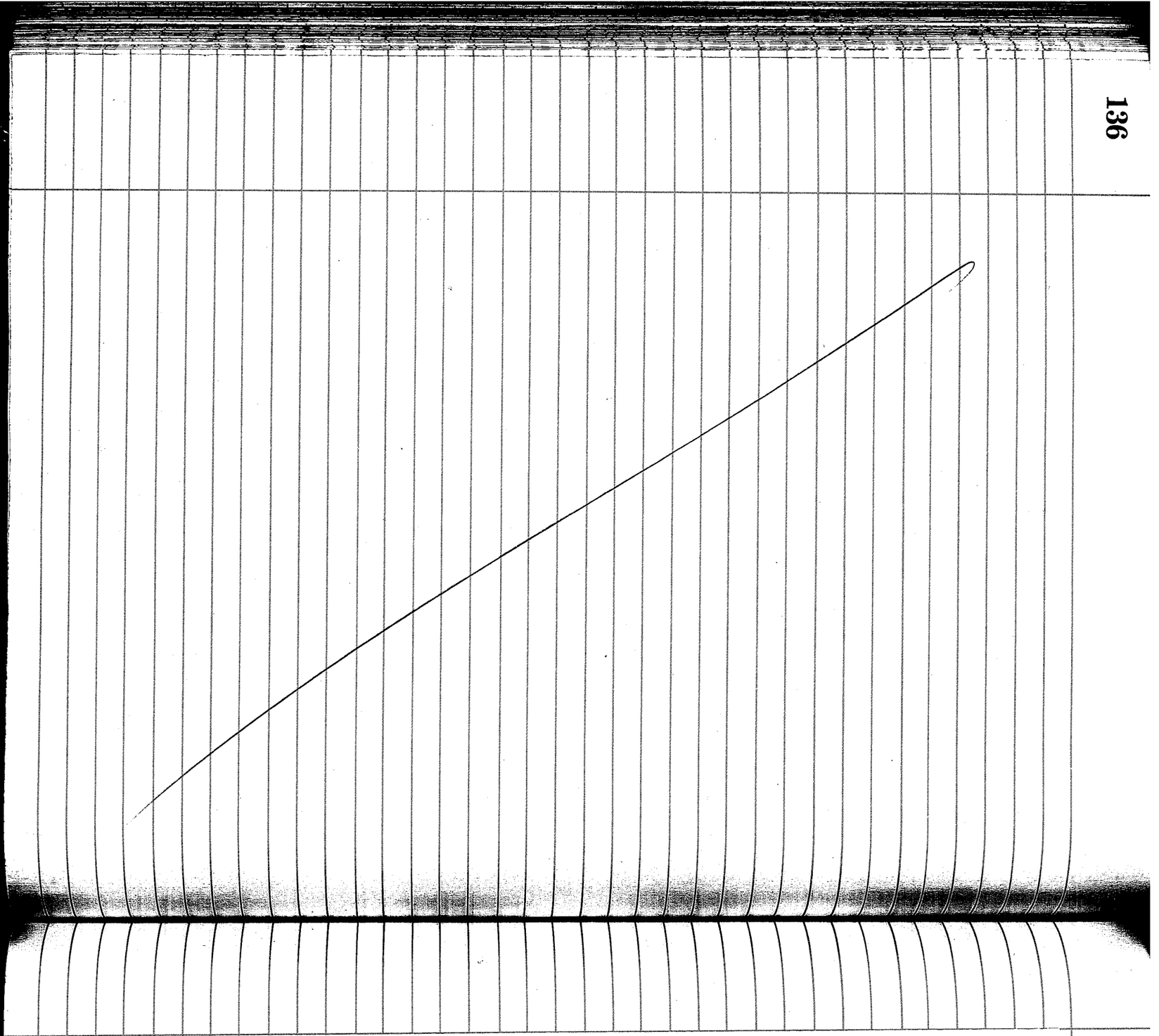
Solution pH	Volume HNO_3 added (ml)	Molarity of HNO_3
2.00	0.40	1.0
3.00	0.35	0.1
3.50	0.06	0.1
Solution pH	**Vol of $NaHCO_3$ added (ml)**	**Molarity of $NaHCO_3$**
4.00	0.40	0.005
4.50	0.11	0.05
5.00	0.12	0.05
5.25	0.124	0.05
5.50	0.127	0.05
5.75	0.13	0.05
6.00	0.135	0.05
6.25	0.14	0.05
6.50	0.15	0.05
6.75	0.17	0.05
7.00	0.19	0.05
7.25	0.23	0.05
7.50	0.31	0.05
7.75	0.23	0.1
8.00	0.36	0.1
8.50	0.2	0.5
9.00	0.35	1.0



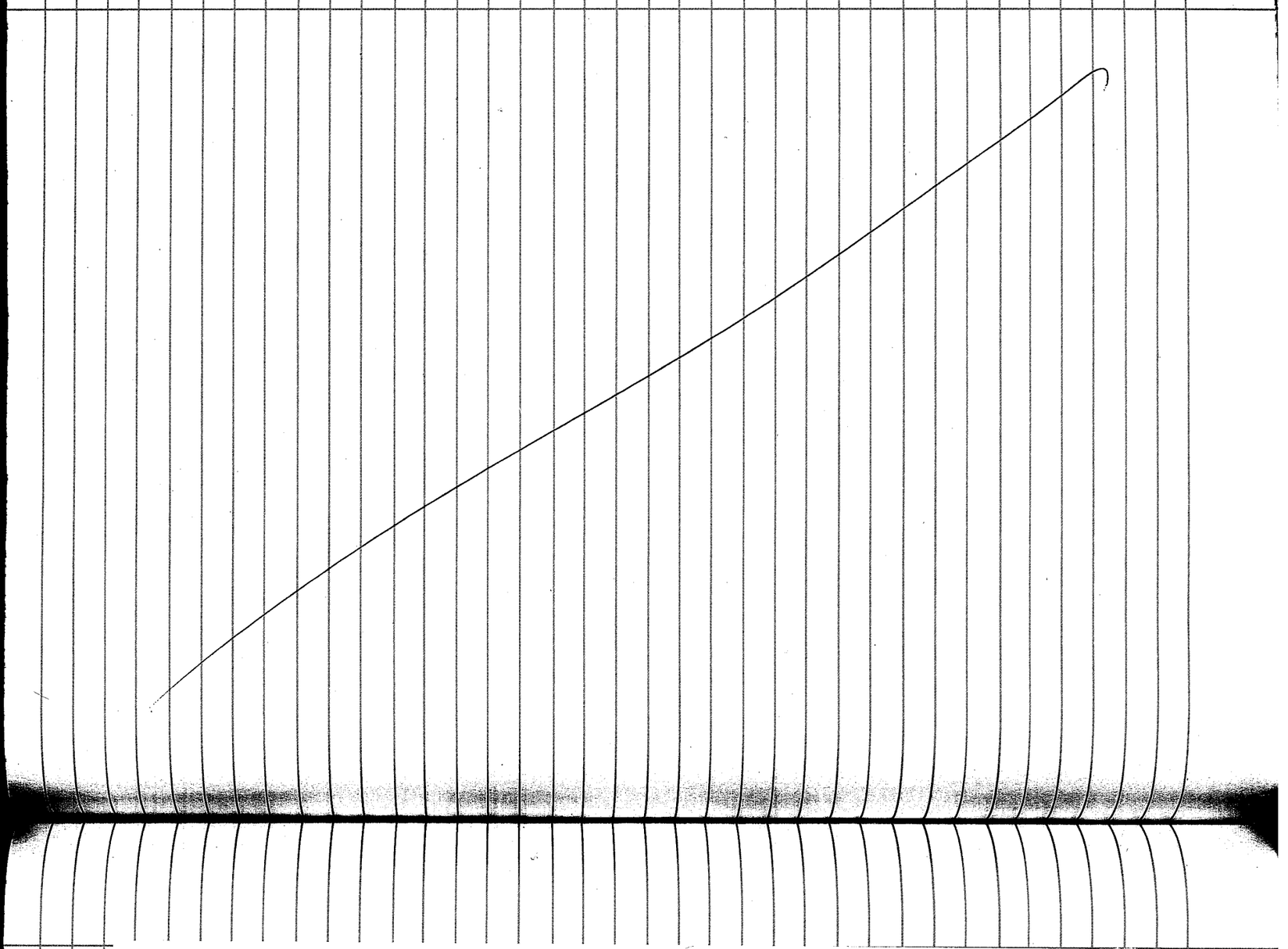
Sample	Wt. 233U soln (g)	Weight (g)	Weight (g)	pH	Weight (g)	Weight (g)	pH
		7/19/93 1400	8/2/93 0915	8/2/93	8/2/93 1600	8/23/93 0850hr	8/23/93 1400hr
CL-100*IU1	41.02	70.03	~~69.01~~ 67.75	3.82 24.8℃	~~65.81~~ 66.66	~~64.14~~ 63.57	3.79 23.4℃
CL-100*IU2	41.02	70.59	~~69.5~~ 68.32	3.82 24.8℃	67.22	64.14 8/2/93	3.80 23.4℃
			8/2/93				
CL-100*pH2.00	40.03	69.44	~~68.25~~ 66.99	1.92 24.8℃	65.81	62.54	1.99 23.3℃
CL-100*pH3.00	40.04	70.06	~~68.83~~ 67.62	2.95 24.8℃	66.47	63.15	2.95 23.3℃
CL-100*pH3.50	40.03	70.21	~~68.95~~ 67.69	3.48 24.8℃	66.54	63.18	3.46 23.3℃
CL-100*pH4.00	40.02	71.11	~~69.85~~ 68.54	3.94 24.8℃	67.45	64.03	3.91 23.3℃
CL-100*pH4.50	40.01	71.14	~~69.86~~ 68.63	4.59 24.8℃	67.48	64.07	4.56 23.3℃
CL-100*pH5.00	40.03	71.21	~~69.93~~ 68.68	4.93 24.9℃	67.55	64.22	4.90 23.4℃
CL-100*pH5.25	40.01	71.76	~~70.44~~ 69.20	5.24 24.9℃	68.08	64.85	5.18 23.4℃
CL-100*pH5.50	40.03	69.82	~~68.87~~ 67.57	6.03 24.8℃	66.46	63.23	5.93 23.4℃
CL-100*pH5.75	40.02	69.52	67.09 8/2/93	5.69 24.8℃	66.01	62.77	5.38 23.4℃
CL-100*pH6.00	40.02	71.59	68.89	6.02 24.8℃	67.79	64.40	5.98 23.4℃
CL-100*pH6.25	40.00	70.33	67.79	6.24 24.8℃	66.69	63.37	6.12 23.5℃
CL-100*pH6.50	40.03	70.34	67.90	6.47 24.9℃	66.80	63.45	6.46 23.5℃
CL-100*pH6.75	40.03	69.65	67.29	6.72 24.9℃	66.18	62.87	6.68 23.5℃
CL-100*pH7.00	40.02	69.65	67.29	6.98 25.0℃	66.17	62.95	6.83 23.5℃
CL-100*pH7.25	40.01	68.93	66.64	7.20 24.9℃	65.53	62.37	7.13 23.7℃
CL-100*pH7.50	40.04	70.80	68.44	7.38 24.9℃	67.33	64.12	7.42 23.6℃
CL-100*ph7.75	40.02	71.31	68.80	7.61 24.9℃	67.71	64.42	7.70 23.6℃
CL-100*pH8.00	40.01	71.54	68.88	7.92 24.9℃	67.78	64.34	7.95 23.6℃
CL-100*ph8.50	40.00	71.27	68.72	8.34 24.9℃	67.58	64.20	8.48 23.6℃
CL-100*pH9.00	40.00	70.46	68.02	8.82 24.9℃	66.92	63.57	8.94 23.6℃



Identification	Date/Time	Vial wt g	Sample+ vial wt g	RACK	VIAL	SAMPLE WT	CPMB	U (ppb)
CL-100*IU*a	7/19/93 1320	7.8427	8.3430	20	15	.5003	1062.43	95.01
CL-100*IU*b	1321	7.8473	8.3463	20	16	.4990	1057.74	94.84
CL-100*IU2*a	1322	7.8899	8.3894	20	17	.4995	1050.43	94.09
CL-100*IU2*b	1323	7.8335	8.3337	20	18	.5002	1041.95	93.20
CL-100*MU1*a	8/2/93 1030	7.8959	8.3972	13	7	.5013	1107.91	98.88
CL-100*MU1*b		7.8319	8.3319	13	8	.5000	1094.96	97.98
CL-100*MU2*a		7.8727	8.3739	13	9	.5012	1112.22	99.29
CL-100*MU2*b		7.8499	8.3500	13	10	.5001	1133.51	101.41
CL-100*IU*pH2.00*a		7.9188	8.4214	11	2	.5026	1157.36	103.03
CL-100*IU*pH2.00*b		7.8933	8.3972		3	.5039	1117.09	99.19
CL-100*IU*pH3.00*a		7.8258	8.3289		4	.5031	1156.06	102.81
CL-100*IU*pH3.00*b		7.8622	8.3640		5	.5018	1129.51	100.71
CL-100*IU*pH3.50*a		7.7993	8.3021		6	.5028	1146.81	102.05
CL-100*IU*pH3.50*b		7.9027	8.4063		7	.5036	1141.93	101.45
CL-100*IU*pH4.00*a		7.8669	8.3698		8	.5029	1102.41	98.08
CL-100*IU*pH4.00*b		7.8338	8.3368		9	.5030	1113.27	99.02
CL-100*IU*pH4.50*a		7.8778	8.3804		10	.5026	1080.59	96.19
CL-100*IU*pH4.50*b		7.8337	8.3364		11	.5027	1101.01	97.99
CL-100*IU*pH5.00*a		7.8729	8.3755		12	.5026	1113.52	99.13
CL-100*IU*pH5.00*b		7.8306	8.3334		13	.5028	1109.93	98.77
CL-100*IU*pH5.25*a		7.9110	8.4142		14	.5032	1086.88	96.64
CL-100*IU*pH5.25*b		7.9311	8.4330		15	.5019	1113.52	99.26
CL-100*IU*pH5.50*a		7.8704	8.3732		16	.5028	1083.97	96.46
CL-100*IU*pH5.50*b		7.8535	8.3554		17	.5019	1051.42	93.73
CL-100*IU*pH5.75*a		7.8172	8.3194		18	.5022	1052.98	93.81
CL-100*IU*pH5.75*b		7.8695	8.3716	12	1	.5021	1072.71	95.59
CL-100*IU*pH6.00*a		7.8852	8.3874		2	.5022	1073.92	95.68
CL-100*IU*pH6.00*b		7.8869	8.3897		3	.5028	1053.45	93.74
CL-100*IU*pH6.25*a		7.8802	8.3829		4	.5027	1057.78	94.15



CL-100*IU*pH6.25*b		7.8583	8.3605		5	.5022	1062.23	94.64
CL-100*IU*pH6.50*a		7.8383	8.3405		6	.5022	1081.07	96.31
CL-100*IU*pH6.50*b		7.8696	8.3716		7	.5020	1073.20	95.65
CL-100*IU*pH6.75*a		7.8534	8.3555		8	.5021	1059.69	94.43
CL-100*IU*pH6.75*b		7.8248	8.3256		9	.5008	1018.52	91.00
CL-100*IU*pH7.00*a		7.8455	8.3480		10	.5025	1088.84	96.95
CL-100*IU*pH7.00*b		7.9151	8.4170		11	.5019	1071.08	95.48
CL-100*IU*pH7.25*a		7.8129	8.3150		12	.5021	1078.92	96.14
CL-100*IU*pH7.25*b		7.8766	8.3775		13	.5009	1064.55	95.09
CL-100*IU*pH7.50*a		7.8521	8.3532		14	.5011	1093.73	97.66
CL-100*IU*pH7.50*b		7.9026	8.4048		15	.5022	1132.40	100.89
CL-100*IU*pH7.75*a		7.8813	8.3800		16	.4987	1119.16	100.41
CL-100*IU*pH7.75*b		7.8352	8.3347		17	.4995	1137.20	101.86
CL-100*IU*pH8.00*a		7.9259	8.4246		18	.4987	1127.47	101.15
CL-100*IU*pH8.00*b		7.8571	8.3558	13	1	.4987	1127.40	101.15
CL-100*IU*pH8.50*a		7.9194	8.4187		2	.4993	1133.77	101.60
CL-100*IU*pH8.50*b		7.8769	8.3773		3	.5004	1125.29	100.61
CL-100*IU*pH9.00*a		7.8871	8.3856		4	.4985	1099.91	98.72
CL-100*IU*pH9.00*b		7.8801	8.3799		5	.4998	1110.93	99.45
CL-100*pH2.00*a	8/23/93 0900	7.8625	8.3658	12	2	.5033	1249.88	111.11
CL-100*pH2.00*b		7.8495	8.3519		3	.5024	1224.97	109.09
CL-100*pH3.00*a		7.8168	8.3195		4	.5027	1234.95	109.91
CL-100*pH3.00*b		7.8008	8.3036		5	.5028	1211.00	107.76
CL-100*pH3.50*a		7.8118	8.3131		6	.5013	1249.39	111.15
CL-100*pH3.50*b		7.8725	8.3740		7	.5015	1282.63	114.43
CL-100*pH4.00*a		7.7951	8.2971		8	.5020	1212.64	108.08
CL-100*pH4.00*b		7.8670	8.3703		9	.5033	1244.41	110.62
CL-100*pH4.50*a		7.8715	8.3750		10	.5035	1250.88	111.16
CL-100*pH4.50*b		7.8317	8.3338		11	.5021	1244.41	110.62
CL-100*pH5.00*a		7.8421	8.3438		12	.5017	1184.52	105.67
CL-100*pH5.00*b		7.7552	8.2560		13	.5008	1185.56	105.92
CL-100*pH5.25*a		7.8126	8.3148		14	.5022	1167.58	104.02



