

PROCEDURE:

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

- Initial $\Sigma U = 500$ 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. zeolite to use = 0.200 ± 0.001
- Initial $[\text{Na}^+] = 0.1$ M $\text{NaNO}_3 + [\text{NaHCO}_3]$ added
- $p\text{CO}_2 = \text{atmospheric} = 10^{-3.48}$ bar

a) Into each of 29 125-ml PP bottle labeled B-II* $p\text{Hi}$ [where i is the approximate initial pH of the solution (see below)], tare 100 g of the 500 ppb uranium solution.

Into each of 5 125-ml PP bottle labeled B-II-C* $p\text{Hi}$ *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 500 ppb uranium solution. These are control solutions to determine uranium loss to the container walls as a function of pH. In contrast to experiments B-I and B-III, only one bottle per pH value will be used to reduce the amount of 500 ppb U solution needed.

Take two 5-ml samples from the 500 ppb stock solution (for this purpose called B-II*IU) with an Eppendorf pipet, transfer into pre-labeled [e.g., B-II-IU*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.

c) 1. For each solution B-II* $p\text{Hi}$ and B-II-C* $p\text{Hi}$, where $i \leq 3.2$:

Measure and record the initial pH (~ 3.2 , 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 dropper HNO_3 solution, the concentration and approximate amount of which is given in Table B-II-1. Swirl the solutions by hand. Record the number of drops 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.

[Calibrate droppers used for each HNO_3 concentration (ml/drop)].

2. For each solution B-II* $p\text{Hi}$ and B-II-C* $p\text{Hi}$, where $i > 3.2$:

Measure and record the initial pH (~ 3.2 , based on EQ3 calculation). Tare onto weighing paper reagent grade NaHCO_3 in the amounts listed in Table B-II-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 $\text{CO}_2(\text{g})$. This equilibration process may take at least ten days.

d) Tare 0.200 ± 0.001 gm of Na-clinoptilolite onto weighing paper, and carefully transfer into each of the B-II* $p\text{Hi}$ (not the B-II-C* $p\text{Hi}$) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker.

e) After equilibrium is reached (at least 10 days), take 2 5-ml samples from each bottle B-II* $p\text{Hi}$ and B-II-C* $p\text{Hi}$ with an Eppendorf pipet, transfer into pre-labeled [e.g., B-II- $p\text{Hi}$ *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-II* $p\text{Hi}$ and B-II-C* $p\text{Hi}$. Make sure to rinse the pH electrode very well before transferring into another solution.

f) Analyze the U concentration by alpha-spectrometry.

Hold Point. Check quality of experimental data.

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.

Procedure for reversibility and reproducibility experiments will be written later.

PREPARATION:

1. Preclean:

- 1 125-ml PP bottle (to contain B-II*IU)
- 34 125-ml PP bottles (to contain experimental mixtures and control solutions)
- 70 50-ml centrifuge tubes (to contain samples for U analysis)
- 2 100-ml volumetric pipet (1 to prepare 500 ppb U solution and 1 to pipet 500 ppb U solution into PP bottles)
- 1 5-ml volumetric pipet (to pipet 0.02 M HNO_3 into 50-ml centrifuge tubes)
- 1 4000-ml plastic bottle (for preparation of 500 ppb U solution)
- 1 glass droppers (for adjusting pH by addition of HNO_3)

2. Prepare:

- 500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike
- 4 L 0.1 M NaNO_3 stock solution
- 1000 ml stock solution of 1.0 M HNO_3
- 1000 ml stock solution of 0.02 M HNO_3