CL-100*pH5.25*b		7.8460	8.3485		15	.5025	1189.02	105.87
CL-100*pH5.50*a		7.7754	8.2767		16	.5013	1125.73	100.47
CL-100*pH5.50*b		7.8467	8.3482		17	.5015	1167.05	104.12
CL-100*pH5.75*a		7.8623	8.3637		18	.5014	1191.95	106.36
CL-100*pH5.75*b		7.7970	8.2997	13	1	.5027	1159.45	103.19
CL-100*pH6.00*a		7.8715	8.3738		2	.5023	1156.17	102.98
CL-100*pH6.00*b		7.7952	8.2980		3	.5028	1130.97	100.64
CL-100*pH6.25*a		7.8631	8.3655		4	.5024	1163.72	103.64
CL-100*pH6.25*b		7.8418	8.3437		5	.5019	1166.27	103.97
CL-100*pH6.50*a		7.8392	8.3412		6	.5020	1173.78	104.62
CL-100*pH6.50*b		7.7913	8.2935		7	.5022	1170.93	104.32
CL-100*pH6.75*a		7.8390	8.3421		8	.5031	1172.72	104.29
CL-100*pH6.75*b		7.8244	8.3268		9	.5024	1148.60	102.29
CL-100*pH7.00*a		7.7961	8.2984		10	.5023	1180.57	105.16
CL-100*pH7.00*b		7.8453	8.3476		11	.5023	1170.48	104.26
CL-100*pH7.25*a		7.7909	8.2897		12	.4988	1182.39	106.06
CL-100*pH7.25*b		7.7932	8.2928		13	.4996	1179.77	105.65
CL-100*pH7.50*a		7.9251	8.4250		14	.4999	1214.55	108.70
CL-100*pH7.50*b		7.8122	8.3125		15	.5003	1228.65	109.88
CL-100*pH7.75*a		7.8848	8.3858		16	.5010	1215.97	108.59
CL-100*pH7.75*b		7.8022	8.3029		17	.5007	1237.46	110.58
CL-100*pH8.00*a		7.8408	8.3421		18	.5013	1287.52	114.91
CL-100*pH8.00*b		7.9128	8.4133	14	1	.5005	1239.53	110.81
CL-100*pH8.50*a		7.8666	8.3532		2	.4866	1168.63	107.45
CL-100*pH8.50*b		7.8423	8.3435		3	.5012	1268.51	113.24
CL-100*pH9.00*a		7.8315	8.3323		4	.5008	1254.14	112.05
CL-100*pH9.00*b		7.7855	8.2844		5	.4989	1221.03	109.50
CL-100*FU1*a		7.8623	8.3617		7	.4994	1217.61	109.09
CL-100*FU1*b		7.8565	8.3633		8	.5068	1232.07	108.77
CL-100*FU2*a		7.8414	8.3438		9	.5024	1254.98	111.76
CL-100*FU2*b		7.7999	8.2628		10	.4629	1123.63	108.61

7/19/93 gp
1430 hrs.

Additional samples for Exp B-8005B were started to fill in some gaps in the initial data.

Equipment, supplies, and procedure are same as on pages 85-87.

Amount of HNO_3 or $NaHCO_3$ added to 40 ml 0.1m $NaNO_3$ solution containing 100 ppb U.

Sample	Volume HNO_3 added ml	Molarity of HNO_3
B-8005B *pH3.51	.05	0.1
B-8005B *pH3.73	.01	0.1

	Volume $NaHCO_3$ added ml	Molarity of $NaHCO_3$
B-8005B *pH3.91	.1	.005
B-8005 B* pH4.06	.27	.01
B-8005 B* pH4.26	.38	.01

Sample	Wt ^{233}U soln added	pH date/time 8/2/93 1345	pH date/time 8/23/93 1500 hrs
B-8005B*pH3.51	40.01	3.55 / 24.1°C	4.04 / 23.8°C
B-8005B*pH3.73	40.03	3.78 / 24.3°C	4.56 / 23.8°C
B-8005B*pH3.91	40.00	3.91 / 24.4°C	4.94 / 23.8°C
B-8005B*pH4.06	40.03	3.98 / 23.1°C	6.00 / 23.8°C
B-8005B*pH4.26	40.04	4.16 / 23.1°C	6.53 / 23.8°C

8/2/93 JP

1600 hrs

Added ~ 0.1000 ± .001 g of 8005 alpha alumina to each sample.

Rack	Vial	Sample wt	CPMB	U (ppb)
14	1	.5023	11119.16	99.69
14	2	.5019	11154.50	102.92
14	3	.5011	11123.01	100.27
14	4	.5018	11136.68	101.35
14	5	.5029	11114.27	99.13
14	6	.5024	11178.44	99.55
14	7	.5029	11133.77	100.87
14	8	.5007	11127.73	100.77
14	9	.5019	11125.11	100.30
14	10	.5008	11111.43	99.30
			JP 8/23/93	
2	1	.5002	11177.32	101.94
	2	.5027	11177.32	102.27
	3	.4995	11212.53	97.15
	4	.4991	11185.44	100.40
	5	.4986	11116.15	90.55
	6	.5003	11147.64	91.08
	7	.4993	11023.68	44.29
	8	.5081	11117.23	44.57
	9	.5000	11070.22	39.30
	10	.5047	11044.15	39.85
			11033.25 JP 8/23/93	

Identification	Date/Time	Vial wt	Sample+ vial wt
B-8005B*pH3.51**	8/2/93 1025	7.8684	8.3707
B-8005B*pH3.51*b		7.8820	8.3839
B-8005B* pH3.73**		7.8933	8.3944
B-8005B*pH3.73*b		7.8737	8.3755
B-8005B*pH3.51**		7.8600	8.3629
B-8005B*pH3.51*b		7.8516	8.3540
B-8005B*pH4.06**		7.9172	8.4201
B-8005B*pH4.06*b		7.8779	8.3786
B-8005B*pH4.26**		7.8724	8.3745
B-8005B*pH4.26*b		7.8502	8.3510
B-8005B*pH3.51**	8/23/93 0900	7.8583	8.3535
B-8005B*pH3.51*b		7.8517	8.3544
B-8005B*pH3.73**		7.8508	8.3503
B-8005B*pH3.73*b		7.7888	8.2879
B-8005B*pH3.91**		7.8426	8.3412
B-8005B*pH3.91*b		7.8657	8.3660
B-8005B*pH4.06**		7.8518	8.3511
B-8005B*pH4.06*b		7.8295	8.3326
B-8005B*pH4.26**		7.8246	8.3246
B-8005B*pH4.26*b		7.8192	8.3239

gf 8/31/93
1000hrs.

Prepared alpha-alumina powders for SEM analysis.

Recoved alpha-alumina powders from ~~the~~ following samples by filtering and drying

B-8005B * pH 4.15
B-8006B * pH 5.00
B-8007B* pH 5.60

These powders were placed in glass vials and labeled as follows.

B-8005B * pH 4.15 * REC
B-8006B * pH 5.00 * REC
B-8007B * pH 5.60 * REC.

These samples along with the original NIST surface area standards were prepared for SEM viewing by sprinkling a small amount of the powder onto carbon tape that was attached to an aluminum stub.

The purpose of the SEM viewing is to determine if the surface morphology of the alpha-alumina powders changes when exposed to the U-bearing solutions in the sorption experiments.

Alumina stubs were labeled as follows and the following samples were placed on each stub.

Stub NO	Sample
1	8005
2	8006
3	8007
4	B-8005B * pH 4.15 * REC
5	B-8006B * pH 5.00 * REC
6	B-8007B * pH 5.60 * REC.

Samples were taken to Harold Saldana in div 06 for coating.

9/8/93 SEM analyses were performed on previously prepared α-alumina powders after sputter coating the samples with iridium.

SEM photos of the unreacted and reacted α-alumina powders are kept in a 3-ring binder entitled "alpha-Alumina Experimental Results".

The photomicrographs show dissolution features on the reacted alpha-alumina such as corroded particle edges & boundaries.

10/6/93 Prepared additional α-alumina powders for SEM analysis

Recovered solid from the following sample

B-8006B* pH2.37
B-8006 B * pH8.62

Powders were placed in glass vials + labeled:

B-8006B* pH2.37*REC
B-8006B* pH8.62*REC

A small amount of each sample was sprinkled onto carbon tape attached to aluminum stubs for SEM viewing.

Stubs were labeled as follows:

No	Sample
1	B-8006 B * pH2.37 * REC
2	B-8006 B * pH 8.62 * REC.

Samples were taken to H. Saldana in Div 06 for coating with iridium.

10/11/93 SEM analyses were performed on 8006 alpha alumina powders prepared previously.

SEM photos are kept in a 3-ring binder entitled "alpha-alumina experimental results"

Photomicrograph show that reacted α-alumina powders show evidence of dissolution; corroded edges and boundaries when compared to unreacted α-alumina powder.

10/6/93 JJP

Recovery of α-alumina powders. After recovery the powders will be analyzed for surface area.

For each of the α-alumina experiments (B-8005B, B-8006B, & B-8007B) the recovered powders will be split into 3 subsamples. (1) Those with final pH's of 2.00 to 3.50; (2) Those with final pH's from 3.50 to 7.00; and (3) those with final pH's of 7.00 to 9.00.

The recovered powders will be placed in containers and labelled as follows:

B-8005B

B-8005B * pH 2.00/3.50 * REC
B-8005B * pH 3.50/7.00 * REC
B-8005B * pH 7.00/9.00 * REC

B-8006B

B-8006B * pH 2.00/3.50 * REC
B-8006B * pH 3.50/7.00 * REC
B-8006B * pH 7.00/9.00 * REC

B-8007B

B-8007B * pH 2.00/3.50 * REC
B-8007B * pH 3.50/7.00 * REC
B-8007B * pH 7.00/9.00 * REC

Powders were recovered by filtering the sample solutions. Powders were not washed and were allowed to air dry before placing in containers. Solutions were placed in 1L plastic bottles labelled "B-8005B Solution", "B-8006B Solution", and "B8007B Solution" for later disposal.

Wt of solutions recovered:

B-8005B-A	Solutions	930.3g
B-8005B-B	Solutions	364.0g
B-8006B	Solutions	957.1g
B-8007B	Solutions	957.8g

1/25/94

Recovery of alpha-alumina powders and ~~233~~ uranium solutions for disposal.

Experiments B-8005, B-8006, and B-8007
Solutions will be filtered into 1L plastic bottles and powders recovered and placed in containers labeled as follows.

B-8005*REC
B-8006*REC
B-8007*REC

Plastic bottles containing solutions will be labelled as follows with soln wts.

	wt bottle	wt bottle +soln	wt soln
B-8005-A Solutions	100.97 g	1054.5 g	953.53 g
B-8006 Solutions	100.27 g	921.1 g	820.83 g
B-8007 Solutions	101.04 g	978.7 g	877.66 g
B-8005-B solutions	100.88 g	705.8 g	604.92 g
B-8006-B Solutions	101.85 g	915.4 g	813.55 g
B-8007-B Solutions	100.91 g	944.8 g	843.89 g

Recovered α-alumina powders were dried acid washed, dried again, weighed, and placed in their respective containers. Acid wash solutions were saved and placed in labeled plastic bottles labeled as follows.

	wt bottle	wt bottle +acid	wt acid
B-8005 Acid solution	91.27	1120.9 g	1029.63 g
B-8006 "	91.83	923.5 g	831.67 g
B-8007 "	91.13	1101.1 g	1009.97 g

Experiments TL and FL
Solutions were poured into a single container for U determination and disposal.

Container labeled as follows

	wt bottle	wt bottle +soln	wt soln
"TL and FL Solutions"	102.77 g	667.3 g	564.53 g

Experiment CL-100

Solution poured into a single plastic bottle for U determination and disposal.

Container labeled as follows:

	wt bottle	wt bottle +soln	wt soln
CL-100 Solution	101.2 g	737.3 g	631.1 g

K-α experiment

Solution filtered into 1L plastic bottle + powder recovered.

Plastic bottle containing solution labelled as follows:

	wt bottle	wt bottle + soln	wt soln
K-α Solution	100.49 g	303.77 g	203.28 g

Recovered α-alumina powders were dried, acid-washed, dried again, weighed, + placed in a container labeled:

K-α * REC

Acid-wash solution was placed in plastic bottle labeled as follows for U determination + disposal.

	wt bottle	wt bottle + soln	wt soln
K-α Acid Solution	91.84	1122.7 g	1030.86 g

8/3/94 JP

Glove Box Preparation for Controlled Atmosphere Sorption Experiments.

Obj: attain 1% CO_2(g) atmosphere for U-sorption on clinoptilolite experiments.

Equipment + Materials:
- Labconco controlled atmosphere glove-box
- ORION pH meter and electrode
- Drager tubes for CO_2
- Gyratory shaker.

Supplies:
- 100 ml 0.05 m $NaHCO_3$ in PP bottle
- 100 ml 0.005 m $NaHCO_3$ in PP bottle
- 100 ml 0.001 m $NaHCO_3$ in PP bottle

Procedure:

a) Prepare 0.05 m $NaHCO_3$ solution by dissolving 4.2005 g $NaHCO_3$ (lot #931791B) in 1L H_2O in a 1L volumetric flask. Transfer 100 ml of this solution to a 125 ml PP bottle labeled 0.05 m $NaHCO_3$. Dilute 10 ml of 0.05 m $NaHCO_3$ solution to 100 ml in a volumetric flask; transfer to 125 ml PP bottle labeled 0.005 m $NaHCO_3$. Dilute 2 ml of 0.05 m $NaHCO_3$ solution to 100 ml in a volumetric flask; transfer to 125 ml PP bottle labeled 0.001 m $NaHCO_3$.

b) Transfer pH meter & electrode, Drager tubes, gyratory shaker, and 3 $NaHCO_3$ solutions to the glove box.

c) Set pressure control module on glove box to 1 inch H_2O then purge and fill glovebox with 1% CO_2(g) three times. Open inner door to transfer chamber and allow glove box to bleed atmosphere while filling with 1% CO_2(g) for a few minute. Close transfer chamber door.

d) Place $NaHCO_3$ solutions on gyratory shaker set at about 100 rpm. Make sure caps are removed so solution open to atmosphere. The pCO_2 of the glove box can be determined by the pH of these solutions at equilibrium. pH's will be measured and recorded periodically to determine equilibrium of the pCO_2.

e) The % CO_2 in the glove box will also be measured with Drager Tubes as a check. The advantage of drager Tubes is that you don't have to wait for solution to reach pH equilibrium which may take several days.

f) Results will be recorded on the following pages.

g) Results will determine if 1% CO_2 atmosphere has been achieved. If not more purging, bleeding, and filling will be necessary.

pH of $NaHCO_3$ Solutions

	pH / vol % CO_2			
	.05m	.005m	.001m	°C
8/4/94	8.74/.35	7.87/.30	7.18/.30	22.9
8/8/94	8.39/.80	7.58/.55	6.81/.70	22.0
8/11/94	8.32/.95	7.46/.75	6.66/.95	22.4
8/15/94	8.38/.80	7.52/.65	6.83/.70	22.4

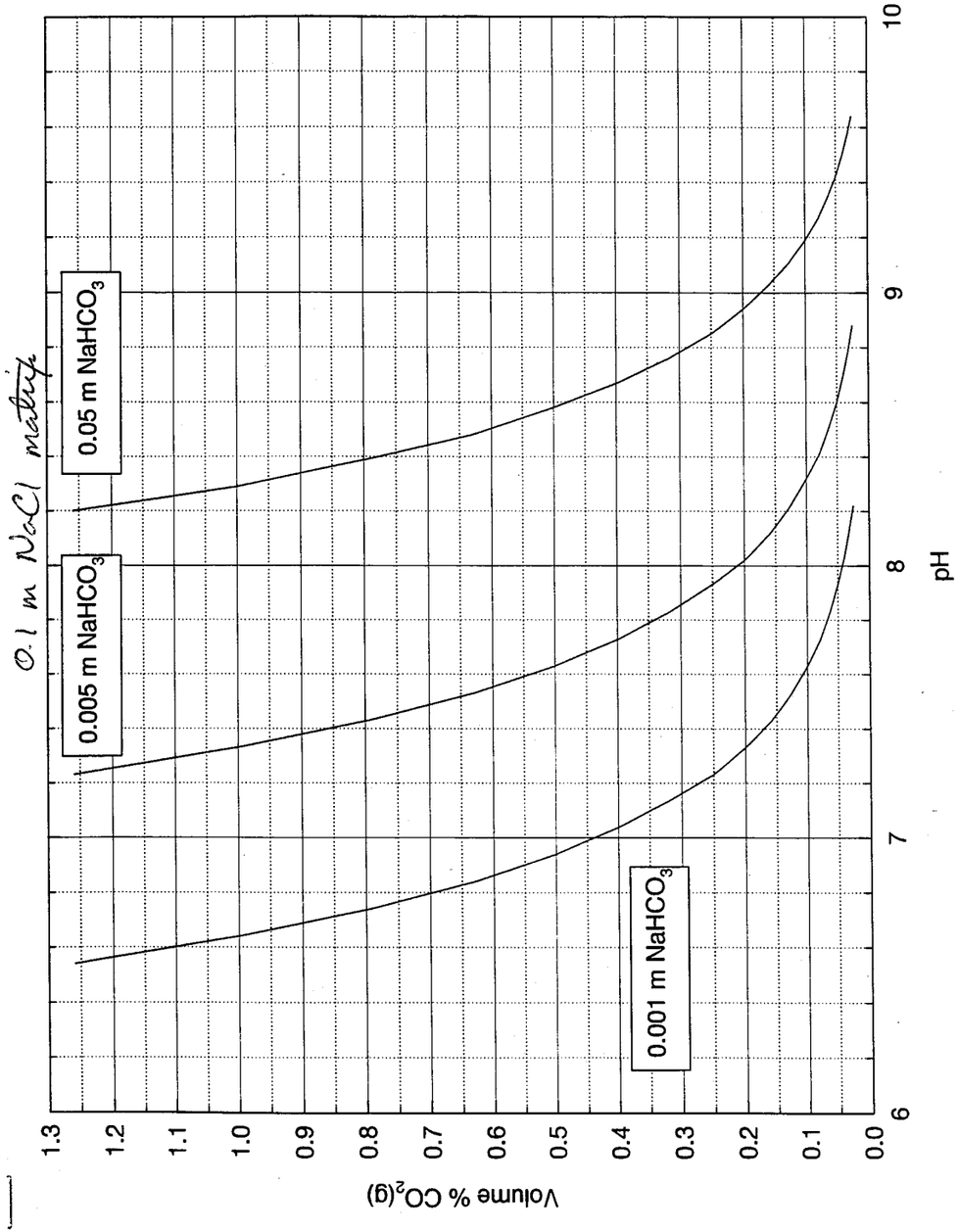
jp 8/15/94 09:00 Caps on pH buffers were removed to allow buffers to equilibrate with glovebox CO_2. After equilibrate (about 10 days) buffers will be measured & these pH (or calibrate values) will be used to determine pH's of samples.

jp 8/22/94 1400 pH meter removed from glove box and calibrated. pH meter returned to glove box and pH buffers measured.

pH 2.00 - 1.96
pH 4.00 - 3.97
pH 7.00 - 6.94
pH 9.00 - 7.93
pH 10.00 - 8.69

cont on p 159

Graph to determine % CO_2 in $NaHCO_3$ solutions by pH.