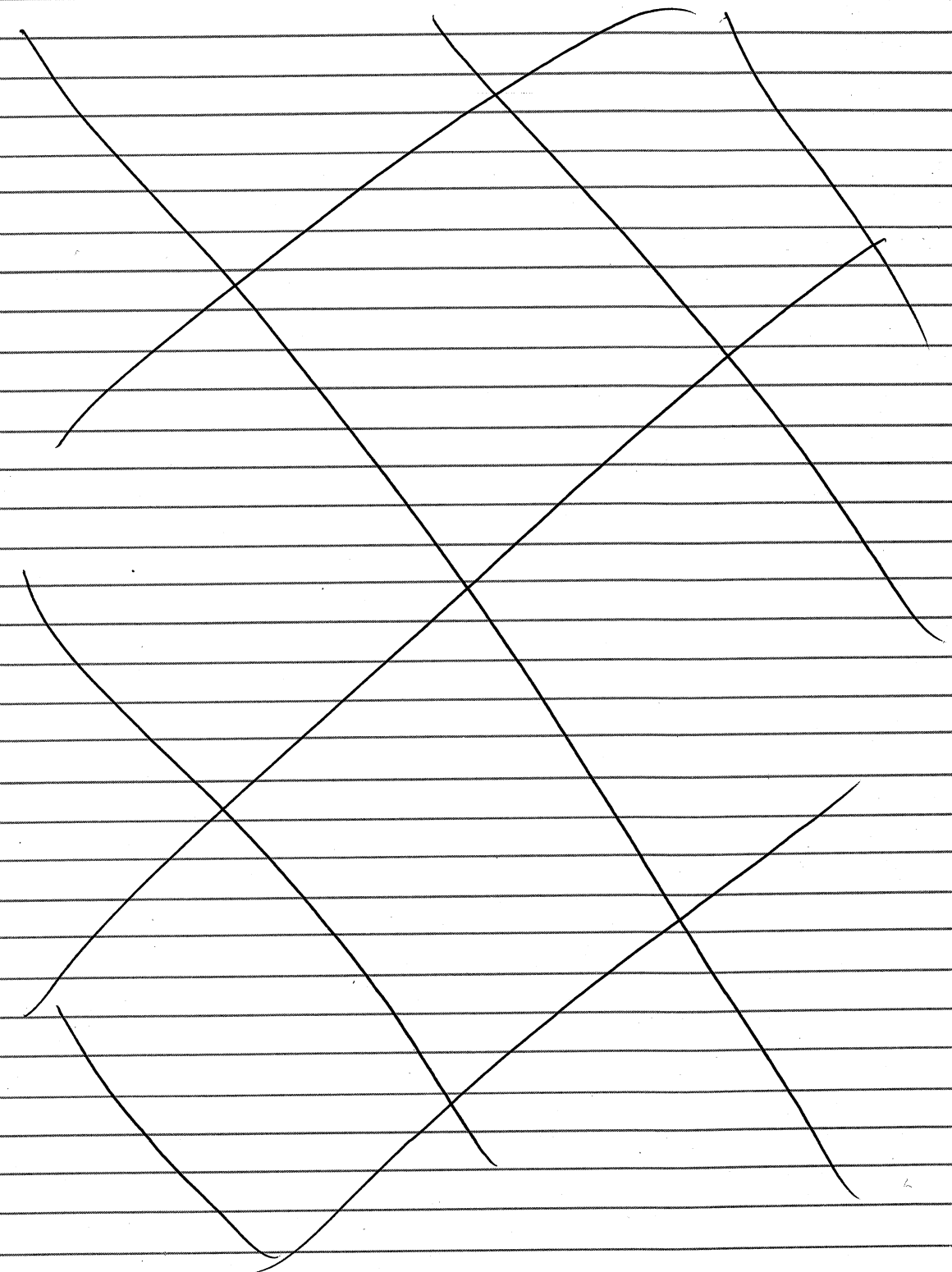
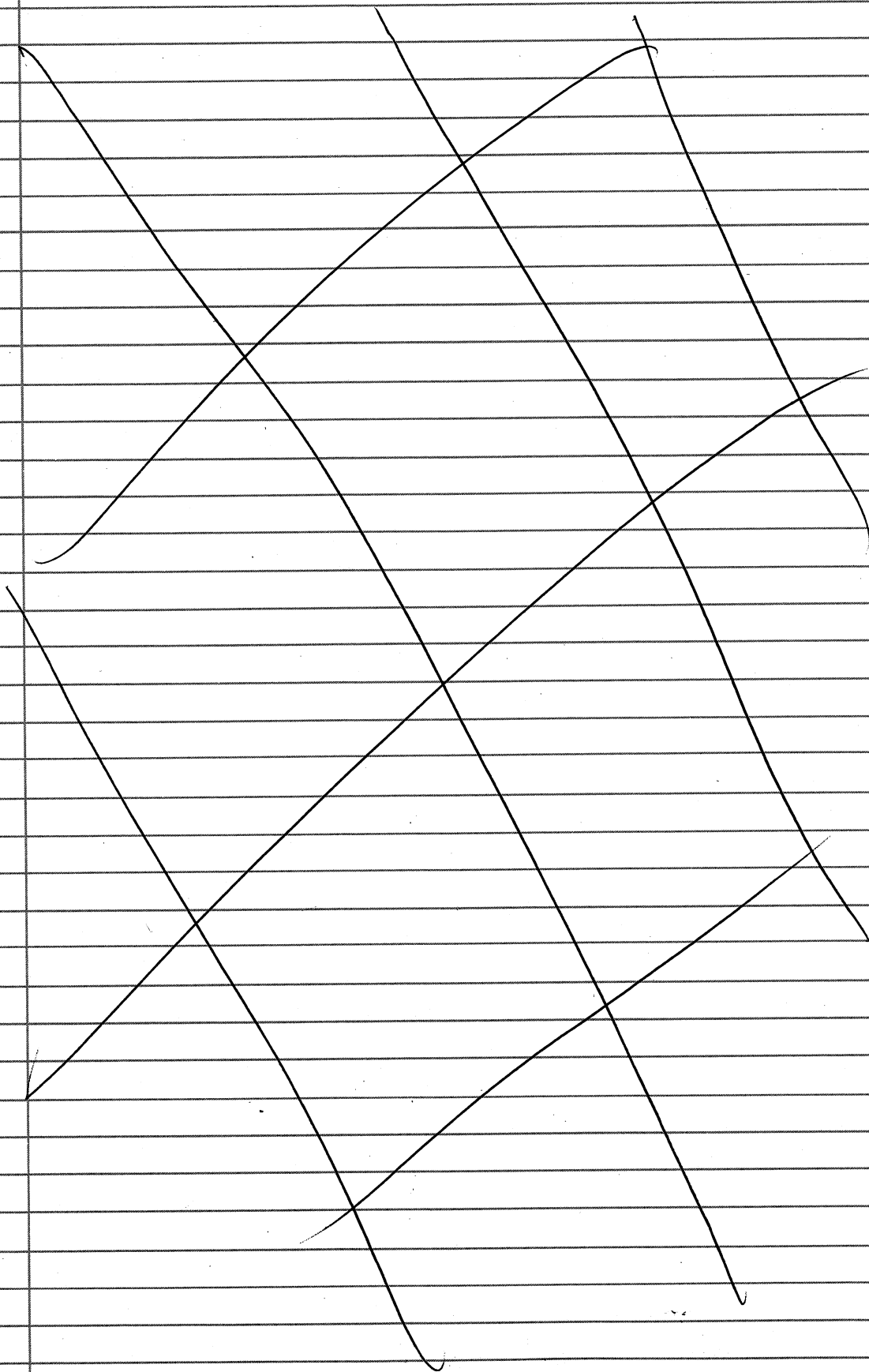


Table B-II-1. Amount of reagent grade HNO_3 or NaHCO_3 to add to 100 ml 0.1 m NaNO_3 solution containing 500 ppb U to result in pH values given in column-1. The amount of reagent to be added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atmospheric $\text{CO}_2(\text{g})$.

Solution pH	Volume of HNO_3 Needed, Drops (0.0394 mL/drop)	Molarity of HNO_3 Used
2.00	28 (1.121 mL)	1.0
2.25	17 (0.680 mL)	1.0
2.50	10 (0.382 mL)	1.0
2.75	5 (0.215 mL)	1.0
3.00	3 (0.121 mL)	1.0
Solution pH	Amount of NaHCO_3 Needed, Grams	
3.25	0.000993	
3.50	0.003485	
3.75	0.004886	
4.00	0.005674	
4.25	0.006118	
4.50	0.006369	
4.75	0.006512	
5.00	0.006598	
5.25	0.006653	
5.50	0.006693	
5.75	0.006728	
6.00	0.006767	
6.25	0.006820	
6.50	0.006907	
6.75	0.007057	
7.00	0.007322	
7.25	0.007792	
7.50	0.008633	
7.75	0.010146	

Solution pH	Amount of NaHCO_3 Needed, Grams	
8.00	0.012875	
8.25	0.017812	
8.50	0.026952	
8.75	0.044428	
9.00	0.079533	
[9.25]	[0.155709]	
[9.50]	[0.342147]	

9/18/92 MJD
1330 hrs-
EXPT. B-II

Initiated B-II expts. Tared 100 ± 0.05
of 500 ppb U into 125 ml bottles
labeled B-II* pH_i , where $i = 2.0$
to 9.0 (at $\Delta \text{pH} = 0.25$) and
B-II-C* pH_j , where $j = 2, 4, 6, 8$
and 9.5.

Took 2 5-ml samples from the
500 ppb stock soln; transferred
into centrif. tubes labeled
B-II* $\text{IU}\#a$ and B-II* $\text{IU}\#b$.
Wt empty Wt w/ 5 ml

Sample B-II* $\text{IU}\#a$ 13.1574 18.1886
" B-II* $\text{IU}\#b$ 13.1478 18.1922

Measured wt. of bottle containing
500 ppb stock soln = 915.3 gms.
Since initial wt was 625.4 gms,
 Δwt was 289.9 gms of
273 U stock soln left.

19 Sept 1992 TD
Experiment B-I

B-I-C (1 and 2)

Adjusted the pH of the remaining solutions according to
Table B-I-1 on page 160. The NaHCO_3 lot # is
897186 H. The Acid was prepared on 5 Aug 1992
(pg 149)

<u>Solution</u>	<u>Adjustment needed</u>	<u>Adjustment Made</u>
B-I *pH 2.00	31 drops 1.0M HNO ₃	31 drops 1.0M HNO ₃
2.25	17 drops	17 drops
2.50	10 drops	10 drops
2.75	5 drops	5 drops
3.00	31 drops 0.1M HNO ₃	31 drops 0.1M HNO ₃
3.25	17 drops	17 drops
3.50	10 drops	10 drops
3.75	5 drops	5 drops
4.00	3 drops	3 drops
4.25	0.00061 g NaHCO ₃	0.00011 g NaHCO ₃
4.50	0.000351 g	0.00034 g
4.75	0.000492 g	0.00051 g
5.00	0.000574 g	0.00057 g
5.25	0.000624 g	TD 0.000624 g 0.00063 g
5.50	0.000658 g	0.00065 g
5.75	0.000687 g	0.00069 g
6.00	0.000721 g	0.00074 g
6.25	0.000772 g	0.00081 g
6.50	0.000858 g	0.00086 g
6.75	0.00101 g	0.00101 g
7.00	0.0013 g	0.0013 g
7.25	0.0017 g	0.0018 g
7.50	0.0026 g	0.0026 g
7.75	0.0041 g	0.0041 g
9/19/92 TD 8.00	—	—
9/19/92 TD 8.25	—	—
9/19/92 TD 8.50	—	—
9/19/92 TD 8.75	—	—
9/19/92 TD 9.00	—	—
B-I-C *pH 2.00 *a	31 drops 1.0M HNO ₃	31 drops
*b	31 drops 1.0M HNO ₃	31 drops
*pH 4.00 *a	3 drops 0.1M HNO ₃	3 drops
*b	3 drops 0.1M HNO ₃	3 drops
*pH 6.00 *a	0.000721 g	0.00073 g
*b	0.000721 g	0.00075 g
9/19/92 TD *pH 8.00 *a	—	—
9/19/92 TD *b	—	—