Drager Tube Readings (vol % CO_2)

gp	8/3/94	.35
gp	8/4/94	.50
gp	8/5/94	.70
gp	8/8/94	.85
gp	8/8/94	1.00
gp	8/11/94	1.10
gp	8/15/94	1.20
gp	8/22/94	1.20

cont. of pH of $NaHCO_3$ solutions from p157

8/22/94 pH meter calibrated using pH's of measured buffers in glovebox (p157).

		pH of $NaHCO_3$ solution			
		.65m	.005m	.001m	°C
gp	8/22/94	8.46/.65	7.58/.57	6.93/.50	

gp 8/23/94 *Note - the graph on p158 is for $NaHCO_3$ solutions in a 0.1 m NaCl matrix, however the $NaHCO_3$ experimental solutions are not in a 0.1 m NaCl matrix. Therefore the previous calculated CO_2 contents are in error.

(see p180 for determination of CO_2 content of glovebox).

8/8/94

URANIUM SORPTION EXPERIMENT E-I:

(Kd vs pH; Equilibrium with $pCO_2=10^{-2.00}$; ΣU=50 ppb; 0.1m $NaNO_3$)

WRITTEN BY: R.T. PABALAN
REVISION NO.: 0

DATE WRITTEN: April 26, 1994
DATE REVISED:

OBJECTIVE:

- To investigate the importance of uranium sorption on the zeolite mineral clinoptilolite as a function of solution pH, total uranium concentration and pCO_2. Experimental data will be correlated with uranium aqueous speciation, and compared with results at $pCO2=10^{-3.5}$ atm.

- To investigate reversibility and reproducibility of uranium sorption reactions.

Note: $NaHCO_3$ solutions are used here to raise the starting pH of most of the uranium solutions. However, because of the higher pCO_2 used in this study, a higher amount of $NaHCO_3$ is needed to raise the starting pH of the uranium solutions. Thus for solutions with starting pH≥7.75, $NaHCO_3$ solid, instead of solution, is added to raise the solution pH. The amounts of HNO_3 and $NaHCO_3$ needed to adjust the pH were calculated using EQ3 V7.2 (neglecting the effects of H^+ ion-exchange at low pH). Weights of the bottles are measured at each step to account for evaporation losses.

EQUIPMENT:

Gyratory shaker or constant temperature shaker bath
Packard liquid scintillation counter
Labconco controlled atmosphere glove-box
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance

SUPPLIES:

	Supply of gas mixture (1% CO_2 in air); with certificate of analysis
	pH buffer (pH = 2,4,7,9,10)
1	60-ml FEP bottle (to contain E-I*IU)
39	60-ml FEP bottles (to contain experimental mixtures and control solutions)
1	2000-ml **polycarbonate** bottle (for preparation of 50 ppb U solution)
1	5-ml Eppendorf pipet (for transferring 5 ml cocktail into scintillation vial)
1	0.5-ml Eppendorf fixed-volume micropipet (for taking samples and for transferring 0.02 M HNO_3 solution into scintillation vial)
various	Eppendorf micropipets (fixed- or variable-volume; for adding HNO_3 or $NaHCO_3$ solutions to uranium solutions)
	scintillation vials
	weighing paper
	Na-clinoptilolite (CDV*100/200*UC*WA*HL*CPT*Naf)
	reagent grade $NaHCO_3$
	500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike (23A)
4 L	0.1 m $NaNO_3$ stock solution (Lot# 930601) 3.499 g $NaNO_3$ / L H_2O
1000 ml	stock solution of 1.0 m HNO_3

Copy of certificate of analysis for $CO_2(c_y)$

2801 MONTOPOLIS DR.
P.O. BOX 17877
AUSTIN, TEXAS 78760
PHONE 512/389-2323
FAX 512/389-2599

Wilson Oxygen

"Quality Today To Insure Tomorrow"

Date Received: August 9, 1994
Date Filled: August 9, 1994

Product: PRIMARY STANDARD MIXTURE
Batch Number: 94-0221-02

Mixture Components	Requested Composition*	Analytical Results*†
Carbon Dioxide	**1.00 %**	**0.99 %**
Air	**Balance**	**Balance**

**Mole Percent*
†±1.0 % Relative to the Concentration of the Minor Component

Cylinders Included In This Batch:

T771127

Approved By:

Title:Lab Technician

1000 ml	stock solution of 0.1 m HNO_3
1000 ml	stock solution of 0.02 m HNO_3
500 ml	stock solution of 1.0 m $NaHCO_3$
500 ml	stock solution of 0.5 m $NaHCO_3$
500 ml	stock solution of 0.1 m $NaHCO_3$
500 ml	stock solution of 0.05 m $NaHCO_3$
500 ml	stock solution of 0.01 m $NaHCO_3$
	ultrapure water

PROCEDURE:

Note: In transferring uranium solutions, avoid using glass or polypropylene labware.

Solution E-I (1 bottle for each pH value)

- Initial $\sum U$ = 50 ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit; adjustments made with HNO_3 or $NaHCO_3$
- Initial volume = 50 ml
- Ionic strength = 0.1 m $NaNO_3$
- Wt. Na-clinoptilolite to use = 0.100±0.001
- Initial $[Na^+]$ = 0.1 m $NaNO_3$ + $[NaHCO_3]$ added
- $pCO_2 = 10^{-2.00}$ atm

3/8/94

a) Prepare 2000 g of 50 ppb U solution in a pre-cleaned 2-liter polycarbonate bottle by diluting 200 g of a 500 ppb stock solution (in 0.1 m $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 2000 g by carefully taring 0.1 m $NaNO_3$ solution into the polycarbonate bottle on a Mettler 4600 balance. Used spike 28A

10:00 hrs

b) Into each of 29 60-ml **preweighed** FEP bottle labeled E-I*pH*i* [where *i* is the approximate initial pH of the solution (see below)], tare 50 g of the 50 ppb uranium solution. Record the weight of each solution.

1010 hrs

Into each of 10 60-ml **preweighed** FEP bottle labeled E-I-C*pH*i* [where *i* is 2, 4, 5, 5.5, 6, 6.5, 7, 7.5, 8, or 9.5, representing the approximate initial pH of the solution], tare 50 g of the 50 ppb uranium solution. Record the weight of each solution. These are control solutions to determine uranium loss to the container walls as a function of pH.

1100 hrs

(Cap the bottles tightly at this time if pH adjustment is to be made at a later time, i.e., a few hours later).

Transfer the remaining solution into a 60-ml FEP bottle labeled E-I*IU. **Add 100 µL of 50% HNO_3 solution**, then mix thoroughly. Take two 0.5-ml samples from E-I*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., E-I-IU*a (or b)] and pre-weighed scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO_3. Reweigh each vial. Homogenize the mixture and save for later analysis of uranium concentration by liquid scintillation counting.

1130 hrs

c) For each solution E-I*pH*i* and E-I-C*pH*i*:

gp 8/3/94 1300hrs

Adjust the pH of each solution to the approximate value *i* by adding HNO_3 or $NaHCO_3$ solution (with an Eppendorf micropipet), or $NaHCO_3$ solid (previously tared on weighing paper). The approximate amount to be added is given in Table E-I-1. Swirl the solutions by hand, then reweigh each bottle. If HNO_3 or $NaHCO_3$ solution was added, record the microliter volume and concentration of solution added. *Do not measure the pH at this time.* Cap each bottle tightly, then transfer into the controlled atmosphere glove box.

gp 8/3/94 1515hrs

see p155-159

Prepare and set the glove box to the correct atmosphere. Replace the screw caps on each bottle with a porous material (e.g., kimwipe). Place the bottles on a gyratory shaker set to ≈120 rpm. Leave the bottles on the shaker for about ten days to allow the solutions to reach equilibrium with the 1% CO_2(g) atmosphere. Monitor the pressure in the glove box frequently.

8/25/94 1400 hrs

d) Measure and record the pH of each solution E-I*pH*i* and E-I-C*pH*i*. *Minimize the amount of time the glass electrode is in contact with the uranium solution. Make sure to rinse the electrode well before transferring into another solution.*

8/26/94 1000 hrs

From each solution E-I*pH*i* and E-I-C*pH*i*, take 2 0.5-ml sample with an Eppendorf pipet, transfer into pre-labeled [e.g., E-I*IU-ph*i**a (or b)] and pre-weighed scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO_3.

8/26/94 1100hrs

Transfer the scintillation vials out of the glove box. Homogenize the mixtures. Reweigh each vial and save for later analysis of uranium concentration by liquid scintillation counting. The measured concentrations are the initial values to be used in the calculation of sorption data.

8/26/94 1130hrs

31 gp 8/25/94

e) Tare ~~29~~ batches of 0.100±0.001 gm of Na-clinoptilolite onto weighing paper; transfer these into the glove box. Then carefully add Na-clinoptilolite into each of the E-I*pH*i* (**not** the E-I-C*pH*i*) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker.

9/6/94 0830hrs

f) After equilibrium is reached (at least 10 days), take 2 0.5-ml samples from each bottle E-I*ph*i* and E-I-C*pH*i* with an Eppendorf pipet, transfer into pre-labeled [e.g., E-I-ph*i**a (or b)] and pre-weighed scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO_3.

Transfer the scintillation vials out of the glove box. Homogenize the mixtures. Reweigh each vial and save for later analysis of uranium concentration by liquid scintillation counting.

Measure and record the pH and temperature of solutions E-I*pH*i* and E-I-C*pH*i*. Make sure to rinse the pH electrode very well before transferring into another solution.

9/7/94 0800hrs

g) Analyze the U concentration by liquid scintillation counting.

****

Hold Point. Check quality of experimental data. Resample if needed.

****

f) If the analytical results are good, reversibility and reproducibility tests can be done by changing the pH of the solutions and re-equilibrating them at the new pH values. Alternatively, reversibility in terms of pCO_2 effect can be tested by taking the solutions out of the glove box and equilibrating them at atmospheric pCO_2 ($10^{-3.5}$ atm).

Procedure for reversibility and reproducibility experiments will be written later.

PREPARATION:

1. Preclean:
   - 39 60-ml FEP bottles (to contain experimental mixtures and control solutions)
   - 1 60-ml FEP bottle (to contain E-I*IU)
   - 1 2000-ml polycarbonate bottle (for preparation of 50 ppb U solution)

2. Prepare:
   - 500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike (238A).
   - 4 L 0.1 m $NaNO_3$ stock solution Lot 930601
   - 1000 ml stock solution of 1.0 m HNO_3
   - 1000 ml stock solution of 0.1 m HNO_3
   - 1000 ml stock solution of 0.02 m HNO_3
   - 500 ml stock solution of 1.0 M $NaHCO_3$ (42.005 g in 500 ml solution)
   - 500 ml stock solution of 0.5 M $NaHCO_3$ (21.003 in 500 ml solution)
   - 500 ml stock solution of 0.1 M $NaHCO_3$ (4.201 g in 500 ml solution)
   - 500 ml stock solution of 0.05 M $NaHCO_3$ (2.100 g in 500 ml solution)
   - 500 ml stock solution of 0.01 M $NaHCO_3$ (0.4201 g in 500 ml solution)

The $NaHCO_3$ solutions should be prepared with *degassed* deionized water and kept in tightly-capped glass reagent bottles.

Table E-I-1. Amount of reagent grade HNO_3 or $NaHCO_3$ solutions or $NaHCO_3$ solid to add to 50 ml 0.1 m $NaNO_3$ solution containing 50 ppb U to result in pH values given in column-1. The amount of reagent to be added was estimated using EQ3 calculations (and rounded off to nearest 5 or 10 microliters).

Solution pH	Volume of HNO_3 needed, microliters	Molarity of HNO_3 to use
2.00	600	1
2.25	340	1
2.50	190	1
2.75	100	1
3.00	560	0.1
3.25	300	0.1
3.50	150	0.1
3.75	330	0.02
4.00	100	0.02
Solution pH	**Volume of $NaHCO_3$ needed, microliters**	**Molarity of $NaHCO_3$ solution to use**
4.25	80	0.01
4.5	240	0.01
4.75	350	0.01
5	450	0.01
5.25	110	0.05
5.5	140	0.05
5.75	200	0.05
6	290	0.05
6.25	230	0.1
6.5	380	0.1
6.75	130	0.5
7	225	0.5
7.25	390	0.5
7.5	350	1

Solution pH	Grams of $NaHCO_3$ reagent to use
7.75	0.0524
8	0.0948
8.25	0.1749
8.5	0.3329
8.75	0.6701
9	1.4947
9.25	3.9844
9.5	13.6056

lot #931739B

grams used

0.0524
0.0955
0.1752
0.3332
0.6720
1.4947
3.9854
13.6066

Sample Weights

gp 8/8/94 1245

I.D.	gp 8/8/94 10:10 Wt. soln added 50 ppb ^{233}U (g)	gp 8/8/94 1230 Wt sample w/o cap (g)	gp 8/8/94 1240 Wt Sample w cap (g)	gp 8/8/94 1420 Wt after pH adj w/cap (g)	RB 8/25/94 1300 wt before taking sample w/cap (g)	gp 8/25/94, 1430 pH/°C	RB 8/26/94 wt after 1030 taking sample w/cap (g)	8/26/94 gp wt after clamps added w/cap (g)
E-I* pH 2.00	50.03	74.37	78.13	78.15	76.79	1.93 / 24.7	75.71	75.82
E-I* pH 2.25	50.01	68.40	72.06	72.41	70.19	2.17 / 24.6	69.18	69.29
E-I* pH 2.50	50.01	73.42	77.27	77.47	75.43	2.40 / 24.5	74.41	74.52
E-I* pH 2.75	50.00	71.74	75.51	75.61	73.11	2.69 / 24.5	72.07	72.18
E-I* pH 3.00	50.04	70.31	74.03	74.59	72.07	2.92 / 24.4	71.06	71.17
E-I* pH 3.25	50.05	67.72	71.60	71.91	69.32	3.18 / 24.2	68.30	68.41
E-I* pH 3.50	50.01	72.58	76.26	76.41	74.24	3.44 / 23.9	73.23	73.34
E-I* pH 3.75	50.02	73.61	77.44	77.77	75.78	3.67 / 24.0	69.22	69.33
E-I* pH 4.00	50.03	70.28	73.97	74.07	71.97	3.95 / 24.1	70.94	71.06
E-I* pH 4.25	50.02	68.57	72.40	72.48	70.42	4.28 / 25.5	69.39	69.50
E-I* pH 4.50	50.04	72.32	76.07	76.31	73.95	4.65 / 25.5	72.92	73.04
E-I* pH 4.75	50.00	74.36	78.23	78.58	76.24	4.95 / 25.5	75.19	75.30
E-I* pH 5.00	50.03	72.74	76.49	76.93	74.46	5.21 / 25.4	73.46	73.58
E-I* pH 5.25	50.02	73.86	77.67	77.78	75.72	5.42 / 25.4	74.67	74.79
E-I* pH 5.50	50.04	69.57	73.25	73.40	70.76	5.57 / 25.3	69.76	69.88
E-I* pH 5.75	50.03	68.99	72.70	72.93	70.27	5.83 / 25.3	69.25	69.37
E-I* pH 6.00	50.01	71.85	75.59	75.87	73.33	6.02 / 25.2	72.29	72.40
E-I* pH 6.25	50.01	72.81	76.56	76.81	74.58	6.26 / 25.2	73.58	73.70
E-I* pH 6.50	50.03	71.70	75.48	75.87	73.69	6.50 / 25.1	72.66	72.77
E-I* pH 6.75	50.02	72.40	76.06	76.19	74.09	6.76 / 25.0	73.08	73.19
E-I* pH 7.00	50.04	72.17	75.96	76.20	73.81	7.02 / 24.9	72.77	72.88
E-I* pH 7.25	50.01	73.68	77.36	77.76	75.38	7.23 / 22.2	74.39	74.50
E-I* pH 7.50	50.01	72.28	75.99	76.35	74.08	7.50 / 22.2	73.07	73.18
E-I* pH 7.75	50.02	72.05	75.78	75.83	73.53	7.73 / 22.2	72.53	72.64
E-I* pH 8.00	50.02	70.93	74.62	74.70	72.14	8.01 / 22.1	71.17	71.28
E-I* pH 8.25	50.02	72.50	76.42	76.58	74.05	8.27 / 22.1	73.05	73.15
E-I* pH 8.50	50.05	72.42	76.11	76.42	74.31	8.53 / 22.1	73.29	73.40
E-I* pH 8.75	50.00	72.04	75.78	76.44	74.24	8.78 / 22.1	73.23	73.34
E-I* pH 9.00	50.04	72.48	76.40	77.87	75.40	9.02 / 22.1	74.36	74.47
E-I* pH 9.25	50.05	72.48	76.25	79.96	77.09	9.24 / 22.0	76.01	76.12
E-I* pH 9.50	50.03	73.45	77.10	90.65	87.46	9.23 / 22.0	86.34	86.45