<u>Solution</u>	<u>Adjustment Needed</u>	<u>Adjustment Made</u>
9/19/92 TD B-I-C *pH 4.50 *a	—	—
9/19/92 TD *b	—	—
B-II-C (1 & 2)		
pH Adjustments were made to the B-II solutions according to Table B-II-1 on pg 170.		
NaHCO ₃ lot # 897186A		
HNO ₃ prepared on 5 Aug 1992 (pg 149)		
<u>SOLUTION</u>	<u>Adjustment Needed</u>	<u>Adjust made</u>
B-II *pH 2.00	28 drops 1.0M HNO ₃	28 drops
2.25	17 drops	17 drops
2.50	10 drops	10 drops
2.75	5 drops	5 drops
3.00	3 drops	3 drops
3.25	0.000993 g NaHCO ₃	0.00092 g
3.50	0.003485 g	0.00351 g
3.75	0.004886 g	0.00495 g
4.00	0.005674 g	0.00568 g
4.25	0.006119 g	0.00608 g
4.50	0.006369 g	0.00634 g
4.75	0.006512 g	0.00649 g
9/19/92 TD 5.00	0.006598 g	0.00656 g
5.25	0.006653 g	0.00665 g
5.50	0.006693 g	0.00669 g
5.75	0.006728 g	0.00672 g
6.00	0.006767 g	0.00678 g
6.25	0.006820 g	0.00685 g
6.50	0.006907 g	0.00698 g
6.75	0.007057 g	0.0071 g
7.00	0.007322 g	0.0073 g
7.25	0.007792 g	0.0079 g
7.50	0.008633 g	0.0086 g
7.75	0.010146 g	0.0104 g
8.00	0.012875 g	0.0124 g
8.25	0.017812 g	0.177 g

Solution	Adjustment Needed	Adjustment Made
B-II * pH 8.50	0.026952 g	0.0271 g
8.75	0.04428 g	0.0441 g
9.00	0.079533 g	0.0795 g
B-II-C * pH 1.0	28 drops 10.4 M HNO ₃	28 drops
* pH 4.0	0.005674 g	0.00564 g
* pH 6.0	0.006767 g	0.00677 g
* pH 8.0	0.012875 g	0.0130 g
* pH 9.5	0.342147 g	0.3426 g

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Remeasured the pH of all K3-1, K3-2, B-I, and B-II solutions.

K3-1	pH/T(°C)
K3-1 * A	5.44/22.0°C TD
* B	6.08/22.0°C TD
* C	5.23/22.0°C TD
K3-2	
K3-2 * A	7.00/22.1°C TD
* B	6.96/22.1°C TD
* C	6.96/22.1°C TD
B-I * phi	
B-I * pH 2.00	2.05/22.9°C TD
2.25	2.25/22.4°C TD
2.50	2.38/22.3
2.75	2.77/22.2
3.00	2.89/22.4
3.25	3.21/22.5
3.50	3.41/22.5
3.75	3.61/22.5
4.00	3.79/22.5
4.25	4.35/22.6
4.50	4.50/22.6
4.75	4.67/22.5
5.00	4.89/22.6
5.25	5.32/22.5

Solution	pH/T(°C)
B-I * pH 5.50	5.44/22.6
5.75	4.99/22.6
6.00	5.21/22.6
6.25	5.48/22.7
6.50	5.17/22.7
6.75	6.13/22.6
7.00	6.63/22.3
7.25	6.80/22.4
7.50	7.18/22.7
7.75	7.37/22.7
8.00	7.79/22.3
8.25	8.01/22.2
8.50	8.34/22.2
8.75	8.35/22.2
9.00	8.74/22.2
B-I-C * pH 2.00 * a	2.04/23.0
* b	1.97/23.0
* pH 4.00 * a	3.81/22.8
* b	3.79/22.8
* pH 6.00 * a	5.12/22.8
* b	6.00/22.7
* pH 8.00 * a	7.78/22.9
* b	7.81/22.9
* pH 9.50 * a	8.87/22.8
* b	8.89/22.8
B-II * phi	
B-II * pH 2.00	2.08/23.0
2.25	2.26/22.9
2.50	2.43/23.0
2.75	2.72/23.2
3.00	2.83/23.3
3.25	3.20/23.3
3.50	3.58/23.3
3.75	3.66/23.2
4.00	3.70/23.2
4.25	4.26/23.3

<u>SOLUTION</u>	<u>pH/T(°C)</u>
B-II * pH 4.50	4.12/23.2
4.75	4.30/23.2
5.00	4.28/23.2
5.25	4.74/23.2
5.50	4.86/23.2
5.75	5.85/23.2
6.00	4.20/23.2
6.25	4.68/23.1
6.50	4.59/23.2
6.75	5.48/23.1
7.00	5.85/23.1
7.25	6.21/23.0
7.50	6.37/23.1
7.75	6.90/23.1
8.00	6.82/23.1
8.25	7.06/22.8
8.50	7.75/23.0
8.75	7.62/23.0
9.00	8.43/23.0
B-IE-C * pH 2.0	2.06/23.3
4.0	3.70/23.3
6.0	4.35/23.3
8.0	6.78/23.4
9.5	8.83/23.4

All solutions were returned to the Gyrotory Shakers set at ~120 rpm. A pH 7.00 standard was checked periodically (about every 20-30 min) to check the drift of the electrode.

21 Sept 1992 TD

B-II

Nitric acid was not added to the B-II * I U a
Samples so 5.0625g was added to a
and 5.1204g to b.

21 Sept 1992

TD

URANIUM SORPTION EXPERIMENT B-III:

Kd vs pH; Equilibrium with atmospheric pCO₂; Initial ΣU=5 ppb

WRITTEN BY: R.T. PABALAN

DATE WRITTEN: August 5, 1992

REVISION NO.: 0

DATE REVISED:

OBJECTIVE:

- To investigate the importance of uranium sorption on the zeolite mineral clinoptilolite as a function of solution pH and total uranium concentration. Experimental data will be correlated with uranium aqueous speciation.
- To investigate reversibility and reproducibility of uranium sorption reactions.

EQUIPMENT:

Gyratory shaker or constant temperature shaker bath
EG&G alpha-spectrometer
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance

SUPPLIES:

- pH buffer (pH = 2,4,7,9,10)
- 1 125-ml PP bottle (to contain B-III*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 0.02 M HNO₃ 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 HNO₃)
 - weighing paper
 - 1 Eppendorf pipet (for taking 5-ml samples)
 - Na⁺-clinoptilolite (CDV*100/200*UC*WA*HL*CPT*Naf)
 - reagent grade NaHCO₃
 - 500 ppb U stock solution prepared from 50 ppm ²³⁵U commercial spike
 - 4 L 0.1 M NaNO₃ stock solution
 - 1000 ml stock solution of 1.0 M HNO₃
 - 1000 ml stock solution of 0.1 M HNO₃
 - 1000 ml stock solution of 0.02 M HNO₃
 - 1000 ml stock solution of 0.01 M HNO₃
 - ultrapure water

PROCEDURE:

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

- Initial $\Sigma U = 5$ 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. zeolite to use = 0.200 ± 0.001
- Initial $[Na^+] = 0.1$ M $NaNO_3 + [NaHCO_3]$ added
- $pCO_2 = \text{atmospheric} = 10^{-3.48}$ bar

a) In a pre-cleaned 4 liter 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 the plastic bottle on a Mettler 4600 balance.

b) Into each of 29 125-ml PP bottle labeled B-III* pHi [where i is the approximate initial pH of the solution (see below)], tare 100 g of the 5 ppb uranium solution.

Into each of 10 125-ml PP bottle labeled B-III-C* pHi * 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 uranium 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-III*IU. Take two 5-ml samples from B-III*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-III-IU*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.

c) 1. For each solution B-III* pHi and B-III-C* pHi , where $i \leq 5.1$:

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 dropper 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 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.

[Calibrate droppers used for each HNO_3 concentration (ml/drop)].

2. For each solution B-III* pHi and B-III-C* pHi , 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 equilibration process may take at least ten days.

d) Tare 0.200 ± 0.001 gm of Na-clinoptilolite onto weighing paper, and carefully transfer into each of the B-III* pHi (not the B-III-C* pHi) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker.

e) After equilibrium is reached (at least 10 days), take 2 5-ml samples from each bottle B-III* pHi and B-III-C* pHi with an Eppendorf pipet, transfer into pre-labeled [e.g., B-III- pHi * 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-III* pHi and B-III-C* pHi . Make sure to rinse the pH electrode very well before transferring into another solution.

f) Analyze the U concentration by alpha-spectrometry.

Hold Point. Check quality of experimental data.

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.

Procedure for reversibility and reproducibility experiments will be written later.

PREPARATION:

1. Preclean:

- 1 125-ml PP bottle (to contain B-III*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 0.02 M 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

2. Prepare:

500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike

- 4 L 0.1 M $NaNO_3$ stock solution
- 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
- 1000 ml stock solution of 0.01 M HNO_3

Table B-III-1. Amount of reagent grade HNO_3 or NaHCO_3 to add to 100 ml 0.1 M NaNO_3 solution containing 5 ppb U to result in pH values given in column-1. The amount of reagent to be added was estimated using EQ3 calculations and assuming the solutions are in equilibrium with atmospheric $\text{CO}_2(\text{g})$.

Solution	Volume HNO_3 Needed, Drops (.0394 mL/drop)	Molarity of HNO_3 Used
2.00	31 (1.21 mL)	1.0
2.25	17 (0.68 mL)	1.0
2.50	10 (0.38 mL)	1.0
2.75	5 (0.21 mL)	1.0
3.00	31 (1.21 mL)	0.1
3.25	17 (0.68 mL)	0.1
3.50	10 (0.38 mL)	0.1
3.75	5 (0.21 mL)	0.1
4.00	3 (0.12 mL)	0.1
4.25	17 (0.68 mL)	0.01
4.50	10 (0.38 mL)	0.01
4.75	5 (0.21 mL)	0.01
5.00	3 (0.11 mL)	0.01
Solution pH	Amount of NaHCO_3 to be Added, Grams	
5.25	0.000021	
5.50	0.000054	
5.75	0.000083	
6.00	0.000117	
6.25	0.000168	
6.50	0.000253	
6.75	0.000402	
7.00	0.000664	
7.25	0.001132	
7.50	0.001967	

Solution pH	Amount of NaHCO_3 to be Added, Grams	
7.75	0.003464	
8.00	0.006163	
8.25	0.011080	
8.50	0.020204	
8.75	0.037654	
9.00	0.072706	
[9.25]	[0.148756]	
[9.50]	[0.334885]	

1200 Prepared 2 1000g solutions of 0.1M NaHCO_3 by dissolving 8.499g NaNO_3 in 1000g H_2O . ^{NO_3 96/92}

Wt used 1 8.4990g
2 8.4995g

1230 Began Experiment B-III by Diluting 40.0g 500ppb ^{^{235}U} solution ^{91/92} to 4000g using 0.1M NaNO_3 to make a 5ppb ^{^{235}U} solution.