Sample Weights

	I.D.	8/8/94 1100 wt 500ppb 233y soln added (g)	8/8/94 1250 wt sample w/o cap (g)	8/8/94 1255 wt sample w/cap (g)	8/8/94 1420 wt after pH w/cap (g)	PB 8/25/94 1300 wt before tare, sample w/cap (g)	gp 8/25/94 1425 pH/°C	PB 8/26/94 wt after 10hrs tare, sample w/cap (g)	8/26/94 gp wt after Cl2 added w/cap (g)
gp 8/8/94	E-I-C*pH2.00	50.02	71.87	75.60	76.20	73.86	1.93/24.6	72.82	72.83
	E-I-C*pH4.00	50.08	74.18	77.96	78.06	75.72	3.97/24.3	74.70	74.71
	E-I-C*pH5.00	50.04	71.66	75.61	76.06	73.41	5.22/25.4	72.41	72.42
	E-I-C*pH5.50	50.06	72.14	75.87	76.01	73.86	5.54/25.3	72.84	72.85
	E-I-C*pH6.00	50.04	70.41	74.36	74.65	72.05	6.00/25.2	71.04	71.04
	E-I-C*pH6.50	50.00	70.32	74.16	74.54	72.04	6.49/25.1	71.00	71.01
	E-I-C*pH7.00	50.02	73.13	76.94	77.16	74.70	7.00/24.9	73.69	73.70
	E-I-C*pH7.50	50.01	73.99	77.88	78.23	75.85	7.49/22.2	74.86	74.87
	E-I-C*pH8.00	50.01	73.53	77.16	77.25	74.93	8.01/22.2	73.92	73.94
	E-I-C*pH9.50	50.05	72.38	76.59	90.19	85.95	9.22/22.1	84.80	84.81

Results

ID	Date/Time	Vial wt +5ml cocktail +0.5ml 0.02M HNO_3	Sample+ vial wt +5ml cocktail +0.5ml 0.02M HNO_3	RACK	VIAL	Sample Wt	CPMB	U(PPM)
E-I-Iu*a	8/8/94 1315	12.7356	13.2338	16	2	.4982	524.277	47.084
E-I-Iu*b	8/8/94 1315	12.8157	13.3144	16	3	.4987	541.914	48.619
E-I*Iu* pH2.00*a	8/25/94 0800	12.7285	13.2233	16	5	.4948	529.731	47.901
E-I*Iu*pH2.00*b		12.6840	13.1933	16	6	.5093	563.135	49.471
" pH2.25*a		12.7527	13.2529	16	7	.5002	544.544	48.709
" pH2.25*b		12.6982	13.2004	16	8	.5022	552.638	49.236
" pH2.50*a		12.6684	13.1644	16	9	.4960	532.180	48.006
" pH2.50*b		12.6622	13.1485	16	10	.4863	521.786	48.007
" pH2.75*a		12.6940	13.1903	16	11	.4963	549.632	49.550
" pH2.75*b		12.7112	13.2190	16	12	.5078	551.127	48.560
" pH3.00*a		12.7671	13.2699	16	13	.5028	552.638	49.177
" pH3.00*b		12.7106	13.2143	16	14	.5037	558.633	49.622
" pH3.25*a		12.7528	13.2512	16	15	.4984	546.082	49.023
" pH3.25*b		12.7558	13.2549	16	16	.4991	535.911	48.042
" pH3.50*a		12.7358	13.2357	16	17	.4999	543993	48.688
" pH3.50*b		12.7226	13.2312	16	18	.5086	530.492	46.668
" pH3.75*a		12.7115	13.2157	17	1	.5042	502440	44.586
" pH3.75*b		12.7217	13.2215	17	2	.4998	512.415	45.871
" pH4.00*a		12.7061	13.2069	17	3	.5008	523.178	46.741
" pH4.00*b		12.8164	13.2977	17	4	.4813	497.314	46.231
" pH4.25*a		12.6561	13.1578	17	5	.5017	467.220	41.667
" pH4.25*b		12.7248	13.2338	17	6	.5090	473.272	41.601
" pH4.50*a		12.7220	13.2227	17	7	.5007	298.061	26.634
" pH4.50*b		12.6872	13.1968	17	8	.5096	302.346	26.545
" pH4.75*a		12.7203	13.2116	17	9	.4913	341.367	31.088
" pH4.75*b		12.6869	13.2148	17	10	.5279	321.492	27.248
" pH5.00*a		12.7490	13.2491	17	11	.5001	181.657	16.252
" pH5.00*b		12.6824	13.1812	17	12	.4988	183.089	16.423
" pH5.25*a		12.7113	13.2127	17	13	.5014	355.894	31.758
" pH5.25*b		12.7578	13.2659	17	14	.5081	369.854	32.568
" pH5.50*a		12.7171	13.2161	17	15	.4990	362.865	32.536
" pH 5.50*b		12.7021	13.2042	17	16	.5021	370.712	33.034

Results

ID	Date/Time	Vial wt +5ml cocktail +0.5ml @0.2m HNO_3	Sample+vial wt +5ml cocktail +0.5ml @0.2m HNO_3
E-I*IW*pH 5.75*a	8/24/94 0800	12.6551	13.1528
" pH 5.75*b		12.7284	13.2290
" pH6.00*a		12.6746	13.1738
" pH6.00*b		12.7549	13.2600
" pH6.25*a		12.6951	13.1931
" pH6.25*b		12.7069	~~13.20~~ 13.2100 jp 8/26/94
" pH6.50*a		12.6945	13.1947
" pH6.50*b		12.7144	13.2202
" pH6.75*a		12.6755	13.1769
" pH6.75*b		12.7068	13.2040
" pH7.00*a		12.7374	13.2391
" pH7.00*b		12.7124	13.2199
" pH7.25*a		12.6798	13.1783
" pH7.25*b		12.7328	13.2326
" pH7.50*a		12.7284	13.2279
" pH7.50*b		12.6794	13.1781
" pH7.75*a		12.7180	13.2071
" pH7.75*b		12.7352	13.2467
" pH8.00*a		12.6786	13.1784
" pH8.00*b		12.7467	13.2465
" pH8.25*a		12.7252	13.2270
" pH8.25*b		12.7570	13.2656
" pH8.50*a		12.7492	13.2531
" pH8.50*b		12.7132	13.2173
" pH8.75*a		12.7404	13.2422
" pH8.75*b		12.7237	13.2285
" pH9.00*a		12.7490	13.2596
" pH9.00*b		12.7343	13.2473
" pH9.25*a		12.6998	13.2213
" pH9.25*b		12.7645	13.3027
" pH9.50*a		12.7044	13.2399
" pH9.50*b		12.7695	13.3307

RACK	VIAL	Sample wt	CPMB	Th (ppm)
17	17	.4977	259.715	23.348
17	18	.5006	260.027	23.240
18	1	.4992	382.575	34.289
18	2	.5051	385.197	34.121
18	3	.4980	472.467	42.448
18	4	.5031	486.715	43.285
18	5	.5002	478.111	42.766
18	6	.5058	509.802	45.096
18	7	.5014	537.879	47.997
18	8	.4972	520.756	46.861
18	9	.5017	546.885	48.772
18	10	.5075	541.246	47.717
18	11	.4985	551.694	49.516
18	12	.4998	556.135	49.785
18	13	.4995	542.037	48.552
18	14	.4987	563.602	50.565
18	15	.4891	534.895	48.931
18	16	.5115	569.576	49.822
18	17	.4998	554.856	49.671
18	18	.4998	554.605	49.648
5	1	.5018	568.360	50.677
5	2	.5085	566.638	49.857
5	3	.5039	543.624	48.269
5	4	.5041	567.151	50.338
5	5	.5018	543.747	48.482
5	6	.5048	551.444	48.876
5	7	.5106	556.135	48.732
5	8	.5130	557.168	48.594
5	9	.5215	548.623	47.069
5	10	.5382	550.810	45.790
5	11	.5355	471.344	39.382
5	12	.5612	423.823	33.790

Results

ID	Date/Time	Vial wt +5ml cocktail +0.5ml 0.02m HNO_3	Sample+Vial wt +5ml cocktail +0.5ml 0.02M HNO_3
E-I-C*Iu*pH2.00*a	8/24/44 0800	12.7119	13.2116
" pH2.00*b		12.6767	13.1756
" pH4.00*a		12.6944	13.1923
" pH4.00*b		12.7121	13.2144
" pH5.00*a		12.7899	13.2868
" pH5.00*b		12.7107	13.2076
" pH5.50*a		12.7106	13.2073
" pH5.50*b		12.7477	13.2511
" pH6.00*a		12.7449	13.2450
" pH6.00*b		12.6890	13.1894
" pH6.50*a		12.7270	13.2248
" pH6.50*b		12.7325	13.2327
" pH7.00*a		12.7637	13.2593
" pH7.00*b		12.7241	13.2246
" pH7.50*a		12.7121	13.2102
" pH7.50*b		12.6812	13.1832
" pH8.00*a		12.7041	13.2029
" pH8.00*b		12.7074	13.2067
" pH9.50*a		12.7109	13.2591
" pH9.50*b		12.6748	13.2361

RACK	VIAL	Sample wt	CPMB	W(ppm)
5	14	.4997	557.294	49.898
5	15	.4989	555.809	49.846
5	16	.4979	527.187	47.374
5	17	.5023	517.013	46.053
5	18	.4969	324.925	29.257
1	1	.4969	338.036	30.437
1	2	.4967	470.333	42.367
1	3	.5034	471.424	41.900
1	4	.5001	343.055	30.692
1	5	.5004	365.461	32.677
1	6	.4978	486.065	43.687
1	7	.5002	489.108	43.750
1	8	.4956	517.013	46.675
1	9	.5005	520.077	46.492
1	10	.4981	548.747	49.291
1	11	.5020	561.486	50.044
1	12	.4988	547.057	49.089
1	13	.4993	568.874	50.976
1	14	.5482	445.175	36.334
1	15	.5613	428.064	34.122

8/23/94 gp CO_2 content of glovebox - cont from p 159.

The pH of $NaHCO_3$ solution in glovebox will continued to be measured. A graph showing the CO_2 content of these solutions with respect to pH is shown on the following page.

In addition $NaHCO_3$ solution in 0.1 m NaCl were prepared and placed in glovebox. The pH of these solution will also be monitored to determine the CO_2 content of the glovebox. The graph on p 158 will be used to determine the CO_2 content of these solutions.

$NaHCO_3$ solutions in 0.1 m NaCl matrix were prepared as follows:

0.05 m $NaHCO_3$ in 0.1 m NaCl - 100 ml prepared by dissolving .5844 g NaCl (lot 912763) and .42 g $NaHCO_3$ (lot 931739B) in H_2O in a 100 ml volumetric flask.

0.005 m $NaHCO_3$ in 0.1 m NaCl - 100 ml prepared by dissolving .5844 g NaCl (lot 912763) and .042 g $NaHCO_3$ (lot 931739B) in H_2O in a 100 ml volumetric flask.

0.001 m $NaHCO_3$ in 0.1 m NaCl - 100 ml prepared by dissolving .5844 g NaCl (lot 912763) and .0084 g $NaHCO_3$ (lot 931739B) in H_2O in a 100 ml volumetric flask.

These solutions were placed in 125 ml labeled PP bottles and placed in glovebox.

8/25/94 gp Measurement of pH buffers in glovebox

-pH meter was removed from glovebox and calibrated. pH meter was then returned to glovebox & the pH of buffers inside box (equilibrated with 1% CO_2) were measured.

pH 2.00	- 1.96	21.6°C
pH 4.00	- 3.98	21.6°C
pH 7.00	- 6.94	21.8°C
pH 9.00	- 7.94	21.8°C
pH 10.00	- 8.71	21.8°C

The measured pH's of the buffers within the glovebox will be used to calibrate meter & measure pH of samples.

8/25/94 gp Measurement of $NaHCO_3$ solutions

$NaHCO_3$ solutions in H_2O

		.05 m	.005 m	.001 m	
gp	8/25/94	8.45	7.58	6.89	22.0°C
gp	9/7/94	8.45	7.56	6.95	21.1°C

$NaHCO_3$ solutions in .1m NaCl matrix

		.05m	.005m	.001 m	
gp	8/25/94	8.33/.92	7.37/.92	6.67/.94	22.2°C
gp	8/31/94	8.30/.98	7.34/.98	6.66/.96	20.6°C
gp	9/7/94	8.35/.88	7.42/.82	6.71/.86	21.1°C
gp	9/9/94	8.32/.94	7.38/.89	6.71/.86	19.7°C
gp	9/21/94	8.34/.89	7.41/.84	6.71/.86	20.5°C

9/6 E-I Sample Weights (cont) 9/6 gp 9/6/94

~~6/9/94~~ gp 1030hrs ~~6/9/94~~ gp 9/6/94 1430 ~~6/9/94~~ gp 9/6/94 gp

ID	wt before sample taken w/cap (g)	pH/°C	wt after sample + pH adjustment w/cap (g)
E*I-pH2.00	75.13	1.98/21.6	74.11
E-I*pH2.25	68.55	2.21/21.6	67.55
" pH2.50	73.76	2.44/21.7	72.74
" pH2.75	71.25	2.75/21.7	70.22
" pH3.00	70.27	2.98/21.9	69.24
" pH3.25	67.48	3.23/21.9	66.44
" pH3.50	72.61	3.54/22.0	71.59
" pH3.75	68.63	6/9/94 gp ~~3.85~~ 3.89/22.0	67.62
" pH4.00	70.33	4.23/22.3	69.32
" pH4.25	68.59	4.58/22.4	67.59
" pH4.50	72.01	4.99/22.5	70.99
" pH4.75	74.50	5.15/22.6	73.48
" pH5.00	72.75	5.26/22.7	71.73
" pH5.25	74.11	5.47/22.7	73.09
" pH5.50	69.14	5.65/22.7	68.11
" pH5.75	68.53	5.89/22.8	67.52
" pH6.00	71.52	6.06/22.8	70.46
" pH6.25	72.90	6.31/22.9	71.88
" pH6.50	71.76	6.54/22.9	70.73
" pH6.75	72.35	6.78/23.0	71.34
" pH7.00	72.06	7.05/19.9	71.06
" pH7.25	73.66	7.26/20.0	72.64
" pH7.50	72.39	7.51/20.1	71.37
" pH7.75	71.99	7.75/20.2	70.98
" pH8.00	70.35	8.02/20.2	69.35
" pH8.25	72.26	8.28/20.3	71.28
" pH8.50	72.60	8.54/20.3	71.60
" pH8.75	72.47	8.80/20.3	71.42
" pH9.00	73.77	9.04/20.3	72.73
" pH9.25	75.26	9.27/20.4	74.19
" pH9.50	85.63	9.32/20.5	84.47

ID	Sample Weights 9/6/94 gf wt before sample taken w/cap (g)	9/6/94 1430 gf pH/°C	9/6/94 wt after sample & pH measurement w/cap (g)
E-I-C * pH2.00	71.99	1.91/21.8	70.40
" pH4.00	74.16	4.01/21.5	73.15
" pH5.00	71.58	5.34/21.3	70.64
" pH5.50	72.19	5.60/21.3	71.18
" pH6.00	70.17	6.06/23.1	69.15
" pH6.50	70.35	6.53/23.2	69.35
" pH7.00	73.00	7.04/20.5	72.01
" pH7.50	74.29	7.52/20.6	73.29
" pH8.00	73.44	8.03/20.6	72.43
" pH9.50	84.27	9.31/20.7	83.13

Results

ID	Date/Time	gp 10/4/94 Vial wt + ~~5.5~~ ml cocktail + 0.5 ml 0.02 M HNO_3	gp 10/4/94 Sample + vial wt + ~~5.5~~ ml cocktail + 0.5 ml 0.02 M HNO_3
E-I * pH2.00 *a	9/6/94 0915	12.7918	13.2959
" pH2.00 *b		12.7471	13.2524
" pH2.25 *a		12.7599	13.2662
" pH2.25 *b		12.7795	13.2843
" pH2.50 *a		12.7500	13.2524
" pH2.50 *b		12.7189	13.2215
" pH2.75 *a		12.6974	13.1992
" pH2.75 *b		12.7337	13.2361
" pH3.00 *a		12.6609	13.1626
" pH3.00 *b		12.7118	13.2128
" pH3.25 *a		12.7122	13.2147
" pH3.25 *b		12.7690	13.2731
" pH3.50 *a		12.7585	13.2597
" pH3.50 *b		12.7675	13.2688
" pH3.75 *a		12.6818	13.1851
" pH3.75 *b		12.7784	13.2792
" pH4.00 *a		12.7361	13.2378
" pH4.00 *b		12.7228	13.2255
" pH4.25 *a		12.7128	13.2134
" pH4.25 *b		12.7522	13.2524
" pH4.50 *a		12.7798	13.2785
" pH4.50 *b		12.7680	13.2682
" pH4.75 *a		12.8276	13.3272
" pH4.75 *b		12.7930	13.2923
" pH5.00 *a		12.7661	13.2686
" pH5.00 *b		12.7288	13.2286
" pH5.25 *a		12.7597	13.2588
" pH5.25 *b		12.7724	13.2715
" pH5.50 *a		12.7474	13.2467
" pH5.50 *b		12.7448	13.2411
" pH5.75 *a		12.7587	13.2600
" pH5.75 *b		12.7700	13.2703

RACK	VIAL	Sample wt	CPMB	U (ppm) gp 8/31/94
14	3	.5041	557.567	49.488
14	4	.5053	551.223	48.808
14	5	.5063	549.844	48.590
14	6	.5048	565.880	50.156
14	7	.5024	563.447	50.179
14	8	.5026	551.031	49.053
14	9	.5018	555.604	49.539
14	10	.5024	552.041	49.163
14	11	.5017	541.163	48.261
14	12	.5010	546.858	48.837
14	13	.5025	534.578	47.598
14	14	.5041	536.537	47.621
14	15	.5012	509.413	45.475
14	16	.5023	511.905	45.598
14	17	.5033	460.493	40.937
14	18	.5008	463.732	41.430
15	1	.5017	444.997	39.685
15	2	.5027	442.001	39.340
15	3	.5006	354.661	31.698
15	4	.5002	359.467	32.154
15	5	.4987	179.765	16.128
15	6	.5002	174.941	15.648
15	7	.4996	226.802	20.311
15	8	.4993	229.646	20.578
15	9	.5025	157.894	14.059
15	10	.4998	156.179	13.981
15	11	.4991	242.870	21.772
15	12	.4991	241.864	21.682
15	13	.4993	233.371	20.912
15	14	.4963	235.663	21.245
15	15	.5013	165.607	14.781
15	16	.5003	168.974	15.111

Results.