1300 B-III-b

Tared 100 \pm 0.05g 5ppb ^{^{235}U} into 29 PP bottles labeled B-III-xpHi (i = 2.0 - 9.0, $\Delta\text{pH} = 0.25$) and also into 10 bottles labeled B-III-CxpHi xalorb (i = 2.0, 4.0, 6.0, 8.0, 9.5). The remaining solution was placed in a bottle labeled B-III-xIU. 2-5ml samples of the IU Soln were taken and weighed.

B-III-xIU x a 4.9743g
x b 5.0262g

~5g of 0.02M HNO_3 was added.

1330 Δ B-III-C

The solution pH of each B-III solution was adjusted according to table B-III-1* (pg 180-181). The solutions were covered with a Kimwipe and placed on a gyratory shaker set to 120 rpm.

<u>Solution</u>	<u>Adjustment made</u>
B-III *pH 2.00	31 drops 1.0M HNO ₃
2.25	17 drops
2.50	10 drops
2.75	5 drops
3.00	31 drops 0.1M HNO ₃
3.25	17 drops
3.50	10 drops
3.75	5 drops
4.00	3 drops
4.25	17 drops 0.001M HNO ₃
4.50	10 drops
4.75	5 drops
5.00	3 drops
5.25	0.00002 g NaHCO ₃
5.50	0.00005 g
5.75	0.00007 g
6.00	0.00013 g
6.25	0.00016 g
6.50	0.00027 g
6.75	0.00044 g
7.00	0.00065 g
7.25	0.00112 g
7.50	0.00205 g
7.75	0.00353 g
8.00	0.00631 g
8.25	0.01100 g
8.50	0.02068 g
8.75	0.03766 g
9.00	0.07301 g

SOLUTIONADJUSTMENT MADE

B-III-C *pH 2.0 *a	31 drops 1.0M HNO ₃
*b	31 drops
*pH 4.0 *a	3 drops 0.1M HNO ₃
*b	3 drops 0.1M HNO ₃
*pH 6.0 *a	0.00014g NaHCO ₃
*b	0.00012g
*pH 8.0 *a	0.00599g
*b	0.00629g
*pH 9.5 *a	0.33531g
*b	0.33535g

1730

Remeasured the pH of the solutions in K3-1 and K3-2

K3-1 g/L

<u>K3-1</u>	<u>pH/T(°C)</u>
K3-1 *A	5.32 / 24.8
*B	5.98 / 24.8
*C	5.20 / 24.8

K3-2

K3-2 *A	6.86 / 24.8
*B	6.75 / 24.9
*C	6.83 / 24.8

Buffer pH 5	5.04 / 24.8
7	7.02 / 24.8

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KINETICS EXPERIMENT K3

Remeasured the pH of the K3-1 & K3-2 solutions

SOLUTION	pH/T(°C)
K3-1 * A	5.37/20.5
* B	6.02/20.5
* C	5.17/20.5
K3-2 * A	6.85/20.5
* B	6.75/20.6
* C	6.82/20.6
pH 7 buffer	7.02/20.6

1500 TD

The pH of the K3-1 & K3-2 solutions were remeasured using the Ross pH electrode rather than the micro electrode.

SOLUTION	pH/T(°C)
K3-1 * A	5.36/21.3
* B	5.87/21.3
* C	5.25/21.3
K3-2 * A	6.67/21.3
* B	6.63/21.3
* C	6.61/21.3
pH 7 buffer	7.00/21.3

900 TD 24 Sept 1992

EXPERIMENT B-III

Measured the pH of the B-III * pH1 & B-III-C * pH1 solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-III * pH 2.00	1.96/20.7	B-III-C * pH 4.0 * A	4.02/20.8
2.25	2.23/20.7	* B	3.93/20.8
2.50	2.48/20.7	* pH 6.0 * A	5.84/20.8
2.75	2.82/20.7	* B	5.84/20.8
3.00	3.00/20.7	* pH 8.0 * A	8.12/20.8
3.25	3.26/20.7	* B	8.60/20.8
3.50	3.40/20.7	* pH 9.5 * A	8.90/20.8
3.75	3.78/20.7	* B	8.68/20.8
4.00	3.99/20.7	pH 2.00 buffer	2.03/20.8
4.25	4.11/20.7	pH 7.00 buffer	7.02/20.8
4.50	4.28/20.7	pH 10.00 buffer	10.02/20.8
4.75	4.56/20.7		
5.00	4.75/20.7		
5.25	5.38/20.7		
5.50	5.53/20.7		
5.75	5.75/20.7		
6.00	6.09/20.7		
6.25	6.26/20.7		
6.50	6.40/20.7		
6.75	6.74/20.7		
7.00	6.65/20.7		
7.25	7.11/20.7		
7.50	7.36/20.7		
7.75	7.60/20.7		
8.00	7.79/20.7		
8.25	8.05/20.7		
8.50	8.33/20.7		
8.75	8.57/20.7		
9.00	8.73/20.6		
B-III-C * pH 2.0 * A	1.95/20.8		
* B	1.92/20.8		

1100 TD EXPERIMENT B.I

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-I-K pH 2.00	1.94/20.8	B-I-C x pH 6.0 x a	5.10/21.2
2.25	2.17/20.8	x b	6.05/21.2
2.50	2.32/20.8	pH 8.0 x a	7.79/21.2
2.75	2.72/20.8	x b	7.61/21.2
3.00	2.81/20.8	pH 9.5 x a	9.09/21.2
3.25	3.14/20.8	x b	9.10/21.2
3.50	3.35/20.8	pH 2 buffer	2.04/20.9
3.75	3.53/20.8	pH 7 buffer	7.02/20.9
4.00	3.72/20.8	pH 10 buffer	10.02/20.9
4.25	4.26/20.8		
4.50	4.41/20.8		
4.75	4.59/20.8		
5.00	4.82/20.8		
5.25	5.30/20.8		
5.50	5.08/20.8		
5.75	4.92/20.8		
6.00	5.16/20.8		
6.25	5.41/20.8		
6.50	5.10/20.8		
6.75	6.19/20.8		
7.00	6.75/20.8		
7.25	6.91/20.8		
7.50	7.40/20.8		
7.75	7.59/20.8		
8.00	7.77/20.8		
8.25	8.07/20.9		
8.50	8.35/20.9		
8.75	8.60/20.9		
9.00	8.85/20.9		
B-I-C x pH 2.0 x a	1.94/21.2		
x b	1.88/21.2		
pH 4.0 x a	3.75/21.2		
x b	3.73/21.2		

TD 9/24/92
625

5.41/20.8

1430 TD EXPERIMENT B.II

Remeasured the pH of all the solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-II-K pH 2.00	2.03/21.2	B-II-C x pH 9.5	9.01/21.3
2.25	2.19/21.2	pH 2 buffer	2.01/21.3
2.50	2.35/21.2	pH 7 buffer	7.01/21.3
2.75	2.65/21.2	pH 10 buffer	10.03/21.3
3.00	3.76/21.3		
3.25	3.13/21.3		
3.50	3.53/21.2		
3.75	3.59/21.2		
4.00	3.67/21.2		
4.25	4.28/21.3		
4.50	4.26/21.3		
4.75	4.26/21.2		
5.00	4.27/21.2		
5.25	4.75/21.2		
5.50	4.85/21.3		
5.75	6.48/21.1		
6.00	4.13/21.3		
6.25	4.68/21.2		
6.50	4.53/21.2		
6.75	5.96/21.2		
7.00	6.56/21.2		
7.25	7.00/21.3		
7.50	7.12/21.2		
7.75	7.44/21.2		
8.00	7.75/21.2		
8.25	8.02/21.2		
8.50	8.36/21.2		
8.75	8.63/21.2		
9.00	8.85/21.2		
B-I-C x pH 2.0	1.99/21.3		
4.0	3.63/21.3		
6.0	4.32/21.2		
8.0	7.56/21.2		

25 SEPT 1992 TD

EXPT. B-I

Remeasured the pH of all solutions.

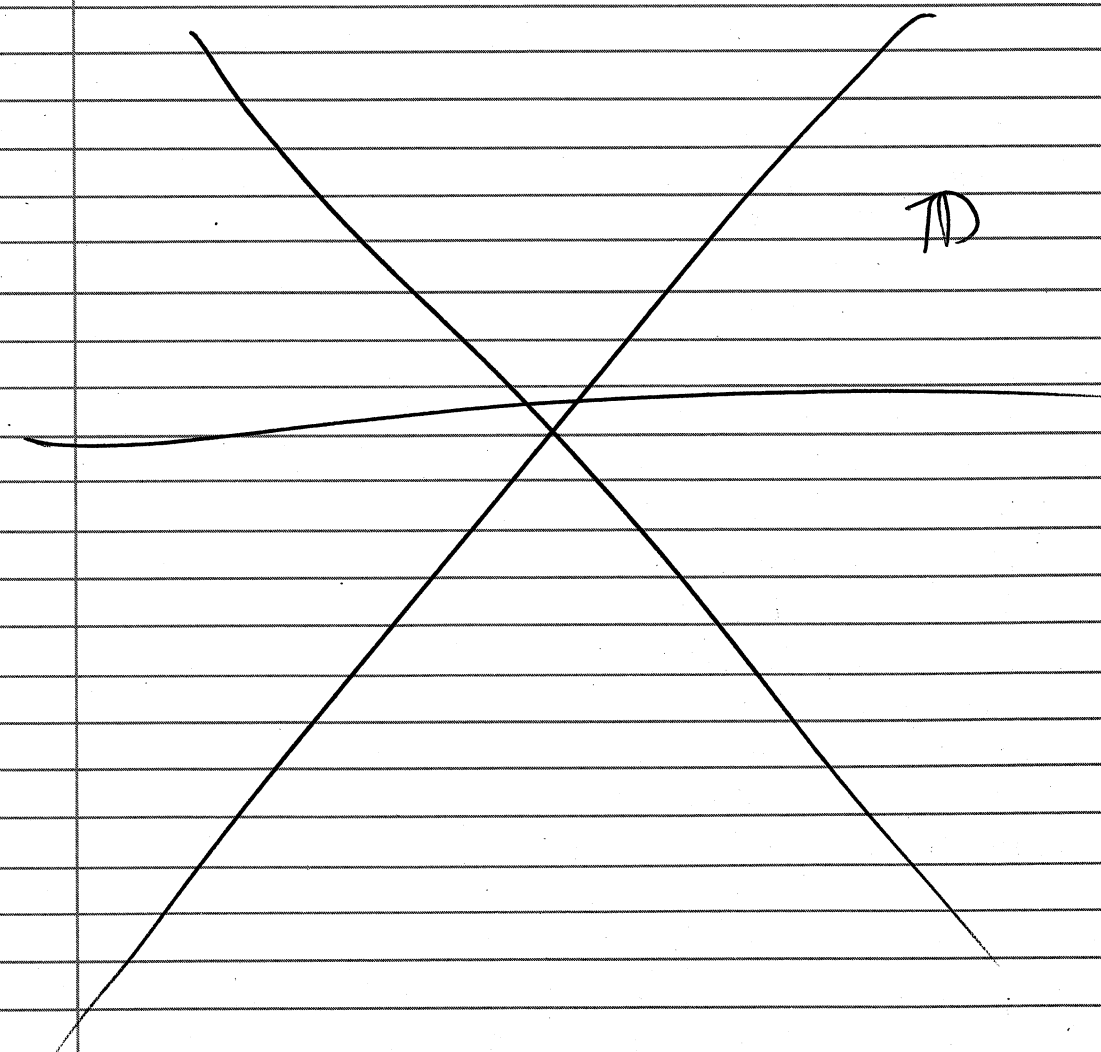
SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-I *pH 2.00	1.92/19.5	B-II-C *pH 6.00 *a	5.05/19.5
2.25	2.13/19.6	*b	6.04/19.5
2.50	2.30/19.5	pH 8.00 *a	7.79/19.4
2.75	2.69/19.6	*b	7.66/19.4
3.00	2.81/19.6	pH 9.50 *a	9.22/19.5
3.25	3.13/19.6	*b	9.23/19.5
3.50	3.34/19.6	pH 7 buffer	7.03/19.7
3.75	3.53/19.6		
4.00	3.71/19.6		
4.25	4.26/19.6		
4.50	4.42/19.6		
4.75	4.58/19.6		
5.00	4.83/19.7		
5.25	5.30/19.7		
5.50	5.08/19.7		
5.75	4.92/19.7		
6.00	5.15/19.7		
6.25	5.46/19.7		
6.50	5.10/19.7		
6.75	6.18/19.8		
7.00	6.74/19.6		
7.25	6.93/19.6		
7.50	7.22/19.5		
7.75	7.49/19.7		
8.00	7.77/19.5		
8.25	8.09/19.5		
8.50	8.38/19.5		
8.75	8.61/19.5		
9.00	8.88/19.5		
B-II-C *pH 7.00 *a	1.91/19.4		
*b	1.85/19.4		
pH 4.00 *a	3.72/19.4		
*b	3.69/19.4		