ID	Date/Time	gp 10/4/94 Vial wt +- 5.0 ~~0.5~~ml cocktail +0.5ml 0.02m HNO_3	gp 10/4/94 Sample+vial wt +5.0 ~~+0.5~~ml cocktail +0.5ml 0.02m HNO_3
E-I*pH6.00*a	9/6/94 0915	12.7132	13.2149
" pH6.00*b		12.7988	13.3011
" pH6.25*a		12.8261	13.3273
" pH6.25*b		12.7565	13.2570
" pH6.50*a		12.7903	13.2889
" pH6.50*b		12.7275	13.2273
" pH6.75*a		12.8153	13.3142
" pH6.75*b		12.8182	13.3190
" pH7.00*a		12.7381	13.2385
" pH7.00*b		12.7240	13.2227
" pH7.25*a		12.7881	13.2880
" pH7.25*b		12.7422	13.2428
" pH7.50*a		12.7996	13.3011
" pH7.50*b		12.7328	13.2342
" pH7.75*a		12.7030	13.2018
" pH7.75*b		12.7245	13.2240
" pH8.00*a		12.7572	13.2596
" pH8.00*b		12.6953	13.1960
" pH8.25*a		12.7583	13.2598
" pH8.25*b		12.7512	13.2533
" pH8.50*a		12.7467	13.2486
" pH8.50*b		12.7570	13.2598
" pH8.75*a		12.7955	13.2992
" pH8.75*b		12.7482	13.2532
" pH9.00*a		12.7720	13.2879
" pH9.00*b		12.8640	13.3820
" pH9.25*a		12.8124	13.3387
" pH9.25*b		12.7022	13.2281
" pH9.50*a		12.7368	13.2893
" pH9.50*b		12.7465	13.3060

RACK	VIAL	Sample wt	cpmB	U(ppb)
15	17	.5017	225.887	20.145
15	18	.5023	253695	22.598
16	1	.5012	340.118	30.362
16	2	.5005	330.218	29.520
16	3	.4986	369.826	33.186
16	4	.4998	377.241	33.772
16	5	.4989	461.291	41.369
16	6	.5008	463.732	41.430
16	7	.5004	521.256	46.607
16	8	.4987	517.453	46.424
16	9	.4999	550.533	49.274
16	10	.5006	562.852	50.306
16	11	.5015	577.028	51.480
16	12	.5014	564.084	50.336
16	13	.4988	580.710	52.089
16	14	.4995	557.392	49.928
16	15	.5024	561.413	49.997
16	16	.5007	566.866	50.655
16	17	.5015	575.777	51.369
16	18	.5021	577.289	51.442
17	1	.5019	562.852	50.176
17	2	.5028	567.085	50.463
17	3	.5037	552.235	49.053
17	4	.5050	560.950	49.699
17	5	.5159	576.897	50.032
17	6	.5180	576.897	49.829
17	7	.5263	556.107	47.276
17	8	.5259	544.886	46.357
17	9	.5525	378.397	30.643
17	10	.5595	359.979	28.787

Results

ID	Date/Time	10/4/94 5.0ml vial wt + 0.5ml cocktail + 0.5ml 0.02m HNO_3	10/4/94 Sample + 5.0ml vial weight + 0.5ml cocktail + 0.5ml 0.02m HNO_3
E-I-C *pH2.00*a	9/6/94 0915	12.7144	13.2150
" pH2.00*b		12.7794	13.2769
" pH4.00*a		12.7900	13.2875
" pH4.00*b		12.7319	13.2280
" pH5.00*a		12.7377	13.2395
" pH5.00*b		12.7426	13.2388
" pH5.50*a		12.7806	13.2789
" pH5.50*b		12.7220	13.2196
" pH6.00*a		12.7094	13.2073
" pH6.00*b		12.7285	13.2257
" pH6.50*a		12.7317	13.2305
" pH6.50*b		12.7720	13.2678
" pH7.00*a		12.7795	13.2771
" pH7.00*b		12.7597	13.2568
" pH7.50*a		12.7550	13.2524
" pH7.50*b		12.8415	13.3378
" pH8.00*a		12.7441	13.2421
" pH8.00*b		12.8009	13.2965
" pH9.50*a		12.7878	13.3358
" pH9.50*b		12.7093	13.2607

RACK	VIAL	Sample wt	CPMB	u(ppb)
17	13	.5006	554.901	49.595
17	14	.4975	551.416	49.591
17	15	.4975	512.986	46.135
17	16	.4961	505.296	45.571
17	17	.5018	330.971	29.510
17	18	.4962	334.432	30.204
18	1	.4983	477.172	42.845
18	2	.4976	463.522	41.678
18	3	.4979	345.707	31.066
18	4	.4972	334.633	30.113
18	5	.4988	485.181	43.520
18	6	.4958	485.499	43.812
18	7	.4976	536.173	48.210
18	8	.4971	533.401	48.009
18	9	.4974	553.753	49.811
18	10	.4963	552.360	49.796
18	11	.4980	560.569	50.363
18	12	.4956	554.201	50.032
18	13	.5480	402.267	32.843
18	14	.5514	395.622	32.102

Copy of certificate of analysis for $CO_{2(g)}$

Wilson Oxygen

2801 MONTOPOLIS DR.
P.O. BOX 17877
AUSTIN, TEXAS 78760
PHONE 512/389-2323
FAX 512/389-2599

"Quality Today To Insure Tomorrow"

Date Received: September 14, 1994
Date Filled: September 14, 1994

Product: PRIMARY STANDARD MIXTURE
Batch Number: 94-0257-01

Mixture Components	Requested Composition*	Analytical Results*†
Carbon Dioxide	**1.00 %**	**1.00 %**
Air	**Balance**	**Balance**

**Mole Percent*
†±1.0 % Relative to the Concentration of the Minor Component

Cylinders Included In This Batch:

007317LK

Approved By:

Title: Lab Technician

FDP 11/2/94

URANIUM SORPTION EXPERIMENT E-1 (continuation of procedure from page 165)

Reversibility and desorption procedures:

Additional Supplies:

15 ml polypropylene bottles

h) For mixtures listed in Table E-I-2, conduct reversibility experiment:

Adjust the pH of the solutions E-I*pH*i* listed in Table E-I-2 up or down by about 1 pH unit by adding HNO3 or NaHCO3 solutions in the amounts given in the table. **Reweigh** each bottle. Swirl each bottle by hand and place on the gyratory shaker.

After equilibrium is reached (at least 10 days), take a 2 0.5 ml sample from each solution using an Eppendorf pipet, transfer into pre-labeled (e.g., E-IR-pH*i**a or b) and pre-weighed scintillation vials containing 0.5 ml of 0.02 M HNO3. Reweigh each vial. Swirl to mix and save for later analysis of uranium concentration by liquid scintillation counting.

Measure and record the pH and temperature of these solutions. Make sure to rinse the pH electrode very well before transferring into another solution.

Cap all bottles tightly to prevent evaportion.

i) For mixtures **not** listed in Table E-I-2, conduct desorption experiment:

Cap bottles and remove from glovebox.

1) **Reweigh** each bottle to determine evaporative losses. Using an Eppendorf micropipet, carefully retrieve as much of the solid phase as possible (taking care to minimize the amount of solution entrained with the solid). Transfer into pre-weighed and pre-labeled (i.e., E-ID-pH*i*) 15 ml polypropylene bottles. **Reweigh** each FEP and PP bottle.

2) Add 3 ml of 0.1 m HNO3 solution to each of the 15 ml PP bottles; swirl to mix. **Reweigh** each PP bottle.

3) To each of the remaining solutions in the FEP bottles E-I*pH*i*, add 0.5 ml of 50% HNO3 solution; swirl to mix. **Reweigh** each FEP bottle.

4) Allow at least ten days for all the solutions to equilibrate. Then take a 2 **1.0** ml sample from each solution using an Eppendorf pipet, transfer into pre-labeled (e.g., E-ID-pH*i**a or b or E-IF-pH*i**a or b) and pre-weighed scintillation vials (no acid needed). Reweigh each vial. Swirl to mix and save for later analysis of uranium concentration by liquid scintillation counting.

5) Cap all bottles tightly to prevent evaporation.

j) Analyze the U concentration by liquid scintillation counting.

SP 11/2/94

TABLE E-I-2. For reverse experiment: Amount of reagent grade HNO3 or NaHCO3 solutions to add to remaining 0.1 m NaNO3 solution containing 50 ppb U to decrease or increase the pH by 1 unit. The amount of reagent to be added was estimated using EQ4 calculations.

Mixture label	Estimated equil. pH (end of forward exp)	Volume of HNO3 needed (microL)	Molarity of HNO3 solution to use
E-I*pH4.50	4.69	170	0.1
E-I*pH5.00	5.17	60	0.1
E-I*pH5.50	5.42	90	0.2
E-I*pH6.00	5.61	50	0.2
Mixture label	**Equilibrium pH (end of forward exp)**	**Volume of NaHCO3 needed (microL)**	**Molarity of NaHCO3 solution to use**
E-I*pH6.50	6.07	100	0.1
E-I*pH6.75	6.36	180	0.1
E-I*pH7.00	6.60	330	0.1
E-I*pH7.50	7.16	110	1.0

URANIUM SORPTION EXPERIMENT E-1 (continuation of procedure from page 165)

Desorption procedure:

Additional Supplies:

15 ml polypropylene bottles

SP 1100hrs
9/21/94

h) Cap sample bottles and remove from glovebox then conduct desorption:

1300hrs 1) **Reweigh** each bottle to determine evaporative losses. Using an Eppendorf micropipet, carefully retrieve as much of the solid phase as possible (taking care to minimize the amount of solution entrained with the solid). Transfer into pre-weighed and pre-labeled (i.e., E-ID-pH*i*) 15 ml polypropylene bottles. **Reweigh** each FEP and PP bottle.

1530hrs 2) Add 3 ml of 0.1 m HNO3 solution to each of the 15 ml PP bottles; swirl to mix. **Reweigh** each PP bottle.

1600hrs 3) To each of the remaining solutions in the FEP bottles E-I*pH*i*, add 0.5 ml of 50% HNO3 solution; swirl to mix. **Reweigh** each FEP bottle.

4) Allow at least ten days for all the solutions to equilibrate. Then take a 2 1.0 ml sample from each solution using an Eppendorf pipet, transfer into pre-labeled (e.g., E-ID-pH*i**a or b or E-IF-pH*i**a or b) and pre-weighed scintillation vials (no acid needed). Reweigh each vial. Swirl to mix and save for later analysis of uranium concentration by liquid scintillation counting.

5) Cap all bottles tightly to prevent evaporation.

i) Analyze the U concentration by liquid scintillation counting.

E-I Sample Weights

ID	gp 9/21/94 1330 Weight before solid removed (g)	gp 9/21/94 1530 Weight after solid removed (g)	gp 9/24/94 1600h Wt after 0.5ml 50% HNO_3 added (g)	gp 10/4/94 wt before sample taken (g)
E*I*pH2.00	74.19 g	67.11	67.73	67.17
E-I*pH2.25	67.57 g	58.43	59.07	58.50
" *pH2.50	72.80	65.63	66.28	65.76
" pH2.75	70.24	64.08	64.73	64.11
" pH3.00	69.26	64.18	64.82	64.19
" pH3.25	66.49	61.38	62.03	61.46
" pH3.50	71.62	66.53	67.18	66.54
" pH3.75	67.66	61.52	62.17	61.47
" pH4.00	69.36 g	65.27	65.91	65.24
" pH4.25	67.62	62.54	63.18	62.81
" pH4.50	70.99	65.92	66.57	66.14
" pH4.75	73.47	68.39	69.03	68.54
" pH5.00	71.73	67.66	68.31	67.74
" pH5.25	73.15	68.03	68.68	68.01
" pH5.50	68.09	63.02	63.66	63.04
" pH5.75	67.46	63.39	64.03	63.31
" pH6.00	70.49	65.41	66.03	65.59
" pH6.25	71.90	66.80	67.45	66.91
" pH6.50	70.67	64.58	65.20	64.53
" pH6.75	71.31	67.24	67.87	67.23
" pH7.00	71.06	65.96	66.59	65.92
" pH7.25	72.64	66.53	67.17	66.58
" pH7.50	71.36	66.31	66.93	66.34
" pH7.75	70.98	6592	66.55	65.93
" pH8.00	69.32	65.23	65.87	65.31
" pH8.25	71.26	66.10	66.73	65.98
" pH8.50	71.62	67.52	68.16	67.34
" pH8.75	71.35	6542	66.00	65.17
" pH9.00	72.73	68.57	69.20	68.00
" pH9.25	74.19	68.79	69.38	68.69
" pH9.50	84.52	84.53 (solid not removed)		

Sample Weights

ID	9/21/94 1330 gp Weight before Solid added (empty) (g)	9/21/94 1540 gp Wt after Solid added (g)	@	9/21/94 1600 hr gp Wt after 3ml 0.1M HNO_3 added (g)	10/4/94 gp Wt before sample taken (g)
E-ID*pH2.00	7.03	14.10		17.09	16.84
E-ID*pH2.25	7.09	16.22		19.19	18.93
pH2.50	7.05	14.20		17.17	16.94
pH2.75	7.02	13.08		16.06	15.68
pH3.00	7.04	12.10		15.07	14.84
pH3.25	7.14	12.23		15.20	15.05
pH3.50	7.12	12.19		15.16	14.94
pH3.75	7.25	13.37		16.34	16.17
pH4.00	7.16	11.23		14.19	14.00 ~~13.99~~ gp 10/4/94
pH4.25	7.09	12.16		15.12	14.96
pH4.50	7.07	12.13		15.10	14.91
pH4.75	7.17	12.22		15.18	14.71
pH5.00	7.11	11.16		14.12	13.74
pH5.25	7.13	12.22		15.17	14.87
pH5.50	7.12	12.19		15.14	14.91
pH5.75	7.04	11.10		14.07	13.66
pH6.00	7.07	12.14		15.10	14.99
pH6.25	7.07	12.15		15.12	14.76
pH6.50	7.09	13.17		16.13	15.90
pH6.75	7.09	11.15		14.11	13.96
pH7.00	7.17	12.25		15.21	15.02
pH7.25	7.15	13.23		16.20	15.86
pH7.50	7.06	12.11		15.08	14.68
pH7.75	7.14	12.18		15.15	14.93
pH8.00	7.09	11.14		14.12	13.82
pH8.25	7.23	12.32		15.30	14.92
pH8.50	7.11	11.19		14.16	13.95
pH8.75	7.01	12.90		15.85	15.63
pH9.00	7.10	11.26		14.22	13.78
pH9.25	7.04	12.36		15.33	14.91
pH9.50	7.18	7.17 (solid wgt added)			

Results

ID	Date/Time	Vial wt + 5.0ml cocktail	Sample + vial wt + 5.0ml cocktail
E-IF*pH2.00*a	gf 10/4/94 1100hrs	12.1600	13.1738
" *pH2.00*b		12.1448	13.1591
" *pH2.25*a		12.2589	13.2620
" *pH2.25*b		12.1778	13.1810
" *pH2.50*a		12.2225	13.2266
" *pH2.50*b		12.2232	13.2258
" *pH2.75*a		12.2357	13.2339
" *pH2.75*b		12.1973	13.1993
" *pH3.00*a		12.3369	13.3432
" *pH3.00*b		12.2698	13.2681
" *pH3.25*a		12.2063	13.2098
" *pH3.25*b		12.2032	13.2068
" *pH3.50*a		12.2199	13.2200
" *pH3.50*b		12.2352	13.2349
" *pH3.75*a		12.1601	13.1596
" *pH3.75*b		12.1880	13.1888
" *pH4.00*a		12.3066	13.3033
" *pH4.00*b		12.2684	13.2705
" *pH4.25*a		12.2876	13.2880
" *pH4.25*b		12.1942	13.1939
" *pH4.50*a		12.2140	13.2150
" *pH4.50*b		12.2133	13.2144
" *pH4.75*a		12.2170	13.2239
" *pH4.75*b		12.2274	13.2254
" *pH5.00*a		12.2680	13.2725
" *pH5.00*b		12.2756	13.2769
" *pH5.25*a		12.2271	13.2305
" *pH5.25*b		12.2221	13.2233
" *pH5.50*a		12.1894	13.1890
" *pH5.50*b		12.2025	13.2011
" *pH5.75*a		12.2816	13.2789
" *pH5.75*b		12.2448	13.2431
" *pH6.00*a		12.2082	13.2109
" *pH6.00*b		12.2041	13.2062

RACK	VIAL	Sample wt	CPMB	u(ppb)
5	3	1.0138	1120.785	49.464
5	4	1.0143	1131.999	49.934
5	5	1.0031	1120.027	49.957
5	6	1.0032	1121.858	50.034
5	7	1.0041	1099.773	49.005
5	8	1.0026	1076.409	48.036
5	9	.9982	1082.161	48.505
5	10	1.0020	1111.582	49.635
5	11	1.0063	1086.746	48.319
5	12	.9983	1107.767	49.148
5	13	1.0035	1097.537	48.935
5	14	1.0036	1099.518	49.018
5	15	1.0001	1048.804	46.921
5	16	.9997	1071.494	47.955
5	17	.9995	1036.047	46.378
5	18	1.0008	1032.144	46.143
14	1	1.0027	995.896	44.438
14	2	1.0022	971.193	43.358
14	3	1.0004	995.447	44.521
14	4	.9997	981.308	43.919
14	5	1.0010	1030.283	46.051
14	6	1.0011	1047.031	46.795
14	7	1.0069	963.534	42.815
14	8	.9980	989.652	44.368
14	9	1.0045	1016.246	45.265
14	10	1.0013	1044.360	46.675
14	11	1.0034	957.486	42.695
14	12	1.0012	957.054	42.769
14	13	.9996	875.690	39.196
14	14	.9986	877.032	39.295
14	15	.9973	959.782	43.059
14	16	.9983	961.297	43.084
14	17	1.0027	904.331	40.353
14	18	1.0021	911.198	40.683

Results

ID	Date/Time	Vial wt +5.0ml cocktail	Vial wt+Sample +5.0ml cocktail
E-IE*pH6.25*a	gp 10/4/94 1100hr	12.2465	13.2497
" *pH6.25*b		12.2754	13.2714
" *pH6.50*a		12.2500	13.2520
" *pH6.50*b		12.2604	13.2571
" *pH6.75*a		12.2730	13.2743
" *pH6.75*b		12.2195	13.2121
" *pH7.00*a		12.2241	13.2291
" *pH7.00*b		12.2137	13.2113
" *pH7.25*a		12.1870	13.1924
" *pH7.25*b		12.1946	13.1967
" *pH7.50*a		12.2504	13.2553
" *pH7.50*b		12.2394	13.2424
" *pH7.75*a		12.2029	13.2114
" *pH7.75*b		12.2572	13.2625
" *pH8.00*a		12.2033	13.2132
" *pH8.00*b		12.2452	13.2493
" *pH8.25*a		12.2702	13.2751
" *pH8.25*b		12.2749	13.2759
" *pH8.50*a		12.2341	13.2392
" *pH8.50*b		12.2295	13.2300
" *pH8.75*a		12.2101	13.2159
" *pH8.75*b		12.2133	13.2177
" *pH9.00*a		12.2145	13.2323
" *pH9.00*b		12.1963	13.2107
" *pH9.25*a		12.2254	13.2788
" *pH9.25*b		12.2493	13.3028
" *pH9.50*a			
" *pH9.50*b			

RACK	VIAL	Sample wt	cpmB	w(ppb)
15	1	1.0032	848.136	37.826
15	2	.9960	822.398	36.944
15	3	1.0020	889.968	39.740
15	4	.9967	866.326	38.890
15	5	1.0013	980.645	43.819
15	6	.9926	965.422	43.517
15	7	1.0050	1070.950	47.678
15	8	.9976	1060.910	47.582
15	9	1.0054	1094.079	48.688
15	10	1.0021	1134.647	50.660
15	11	1.0049	1119.522	49.845
15	12	1.0030	1127.506	50.296
15	13	1.0085	1133.368	50.282
15	14	1.0053	1129.365	50.264
15	15	1.0099	1157.369	51.275
15	16	1.0041	1173.250	52.279
15	17	1.0049	1163.987	51.825
15	18	1.0010	1137.308	50.835
18	1	1.0051	1145.891	51.009
18	2	1.0005	1098.033	49.104
18	3	1.0058	1128.602	50.205
18	4	1.0044	1101.013	49.046
18	5	1.0178	1131.744	49.751
18	6	1.0144	1089.915	48.073
18	7	1.0534	804977	34.191
18	8	1.0535	845557	35.911

Results

ID	Date/Time	Vial wt + 5.0ml cocktail	Vial wt + sample + 5.0ml cocktail
E-ID *pH2.00*a	10/4/94 1100hrs	12.2490	13.2516
" *pH2.00*b		12.2297	13.2301
" *pH2.25*a		12.2242	13.2255
" *pH2.25*b		12.2470	13.2443
" *pH2.50*a		12.2020	13.2041
" *pH2.50*b		12.2139	13.2086
" *pH2.75*a		12.2925	13.2916
" *pH2.75*b		12.3084	13.3016
" *pH3.00*a		12.2797	13.2835
" *pH3.00*b		12.1747	13.1725
" *pH3.25*a		12.2465	13.2441
" *pH3.25*b		12.2555	13.2483
" *pH3.50*a		12.2889	13.2871
" *pH3.50*b		12.3054	13.2992
" *pH3.75*a		12.2744	13.2778
" *pH3.75*b		12.2312	13.2266
" *pH4.00*a		12.2641	13.2618
" *pH4.00*b		12.2035	13.1980
" *pH4.25*a		12.2875	13.2838
" *pH4.25*b		12.2853	13.2763
" *pH4.50*a		12.2946	13.2925
" *pH4.50*b		12.2270	13.2206
" *pH4.75*a		12.2583	13.2498
" *pH4.75*b		12.2151	13.1945
" *pH5.00*a		12.1982	13.1956
" *pH5.00*b		12.2723	13.2613
" *pH5.25*a		12.2273	13.2239
" *pH5.25*b		12.1448	13.1332
" *pH5.50*a		12.2267	13.2239
" *pH5.50*b		12.2407	13.2248
" *pH5.75*a		12.1945	13.1979
" *pH5.75*b		12.2688	13.2619
" *pH6.00*a		12.2939	13.2878
" *pH6.00*b		12.2154	13.2011

RACK	VIAL	Sample wt	CPMB	U(ppb)
18	11	1.0026	791.616	35.327
18	12	1.0004	807.926	36.134
18	13	1.0013	849.694	37.941
18	14	.9973	847.847	38.037
18	15	1.0021	850.154	37.958
18	16	.9947	841.008	37.829
18	17	.9991	877.587	39.300
18	18	.9932	861.581	38.813
13	1	1.0038	866.854	38.638
13	2	.9978	868.028	38.923
13	3	.9976	907.656	40.708
13	4	.9928	949.258	42.780
13	5	.9982	987.239	44.251
13	6	.9938	1004.732	45.234
13	7	1.0034	1081.223	48.212
13	8	.9954	1041.630	46.820
13	9	.9977	1426.055	63.952
13	10	.9945	1427.020	64.201
13	11	.9963	1448.261	65.039
13	12	.9910	1433.569	64.723
13	13	.9979	1334.032	59.813
13	14	.9936	1308.889	58.940
13	15	.9915	1616.431	72.943
13	16	.9794	1579.357	72.150
13	17	9974	1412.400	63.358
13	18	.9890	1387.420	62.767
8	1	.9966	1718.031	77.131
8	2	.9884	1643.832	74.417
8	3	.9972	2105.800	94.482
8	4	.9841	2097.739	95.374
8	5	1.0034	1938.716	86.448
8	6	.9931	1910.529	86.075
8	7	.9939	2048.869	92.233
8	8	.9857	2039.914	92.594

Result

ID	Date/Time	Vial wt + 5.0ml cocktail	Vial wt + sample + 5.0ml cocktail
E-ID*pH6.25*a	10/4/94 1100hr	12.2478	13.2463
" *pH6.25*b		12.2160	13.2069
" *pH6.50*a		12.2703	13.2697
" *pH6.50*b		12.2457	13.2417
" *pH6.75*a		12.2428	13.2107
" *pH6.75*b		12.2494	13.2177
" *pH7.00*a		12.2463	13.2407
" *pH7.00*b		12.2112	13.1985
" *pH7.25*a		12.2542	13.2469
" *pH7.25*b		12.2132	13.1967
" *pH7.50*a		12.2173	13.2102
" *pH7.50*b		12.2015	13.1654
" *pH7.75*a		12.1810	13.1715
" *pH7.75*b		12.2751	13.2506
" *pH8.00*a		12.2448	13.2026
" *pH8.00*b		12.2405	13.2242
" *pH8.25*a		12.2639	13.2647
" *pH8.25*b		12.2342	13.2284
" *pH8.50*a		12.1842	13.1758
" *pH8.50*b		12.2564	13.2461
" *pH8.75*a		12.2216	13.2189
" *pH8.75*b		12.1934	13.1909
" *pH9.00*a		12.2561	13.2576
" *pH9.00*b		12.2330	13.2227
" *pH9.25*a		12.1868	13.1702
" *pH9.25*b		12.2888	13.3055
" *pH9.50*a			
" *pH9.50*b			

RACK	VIAL	Sample wt	CPMB	u(ppb)
8	9	.9985	2208.676	98.969
8	10	.9909	2190.884	98.925
8	11	.9994	1820.975	81.523
8	12	.9960	1821.795	81.838
8	13	.9679	1615.340	74.671
8	14	.9683	1649.955	76.239
8	15	.9944	995.896	44.809
8	16	.9873	1014.644	45.981
8	17	.9927	843.843	38.033
8	18	.9835	824.862	37.525
20	1	.9929	747.385	33.679
20	2	.9639	742.768	34.478
20	3	.9905	755.669	34.134
20	4	.9755	705.567	32.361
20	5	.9578	636.795	29.747
20	6	.9837	640.500	29.132
20	7	1.0008	733.219	32.780
20	8	.9942	740.942	33.345
20	9	.9916	670.886	30.271
20	10	.9897	654.635	29.594
20	11	.9973	774.068	34.727
20	12	.9975	761.855	34.172
20	13	1.0015	689.630	30.809
20	14	.9897	673.964	30.468
20	15	.9834	706.364	32.138
20	16	1.0167	713.059	31.380

Pages 1 through 209 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Folk
SWRI-QA
11/02/94

12/08/94 RDF

1400 hrs

A. Prepared 1000 ppm ^{238}U stock solution by dissolving 2.1095 g $UO_2(NO_3)_2 \cdot 6H_2O$ (Mallinckrodt Lot # 8640-KCAP) in 1000 g deionized H_2O Polypropylene bottle used.

B. Prepared 1000 mL of mixture containing 5 ppm ^{238}U and 500 ppb ^{233}U by taring 5.00 g of 1000 ppm ^{238}U stock solution prepared above and 100 g of 500 ppb ^{233}U stock solution (Spike # 23A) and making up the total weight to 1000 g with deionized H_2O. Teflon bottle used.

12/09/94 RDF

0830 hrs - Prepared 0.025 m ^{238}U solution by dissolving 12.553 g of $UO_2(NO_3)_2 \cdot 6H_2O$ reagent (Mallinckrodt Lot # 8640-KCAP) in 1000 g of deI H_2O. Polypropylene bottle used.

1010 hrs - Measure pH of uranium solutions. Three-point calibration of pH electrode (pH 2, 4, 7).

Solution	pH Measurement 1	2	3
1000 ppm ^{238}U	3.40 ↑	3.58 ↓	3.46
0.025 m ^{238}U	3.12 ↓	3.10 ↓	3.00
5 ppm ^{238}U / 500 ppb ^{233}U	4.04 ↑	4.20	4.19

↑ or ↓ indicate that although the reading has stabilized somewhat as determined by the pH meter (ready light turns on), some increase or decrease in pH is still possible.

For comparison only, EQ3 calculations of pH give:
1000 ppm — pH 3.43
0.025 m — pH 2.90
5 ppm ^{238}U + 500 ppb ^{233}U — pH 4.25

12/13/94 TD

Procedure for Preparation of Uranyl-Loaded Clinoptilolite
for EXAFS Analysis

Written by: R.T. Pabalan — Date: 12/12/94
Revision No.: 0 — Revision Date:

Objective:

Uranyl-loaded clinoptilolite will be studied using EXAFS to determine if there is a structural difference between uranium that is sorbed on clinoptilolite by an ion-exchange mechanism and uranium that is sorbed by surface-adsorption.

This procedure is designed to produce clinoptilolite powder with ion-exchanged uranium and clinoptilolite powder with surface-adsorbed uranium.

Equipment/materials:

Gyratory shaker
pH meter and pH combination electrode
500-ml polypropylene, polycarbonate or teflon bottles
Purified clinoptilolite (CDV*100/200*UC*WA*HL*CPT), ground and sieved to <400 mesh size (<37 microns)
Eppendorf micropipet
{5ppm ^{238}U + 50 ppb ^{233}U} solution
0.025 m $^{238}UO_2(NO_3)_2$ solution
0.1 m $NaHCO_3$ solution
Packard liquid scintillation analyzer

Procedure:

A. Surface-adsorbed uranium

A-1. Transfer 500 ml of the {5ppm ^{238}U + 50 ppb ^{233}U} solution into a 500-ml polycarbonate (or teflon) bottle. Measure the pH of the solution. Then take two 0.5-ml samples and analyze ^{233}U concentration with LSA.

A-2. Add 1.0 g of clinoptilolite powder to the solution. Equilibrate for one week. Then take 2 0.5-ml samples for LSA analysis and measure the pH of the remaining solution.

^{238}U TD 12/17/94

HOLD POINT: Wait for LSA results. Check if U concentration is 2.5 ppm or less. If yes then proceed.

A-3. Adjust the pH of the solution to about 4.9 by adding 290 microliters of a 0.1 m NaHCO3 solution. (Note: EQ3 calculation indicates that a 2.5 ppm U solution will be saturated with uranyl solid phase if pH is adjusted to 5.0). Equilibrate for one week. Then take 2 0.5-ml samples for LSA analysis and measure the pH of the remaining solution.

HOLD POINT: Wait for LSA results. Calculate %U lost from solution and U concentration (ppm) on the clinoptilolite.

If the pH is outside the range 6.0 to 6.5, adjust the pH by addition of $NaHCO_3$ or HNO3 solution (Use EQ3 to calculate the required amount). Let equilibrate for another week and then take 2 0.5-ml samples for LSA analysis and measure the pH of the remaining solution.

HOLD POINT: Wait for LSA results. Calculate %U lost from solution and U concentration (ppm) on the clinoptilolite.

A-4. If U concentration on the solid is sufficient (>2000 ppm U), separate the solid phase from the solution by filtration or ultracentrifugation.

B. Ion-exchanged uranium

B-1. Transfer 500 ml of the 0.025 m $^{238}UO_2(NO_3)_2$ solution into a 500-ml teflon or polypropylene bottle. Measure the pH of the solution. Then take two 0.5-ml samples and analyze ^{238}U concentration with LSA.

B-2. Add 1.44 g of clinoptilolite powder to the solution. Equilibrate for one week. Then take 2 0.5-ml samples for LSA analysis and measure the pH of the remaining solution.

HOLD POINT: Wait for LSA results. Calculate %U lost from solution and U concentration (ppm) on the clinoptilolite.

B-3. If U concentration on the solid is sufficient (>5000 ppm U), separate the solid phase from the solution by filtration or ultracentrifugation.

The loading experiments were begun today (12/13/94).

For the surface adsorption

500 g of the 50 ppb ^{233}U + 5 ppm ^{238}U were tared into a 1000 mL teflon bottle. Soln prepared on 12/8/94 (GC-09-210)

WT. BOTTLE EMPTY: 190.40 g
WT. SOL'N ADDED: 500.00 g

The pH / T(°C) of the solution was then measured.

pH / T(°C) = 4.15 / 19.8

Two 0.5 mL samples were then taken and prepared for LSA

	1	2
WT. VIAL (g)	7.3769	7.3990
WT. VIAL + SAMPLE (g)	7.8630	7.8793
WT. SAMPLE (g)	0.4861	0.4803

1.00 g clinoptilolite ~~were~~ was then added, the bottle loosely capped and placed on a gyratory shaker. TD 12/13/94

Wt. ~~Eps~~ TD 12/13/94 CLINOP. ADDED : 1.00g
FINAL BOTTLE WT: 691.4g

ION EXCHANGE

500 g 0.025 M $^{238}UO_2(NO_3)_2$ solution was tared into a 500mL PP bottle.

WT. BOTTLE EMPTY (g): 54.54
WT. SOLUTION ADDED(g): 500.01

The pH /T(°C) was then measured.

pH/T(°C) : 2.80 / 19.8

Two 0.5 mL samples were taken for LSA

	1	2
WT. VIAL (g)	7.3238	7.3269
WT. VIAL + SAMPLE (g)	7.3704	7.8125
WT. SAMPLE (g)	0.4866	0.4856

1.44g clinoptilolite were added, the bottle capped and placed on a gyratory shaker.

WT. CLINOP. ADDED: 1.44g
FINAL WT : 555.99 g

12/14/94 TD VERIFICATION OF ^{238}U CALCULATIONS

The LSA calculations for the ^{238}U solutions will be verified and explained here. Verification of ^{233}U can be found on pages 143-144 of GC-11 (CNWRA controlled copy 081).

The first step is to correct the CPM B column for the mass of the sample. This is done to get the activity per gram of sample using the following equation.

$$A = \frac{\text{CPM B}}{\text{wt. sample (g)}}$$

This result can be found in the column "MASS CONV." The next step is to convert the counts per gram to Atoms per gram.

$$N = \frac{A}{\lambda}$$

$$\lambda = \text{decay constant} = \frac{\text{TD 12/14/94}\ \ln 2}{\text{½ life (min)}} = \frac{\ln 2}{\text{½ life (min)}} = 2.9569 \times 10^{-16}$$

$$\tfrac{1}{2}\text{ life} = 4.46 \times 10^9 \text{ y}$$

This result can be found in "ATOM CONV." The number of moles is found by dividing the number of atoms by Avagadro's number.

$$\text{Moles} = \frac{N}{6.023 \times 10^{23}}$$

The result can be found in "MOLE CONV." The number of moles is converted to grams and then to ppm ~~ppb~~ U. TD 12/14/94

$$g\ U = (\text{Moles})\left(238.050784\ \tfrac{g}{mol}\right)$$

$$\text{ppm } U = \frac{g\ U}{1 \times 10^{-6}} \quad \text{TD 12/14/94}$$

TD 12/14/94

This result can be found in the "ppm ~~ppb~~ U" column. 238.050784 is the atomic weight of ^{238}U. A Sample calculation using the following data can be found on the next page.

CPM B = 2000
SAMPLE WT = 0.5 g

$A = {}^{2000}/_{0.5} = 4000$ counts/g

$N = \frac{A}{\lambda} = \frac{4000}{2.9569 \times 10^{-16}} = 1.353 \times 10^{19}$ atoms

MOLES $= \frac{N}{6.02 \times 10^{23}} = 2.246 \times 10^{-5}$ moles

g U = (MOLES)(238.050784) = 5.347×10^{-3} g

ppm U $= \frac{g\,U}{1 \times 10^{-6}} = 5347$ ppm U.

19 Dec 1994 TD EXAFS

The remaining stock solutions (5 ppm ^{234}U + 50 ppb ^{233}U) will be used to start another loading experiment. The amounts of clinoptilolite used previously will be added to each and the experiment will proceed as before. The bottles will be labeled EXAFS A1 and EXAFS IE1.

A1

WT. BEFORE CLINOP. ADDITION (g)		686.6
WT. CLINOP. ADDED (g)		1.00
FINAL WT (g)		687.6
pH / T(°C)	4.15 /	19.7
VIAL:	1	2
WT. VIAL (g)	7.2496	7.3750
WT. VIAL + SAMPLE (g)	7.7556	7.8720
WT. SAMPLE (g)	0.5060	0.4970

IE 1

WT. BEFORE CLINOP. ADDITION (g)	609.9
WT. CLINOP. ADDED (g)	1.44
FINAL WT. (g)	611.3

IE1

pH / T(°C)	2.82 / 19.7	
VIAL:	1	2
WT. VIAL (g)	7.3732	7.2810
WT. VIAL + SAMPLE (g)	7.8791	7.7865
WT. SAMPLE (g)	0.5059	0.5055

The mixtures were then placed on ~~the~~ today a gyratory shaker set to ~120 rpm to equilibrate for 1 week before proceeding.

12/20/94 TD EXAFS LOADING

The liquid scintillation analysis of the A, IE, A1, and IE1 initial samples has completed. The raw data and results of calculations follow. Note that the U ppb (or ppm) value is effected by the presence of daughter isotopes of ^{238}U TD 12/20/94 and some beta decay. For the adsorption experiment, it is assumed that all the activity is from ^{233}U. In the ion exchange experiment it is assumed that all the activity is from ^{238}U. Verification of the ^{233}U calculations is on pages 143-144 of GC-011 (CNWRA controlled copy 081). ^{238}U verification TD 12/20/94 is on p. 215-216 of this notebook.

15 Dec 94 08:03 ALPHA/BETA - 1.02 Page #1
Protocol #:17 U-233 3% 2 sigma User : Todd Dietrich

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	17.94
Region B:	100 -	350	0	3.0	2.68
Region C:	0.0 -	2000	0	0.1	26.11

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
1	999.99	17.94	1.49	2.675	3.87	26.11	1.24	129.59	B
2	8.35	5.90	57.49	529.540	3.02	537.97	3.06	748.06	
3	8.65	4.26	75.46	511.082	3.02	517.48	3.06	750.09	
4	1.13	2188.26	4.04	3938.918	3.00	6995.13	2.25	428.56	
5	1.12	2270.46	3.98	3970.539	3.00	7069.43	2.25	425.24	

TD 12/20/94 EXAFS A & EXAFS IE INITIAL SAMPLES

20 Dec 94 10:47 ALPHA/BETA - 1.02 Page #1
Protocol #:17 U-233 3% 2 sigma User : Todd Dietrich

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	18.37
Region B:	100 -	350	0	3.0	2.82
Region C:	0.0 -	2000	0	0.1	26.77

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
1	999.99	18.37	1.48	2.819	3.77	26.77	1.22	131.53	B
2	8.39	5.95	57.43	527.098	3.02	537.00	3.05	736.35	
3	8.28	4.46	74.72	534.621	3.01	541.71	3.06	744.50	
4	1.09	2332.09	3.98	4100.851	2.99	7277.82	2.25	420.72	
5	1.10	2235.27	4.05	4071.726	2.99	7139.60	2.26	426.67	

EXAFS A1 & EXAFS IE1

LSA RESULTS: Intitial Samples						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*IU1	2	529.540	1089.364	1.2597E+14	2.0915E-10	48.7404
EXAFS A*IU2	3	511.082	1064.089	1.2305E+14	2.0430E-10	47.6096
EXAFS A1*IU1	2	527.098	1041.696	1.2046E+14	2.0000E-10	46.6076
EAXFS A1*IU2	3	534.621	1075.696	1.2439E+14	2.0653E-10	48.1289
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

LSA RESULTS: Intitial Samples						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE*IU1	4	3938.918	8094.776	2.7376E+19	4.5452E-05	10819.9425
EXAFS IE*IU2	5	3970.539	8176.563	2.7652E+19	4.5911E-05	10929.2637
EXAFS IE1*IU1	4	4100.851	8106.051	2.7414E+19	4.5516E-05	10835.0127
EAXFS IE1*IU2	5	4071.726	8054.849	2.7241E+19	4.5228E-05	10766.5733
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

12/20/94 TD EXAFS Loading

The 1st samples of ... A & ... IE, corresponding to steps A2 & B2 of the procedure on pages 212-213 were taken today. The pH of each solution was also measured, along with the weight of each bottle. Samples were prepared for LSA.

EXAFS A

WT. BOTTLE BEFORE SAMPLING / pH (g): 690.9
pH / T(°C) : 5.38 / 20.8

NAME	WT. VIAL (g)	WT. VIAL + SAMPLE (g)	WT. SAMPLE (g)
EXAFS A*1	7.8147	8.3134	0.4987
EXAFS A*2	7.8364	8.3311	0.4947

WT. AFTER SAMPLING / pH (g): 689.9

EXAFS IE

WT. BEFORE SAMPLING / pH (g): 555.96
pH / T(°C) : 3.15 / 20.8

NAME	WT. VIAL (g)	WT. VIAL + SAMPLE (g)	WT. SAMPLE (g)
EXAFS IE*1	7.3231	7.8237	0.5006
EXAFS IE*2	7.3421	7.8421	0.5000

WT. AFTER SAMPLING / pH (g): 554.91

12/27/94 TD EXAFS Loading

The liquid scintillation analysis of the 1st weeks Adsorption and Ion Exchange samples has finished. Raw data and results of calculations follow. ^{233}U calc. verified on page 221 of this note-book ^{238}U calc. verified on page 215-216.

LSA RESULTS: 1 week						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*1	2	287.584	576.667	6.6684E+13	1.1072E-10	25.8013
EXAFS A*2	3	294.180	594.663	6.8766E+13	1.1417E-10	26.6065
Verification		500	1000.000	1.1564E+14	1.9199E-10	44.7421

LSA RESULTS: ~~Intitial Samples~~ TD 12/27/94 1 WK						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE*1	4	3275.127	6542.403	2.2126E+19	3.6736E-05	8744.9517
EXAFS IE*2	5	3318.082	6636.164	2.2443E+19	3.7262E-05	8870.2779
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

Q

23 Dec 94 09:49 ALPHA/BETA - 1.02 Page #1
Protocol #:17 U-233 3% 2 sigma User : Todd Dietrich

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	18.11
Region B:	100 -	350	0	3.0	2.81
Region C:	0.0 -	2000	0	0.1	26.33

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
1	999.99	18.11	1.49	2.814	3.77	26.33	1.23	132.86	B
2	15.31	3.31	71.84	287.584	3.03	292.03	3.13	730.85	
3	14.97	2.20	106.7	294.180	3.03	298.72	3.12	732.13	
4	1.36	301.74	10.17	3275.127	3.00	3686.91	2.83	543.64	
5	1.34	305.03	10.18	3318.082	3.00	3731.13	2.84	549.39	

Neither experiment has reached a point to continue, more samples will be taken.

12/28/94 TD EXAFS loading

The first sampling of ...A1 & ...IE1 was done today. The bottle wt, pH, and T(°C) was also measured. The first set of mixtures, ...A & ...IE, was also resampled and the same data collected. BEFORE and AFTER below refer to before and after pH measurement and sampling.
~~Adsorption~~ TD 12/28/94

NAME	WT. BOTTLE (g) BEFORE	pH | T(°C)	WT. BOTTLE (g) AFTER
EXAFS A	689.3	TD 12/29/94 ~~5.66 | 19.1~~ 5.62 | ~~17.8~~ TD 12/29/94	688.1
A1	687.0	7.15 | 19.1	685.9
IE	554.86	3.28 | 19.1	553.81
IE1	611.2	3.40 | 19.2	610.1

SAMPLE NAME	WT. VIAL (g)	WT. VIAL + SAMPLE (g)	WT. SAMPLE
EXAFS A*3	TD 12/28/94 ~~7.8276~~ 7.8209	8.3224	0.5015
A*4	7.8624	8.3595	0.4971
A1*1	7.8284	8.3243	0.4959
A1*2	7.8525	8.3486	0.4961
IE*3	7.3075	7.8089	0.5014
IE*4	TD 12/28/94 ~~7.3867~~ 7.3881	7.8883	0.5002
IE1*1	TD 12/28/94 ~~7.3550~~ 7.3562	7.8548	0.4986
IE1*2	TD 12/28/94 ~~7.2704~~ 7.2718	7.7695	0.4977

12/29/94 TD EXAFS loading

Due to problems with the pH electrodes, the pH measurements could not be made yesterday. They will be made today and the data entered on the previous ~~TD~~ TD 12/29/94 page.

12/29/94 TD

A revised decay constant for ^{233}U ~~has been~~ TD 12/29/94 is now being used for ^{233}U calculations. The new value is 9.278×10^{-12} and is used in the atom conv column. A new verification of the calculations is given below.

CPMB = 500.000

WT. SAMPLE = 0.5000g

$$\text{MASS CONV} = \frac{\text{CPM B}}{\text{WT.}} = 1000.000$$

$$\text{ATOM CONV} = \frac{\text{MASS CONV}}{8.278 \times 10^{-12}} = \text{TD } 12/29/94 \ \sim\!\!1.280\sim\!\!\ 1.2080 \times 10^{14}$$

↖ NEW DECAY CONSTANT

$$\text{MOLE CONV} = \frac{\text{ATOM CONV}}{6.023 \times 10^{23}} = 2.0057 \times 10^{-10}$$

$$\text{ppb } ^{233}U = \frac{(\text{MOLE CONV})(\text{M.W. } ^{233}U)}{1 \times 10^{-9}} = 46.7403$$

For the LSA Samples of EXAFS loading Adsorption experiment, [U] have been recalculated.

RECALCULATED [U]

LSA RESULTS: Intitial Samples						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*IU1	2	529.540	1089.364	1.3160E+14	2.1849E-10	50.9172
EXAFS A*IU2	3	511.082	1064.089	1.2854E+14	2.1342E-10	49.7358
EXAFS A1*IU1	2	527.098	1041.696	1.2584E+14	2.0893E-10	48.6892
EAXFS A1*IU2	3	534.621	1075.696	1.2995E+14	2.1575E-10	50.2784
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

LSA RESULTS: 1 week						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*1	2	287.584	576.667	6.9663E+13	1.1566E-10	26.9536
EXAFS A*2	3	294.180	594.663	7.1837E+13	1.1927E-10	27.7947
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

All samples from now on to 12/29/94 will be calculated with the new decay Constant.

1/3/95 RD EXAFS LOADING

The LSA of the samples taken on 12/28/94 has finished. Raw data and results of calculations follow. [^{233}U] calculations. [^{238}U] are verified on pg. 221 of the notebook. [^{238}U] calculations are verified on pages 215-216 of this notebook.

31 Dec 94 08:52 ALPHA/BETA - 1.02 Page #1
Protocol #:17 U-233 3% 2 sigma User : Todd Dietrich

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	18.12
Region B:	100 -	350	0	3.0	2.76
Region C:	0.0 -	2000	0	0.1	26.29

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
1	999.99	18.12	1.49	2.763	3.80	26.29	1.23	130.42	B
2	15.44	1.05	213.2	285.126	3.03	286.72	3.14	729.52	
3	15.42	1.60	142.7	285.629	3.03	288.04	3.14	731.35	
4	54.28	0.27	445.9	79.109	3.11	80.15	3.52	731.52	
5	56.25	1.60	76.02	76.241	3.11	78.65	3.50	720.45	
6	1.34	304.27	10.20	3338.282	2.99	3749.08	2.83	540.13	
7	1.37	300.13	10.16	3249.062	3.00	3651.81	2.84	541.82	
8	1.33	256.32	11.21	3344.605	3.00	3687.99	2.87	549.44	
9	1.39	276.13	10.54	3227.453	2.99	3591.69	2.84	546.61	

LSA RESULTS: 2 weeks						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*3	2	285.126	568.546	6.8682E+13	1.1403E-10	26.5740
EXAFS A*4	3	285.629	574.591	6.9412E+13	1.1524E-10	26.8565
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A1*1	4	79.109	159.526	1.9271E+13	3.1996E-11	7.4563
EXAFS A1*2	5	76.241	153.681	1.8565E+13	3.0823E-11	7.1831
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

LSA RESULTS: 2 weeks						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE*3	6	3338.282	6657.922	2.2517E+19	3.7384E-05	8899.3606
EXAFS IE*4	7	3249.062	6495.526	2.1967E+19	3.6472E-05	8682.2928
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

LSA RESULTS: 1 week						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE1*1	8	3344.605	6707.992	2.2686E+19	3.7665E-05	8966.2878
EXAFS IE1*2	9	3227.453	6484.736	2.1931E+19	3.6412E-05	8667.8702
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

1/3/95 EXAFS LOADING

Samples were ~~will~~ to 1/9/95 taken from each of the adsorption and ion exchange mixtures. The solution pH and weight of each bottle before and after sampling was also measured. The ion exchange mixtures were filtered and the solid rinsed with DI H_2O and dried. The solutions were saved for further analysis.

NAME	WT. BOTTLE(g), BEFORE	WT. BOTTLE(g), AFTER	pH /T(°C)
EXAFS A	687.8	686.7	5.56 /16.2
A1	685.5	684.5	7.21 /16.2
IE	553.81	552.73	3.22 /16.3
IE1	610.1	609.1	3.40 /16.3

NAME	WT. VIAL(g)	WT. VIAL + SAMPLE (g)	WT. SAMPLE(g)
EXAFS A*5	7.7982	8.3004	0.5022
A*6	7.8197	8.3193	0.4996
A1*3	7.8792	8.3775	0.4983
A1*4	7.8069	8.3094	0.5025
IE*5	~~7.8272843~~ to 1/9/95 7.7882	7.7882	0.5039
IE*6	7.3336	7.8371	0.5035
IE1*3	7.3696	7.8706	0.5010
IE2*4	7.3166	7.8171	0.5005

1/4/95 EXAFS LOADING

The clinoptilolite used in this experiment was prepared in two ways. ~~The~~ TD 1/4/95 In ...A and ...E, it was ground by hand in a mortar and pestle before sieving. In ...A1 and ...E1, it was milled in a ball mill with TD 1/4/95 using a tungsten carbide vial and balls. Although both were sieved to less than 400 mesh, the milled clinoptilolite is probably a smaller size than the clinoptilolite that was ground by hand. This would account for differences in pH and sorption in the adsorption portion of the experiment.

1/6/95 TD EXAFS LOADING.

The LS analysis of the samples taken on 1/3/95 has finished. Raw data and results of calculations follow. ^{233}U calculations are verified on pg 143-144 of GC-1C (CNWRA controlled copy 081) and page 221 of this notebook. ^{234}U calculations are verified on page 215-216 of this notebook.

05 Jan 95 05:38 ALPHA/BETA - 1.02 Page #1
Protocol #:17 U-233 3% 2 sigma User : Todd Dietrich

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	18.10
Region B:	100 -	350	0	3.0	2.81
Region C:	0.0 -	2000	0	0.1	26.29

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
1	999.99	18.10	1.49	2.806	3.78	26.29	1.23	131.75	B
2	14.81	0.87	261.9	297.329	3.03	299.30	3.14	732.34	
3	15.04	2.11	110.6	292.872	3.03	296.12	3.13	729.14	
4	54.43	0.91	132.9	78.840	3.11	80.99	3.49	730.59	
5	54.28	0.85	141.9	79.066	3.11	80.79	3.50	727.79	
6	1.35	244.12	11.42	3289.787	3.00	3624.82	2.87	548.83	
7	1.37	236.64	11.53	3245.369	3.00	3572.98	2.87	554.37	
8	1.30	549.59	7.60	3437.194	2.99	4187.56	2.72	523.59	
9	1.31	513.19	7.85	3402.538	3.00	4106.54	2.74	528.20	

LSA RESULTS: 3 weeks						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A*5	2	297.329	592.053	7.1521E+13	1.1875E-10	27.6727
EXAFS A*6	3	292.872	586.213	7.0816E+13	1.1758E-10	27.3998
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

LSA RESULTS: 2 weeks						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS A1*3	4	78.840	158.218	1.9113E+13	3.1733E-11	7.3952
EXAFS A1*4	5	79.066	157.345	1.9008E+13	3.1558E-11	7.3544
Verification		500	1000.000	1.2080E+14	2.0057E-10	46.7403

LSA RESULTS: 3 weeks						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE*5	6	3289.787	6528.651	2.2079E+19	3.6658E-05	8726.5692
EXAFS IE*6	7	3245.369	6445.619	2.1799E+19	3.6192E-05	8615.5841
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

LSA RESULTS: 2 week						
SOLN. NAME	S#	CPM B	MASS CONV	ATOM CONV	MOLE CONV	ppb U(233)
EXAFS IE1*3	8	3437.194	6860.667	2.3202E+19	3.8523E-05	9170.3611
EXAFS IE1*4	9	3402.538	6798.278	2.2991E+19	3.8172E-05	9086.9684
Verification		2000	4000.000	1.3528E+19	2.2460E-05	5346.6297

6 Jan 1995 TD EXAFS Loading

The solutions used for loading in the Ion Exchange portion were filtered and the solution and solid saved separately. The solid will be used for EXAFS and the solution analyzed by polarography (Square Wave Voltammetry).

U standards were prepared for calibration for SWV. Standard from 10 to 100 ppm were prepared by diluting various amounts of 1000 ppm U standard (prepared on 12/8/94 p.210) to 50 mL. 0.41 mL of concentrated HCl were added ~~to~~ TD 1/6/95 before dilution to have a final HCl concentration of 0.1 M.

Std	Amt 1000 ppm
10 ppm	0.5
20	1.0
TD 1/6/95 ~~30~~ 40	2.0
TD 1/6/95 ~~40~~ 60	3.0
TD 1/6/95 ~~50~~ 80	4.0
TD 1/6/95 ~~60~~ 100	5.0
TD 1/6/95 ~~70~~	
TD 1/6/95 ~~80~~	

1/6/95 TD EXAFS loading

The standards prepared earlier were used to calibrate the ~~polarography~~ TD 1/6/95 polarograph. The settings on the polarograph are given below.

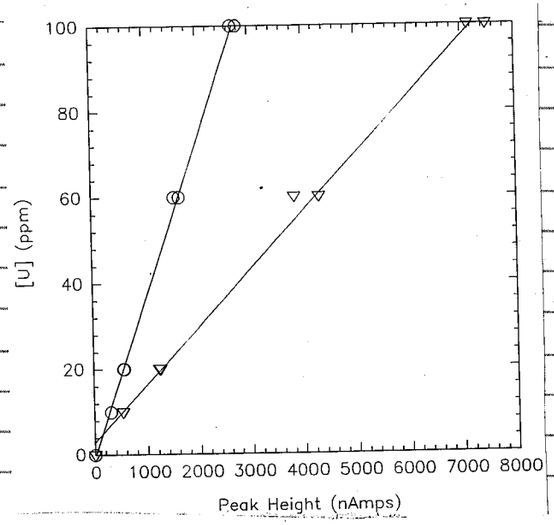
SWV
INITIAL E 0.000 V
FINAL E -1.120 V
PURGE 480 SECONDS
CALCULATED DROP TIME 15.6 SECONDS
SCAN INCREMENT 2 MV
CONDITION 0 SECONDS
CONDITION 0.000 V
EQUILIBRATE 10 SECONDS
DEPOSITION 0 SECONDS
CYCLES 5
FREQUENCY 100
PULSE HEIGHT 0.020 V
REPLICATIONS 1

STANDARD CURVE
BLANK SUBTRACTION: YES
TANGENT FIT: YES
PEAK LOCATION: YES
DERIVATIVE: NO
FORCE LINEAR FIT: NO

A blank of 0.1M HCl was run first, followed by each of the standards. Each standard was run twice and a calibration curve generated.

1695SWV.SP5: Mon, 9-Jan-95

U conc ppm	pk2 ht namps	pk 2 ht namps	Pk1 regress	Pk2 regress
0	0	0	-0.60458	2.9009
10	308	536	0.037129	0.013453
10	309	540	0	0
20	554	1229	0	0
20	572	1249	0	0
60	1544	3831	0	0
60	1641	4300	0	0
100	2671	7140	0	0
100	2782	7480	0	0
			0	0
			0	0
			0.99908	0.99778



The regression of the points for the first peak was a better fit than the 2nd peak, so it was used to calculate the unknown concentrations. The equation of the line is

$$y = 0.037129\,x - 0.60458$$

The unknowns were prepared in the following manner. 0.5 mL of the unknown and 0.41 mL conc. HCl were diluted to 50mL using DI H_2O. This made a dilution factor of 1:100 and a matrix of 0.1 M HCl. These were then run on the polarographs and the concentrations calculated. Each sample was run twice.

NAME	PEAK HT (nAmps)	[U] (ppm), corrected
IE	1614	5932
IE	1623	5965
IE1	1556	5717
IE1	1609	5914

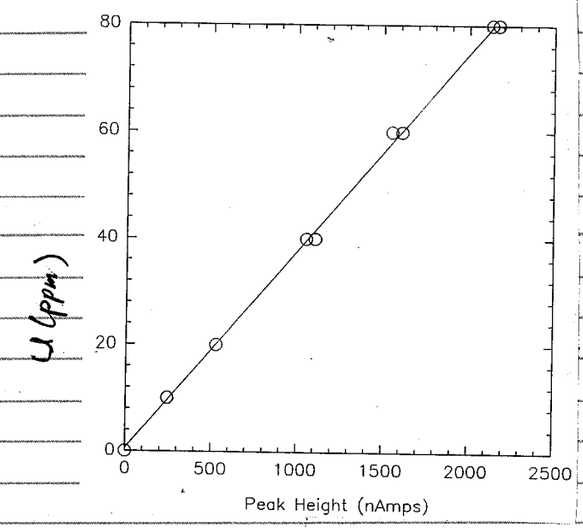
All plots for these runs can be found in a binder entitled "EXAFS Polarography."

1/10/95 TD EXAFS loading

The samples of IE and IE1 were rerun on the polarograph using SWV and the same settings as before (see page 226). The polarograph was calibrated using The 10, 20, 40, 60, and 80 ppm standards prepared previously (see page 225). A calibration curve was generated, and the unknowns run.

U ppm	pk ht (nAmps)	
0	0	0.59975
10	242.3	0.037047
10	240.5	0
20	527	0
20	527	0
40	1053	0
40	1105	0
60	1550	0
60	1608	0
80	2133	0
80	2173	0
		0
		0.99947

SWV Calibration Curve for 1/10/95



NAME	Pk HT (nAmps)	[U] (ppm), corrected
IE	1600	5986
	1660	6210
IE1	1533	5739
	1567	5866

11 Jan 1995 TD

Prepared 250 mL 1.0 M tartaric acid / 1.0 M triethanol amine by dissolving 37.285 g tartaric acid and 37.5225 g triethanol amine in DI H_2O in a volumetric flask.

Wt. tartaric used: 37.2860 g
wt. triethanol amine used: 37.5223 g

Prepared ~~20~~ 250 mL 1.05 M tartaric acid / 1.05 M triethanol amine by dissolving 39.1493 g tartaric acid and 39.3986 g triethanol amine in DI H_2O in a volumetric flask. TD 1/11/95

Wt. tartaric acid used: 39.1502 g
Wt. triethanol amine used: 39.3979 g

A series of U standards were prepared by serial dilution using the 1000 ppm Standard prepared earlier (see pg. 210).
TD 1/11/95 ~~A~~ 50 ppm, 40 ppm, 30 ppm, 20 ppm and 10 ppm standards were prepared by diluting 5, 4, 3, 2, 1 mL of the 1000 ppm standard to 100 mL in a volumetric flask. TD 1/11/95 5, 4, 3, 2, and 1 ppm standards in ~~0.0~~ 0.05 M tartaric acid/triethanol amine (TT) were prepared by diluting 10 mL of the 50, 40, 30, 20 and 10 ppm standard and 5 mL of the 1.0 M TT to 100 mL in a volumetric flask. 500 ppb, 400 ppb and 300 ppb U standards were prepared by diluting 10 mL of the 5, 4, 3 ppm standards and 4.5 mL TT to 100 mL in a volumetric flask. These standards will be used for polarographic analysis of the adsorption portion of the EXAFS loading experiment.

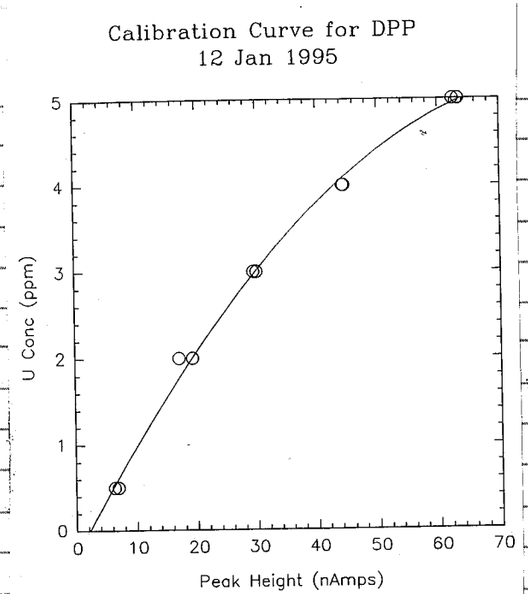
12 Jan 1995 TD EXAFS Loading

10 mL samples were taken from each Adsorption mixture. 0.5 mL of the 1.05 M TT were added to each to get an electrolyte (TT) concentration of 0.5 M. These samples will be analyzed on the polarograph using DPP.

The polarograph was calibrated using the 5, 4, 3, 2, and 0.5 ppm U standards (prepared 11 Jan 95). Parameters used are:

INITIAL E: 0.000 V
TD 1/16/95 FINAL E: ~~0.0~~ -0.500 V
PURGE : 360 s
DROP TIME : 0.5 s
SCAN INCREMENT: 4 MV
PULSE HEIGHT : 0.0200 V
BLANK SUBTRACT: NO

A calibration curve was generated and the samples run. Results can be found on the next page.



11295DPP.SP5: Thu, 12-Jan-95

U ppm	pk ht nAmps	
5	63.3	-0.29023
5	62.4	0.13455 · x
4	44.5	-0.00081009 · x²
4	44.3	0
3	29.44	0
3	29.97	0
2	17.04	0
2	19.29	0
0.5	6.34	0
0.5	6.99	0
		0
		0.99823

NAME	PEAK HT (nAmps)	[U] (ppm), corrected
.. A	25.65	3.32
	25.45	3.29
... A1	8.60	0.910
	9.26	1.00
.. A2	22.64	2.89
	18.80	2.35
... A3	14.78	~~1.78~~ 1.78
	18.92	2.37

1/13/95 TD EXAFS loading.

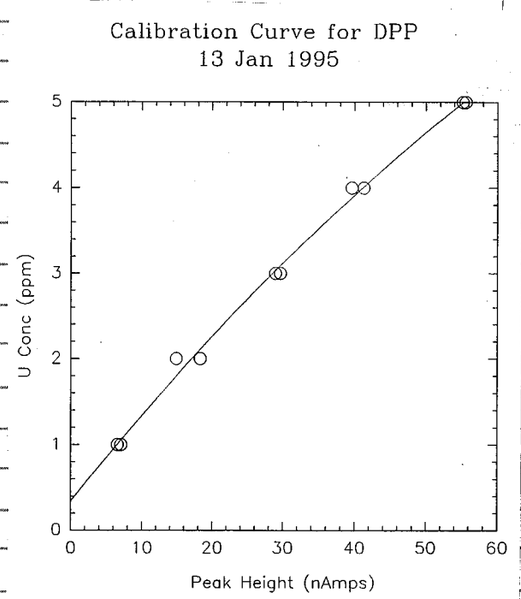
The solutions ...A2 and ...A3 were resampled. Dr. Pabalan had adjusted the pH downward to ~6.5, so these

TD 1/13/95 ~~samples~~ solutions needed to be reanalyzed.

A calibration curve for the polarograph was generated using the 1, 2, 3, 4 and 5 ppm U standards prepared previously (p. 229). The parameters were the same as yesterday (p. 229).

11395DPP.SP5: Fri, 13-Jan-95

U ppm	pk ht nAmps	
1	7.07	0.34433
1	6.56	0.10239
2	14.9	-0.00032826
2	18.3	0
3	28.96	0
3	29.66	0
4	39.58	0
4	41.3	0
5	55.2	0
5	55.6	0
		0
		0.998



NAME	PK HT (nAmps)	[U] (ppm), corrected
... A2x2	18.83	2.39
	19.02	2.40
...A3x2	17.20	2.21
	18.68	2.37

17 Jan 1995 TD EXAFS Loading

Filtered all the solutions and saved both the solid and solution. 0.1g of each solid was separated and saved. The rest was sent to be analyzed with EXAFS.

10 ml of 0.1M HCl was added to each of the 0.1g portions taken from the ~~0.1~~ TD 1/17/95 IE experiments. These were shaken and allowed to sit to desorb the U.

0.5 mL 1.05 M TT and 10 mL H_2O were added to each of the Adsorption solids (0.1g) that were saved. These were shaken and allowed to sit to desorb the U.

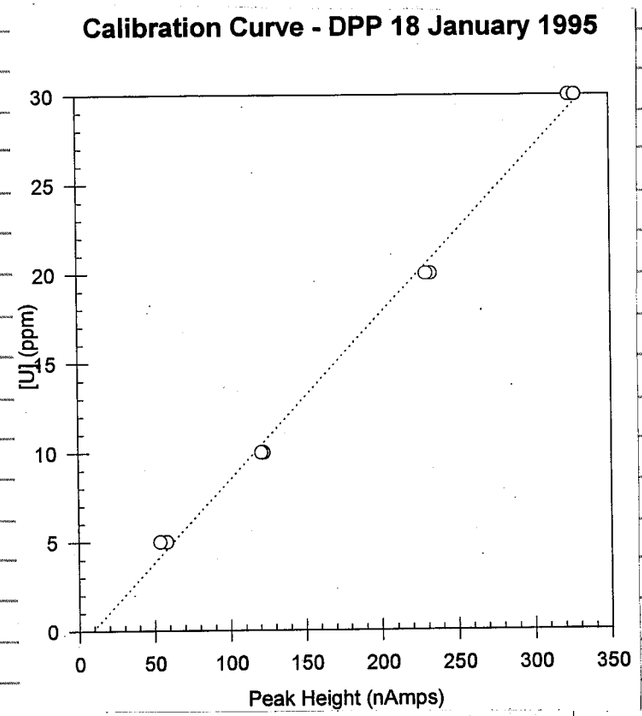
18 Jan 1995 EXAFS Loading TD

Prepared 4 standards of 30, 20, 10, and 5 ppm according to the chart below. All were diluted to 100mL using DI H_2O in volumetric flasks. Two sets were made. One, used for the adsorption samples, had a background electrolyte concentration of 0.05 M TT. The 1 second, used for the Ion exchange samples, was prepared in 0.01 M HCl and 0.05 M TT. 1000 ppm prepared on 12/6/94 (p. 210) TD 1/17/95 NAME

Conc (ppm)	Amt. 1000 ppm added (mL)
30	3
20	2
10	1
5	0.5

5 mL 1.0 M TT was added to each before dilution to 100 mL. The IE standards also had 10 mL 0.1 M HCl added before dilution.

The standards prepared previously were used to calibrate the polarograph for analysis of the Adsorption samples.



	-1- U (ppm)	-2- Ht (nAmp	-3-	-4-
1	5.0000	58.2000	b[0]	-0.8114
2	5.0000	53.8000	b[1]	0.0929
3	10.0000	122.4000	r ²	0.9960
4	10.0000	121.1000		
5	20.0000	232.7000		
6	20.0000	229.7000		
7	30.0000	323.7000		
8	30.0000	327.5000		
9				
10				

Settings for the polarograph are the same as those on 1/12/95 (p. 229).

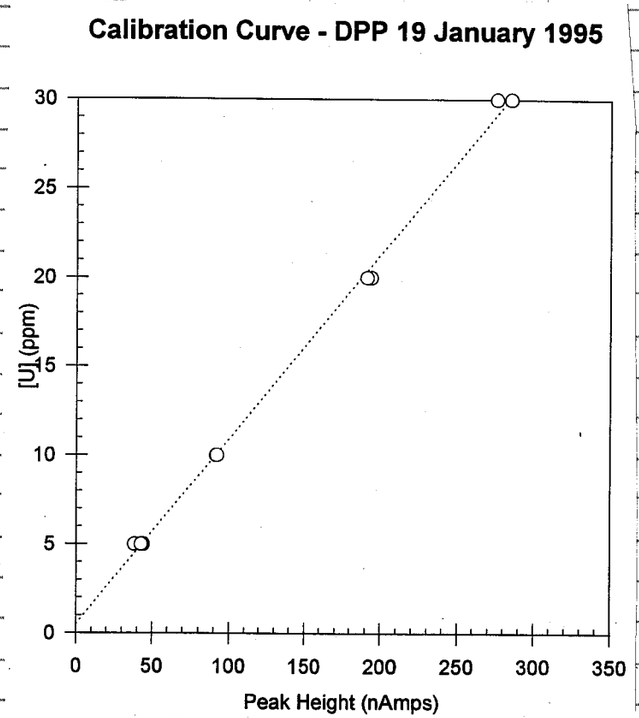
The solution was separated from the solid using a syringe filter. The solution was drawn into the syringe, and then expelled until all the solid was back into the vial (until the solution was clear). The ~~remaining~~ TD 1/18/95 remaining solution was placed in the polarographic cell and analyzed. The results are given below. Two runs of each were done.

NAME	Pk HT (nAmps)	[U] (ppm) uncorrected
A	82.3, 80.3	6.83, 6.65
A1	116.5, 115.0	10.01, 9.87
A2	(386.9, 396.0)	(35.13, 35.98) ← outside calibration.
A3	323.0, 320.9	29.20, 29.00

19 Jan 1995 TD EXAFS loading

The standards ~~containing~~ to 1/19/95 containing 0.01 M HCl were used to calibrate the polarograph. The settings for the polarograph were the same as those on 1/12/95, p. 229.

	-1- U (ppm)	-2- Ht (nAmp	-3-	-4-
1	5.0000	38.4000	b[0]	0.5130
2	5.0000	44.0000	b[1]	0.1039
3	5.0000	42.8000	r ²	0.9981
4	10.0000	91.7000		
5	10.0000	92.2000		
6	20.0000	194.1000		
7	20.0000	191.3000		
8	30.0000	285.1000		
9	30.0000	275.8000		
10				
11				
12				
13				



The samples were prepared in the following manner. ~5mL of solution were removed with a syringe and filter. The solution was expelled until clear to remove any solid and transferred to a small beaker. 1mL was removed and diluted to 10mL in a volumetric flask. 0.5mL ~~pipet~~ 1.0M TT was also added prior to dilution. The samples were then analyzed.

NAME	[U](ppm), uncorrected	
..EE	4.15	> outside calibration.
	4.14	
EE1	13.36	
	13.01	

12/10/96
This project was terminated due to lack of funding.