25 Sept TD Exp. K3

Remeasured the K3-1 & K3-2 solution pHs.

SOLUTION	pH/T(°C)
K3-1 *A	5.33/19.0
*B	6.00/19.0
*C	5.18/19.0
K3-2 *A	6.85/19.0
*B	6.67/19.0
*C	6.71/19.0
pH 7 buffer	7.03/19.1

Since the pH values have stabilized, the experiment will proceed to the next step of zeolite addition and sampling.



25 Sept EXPT BII TD

Remeasured the pH of the solutions in this experiment.

SOLUTION	pH/T(°C)
B-II *pH 2.00	2.00/19.5
2.25	2.16/19.4
2.50	2.33/19.4
2.75	2.62/19.1
3.00	2.72/19.1
3.25	3.09/19.1
3.50	3.47/19.1
3.75	3.54/19.1
4.00	3.62/19.1
4.25	4.25/19.1
4.50	4.23/19.1
4.75	4.23/19.1
5.00	4.32/19.1
5.25	4.72/19.1
5.50	4.81/19.1
5.75	6.68/19.5
6.00	4.25/19.2
6.25	4.66/19.2
6.50	4.58/19.2
6.75	6.15/19.2
7.00	6.57/19.4
7.25	6.96/19.2
7.50	7.14/19.2
7.75	7.57/19.3
8.00	7.83/19.3
8.25	8.04/19.4
8.50	8.36/19.4
8.75	8.63/19.4
9.00	8.87/19.4
B-II-C *pH 2.0	1.95/19.1
4.0	3.61/19.2
6.0	4.29/19.2
8.0	7.53/19.2
9.5	9.14/19.3

26 Sept 1992 TD EXPERIMENT K3

1000 TD

Remeasured the pH of each of the solutions

SOLUTION	pH/T(°C)
K3-1 *A	5.33/19.9
*B	6.04/19.9
*C	5.20/19.9
K3-2 *A	6.91/19.9
*B	6.81/19.9
*C	6.87/19.9

1030

K3-1 (2) - D

2-10 ml aliquots of each of the 6 solutions were taken and placed in a centrifuge tube and weighed. 500 μ L of 1.0M HNO_3 was added.

SAMPLE	WEIGHT(g)
K3-1 *A *I1	10.0174
*I2	10.1028
K3-2 *B *I1	10.2453
*I2	10.1865
*C *I1	9.9670
*I2	10.1283
K3-2 *A *I1	10.2180
*I2	10.1472
*B *I1	10.1917
*I2	10.1209
*C *I1	10.1512
*I2	10.0905

~2.0g Na^+ -clinoptilolite were added next.

SOLUTION	wt Added (Time)	Temp. of room = 67.9°F
K3-1 *A	2.002g (10:35)	
*B	2.001g (10:40)	
*C TD aliquot		
K3-2 *A	2.001g (10:45)	
*B	2.000g (10:50)	

The *C solutions do not get clinoptilolite
The solutions were placed on a gyratory shaker
set at ~120 rpm.

SAMPLES - each sample is weighed and 500 μ l 1.0M HNO₃ is added

K3-1*A*	DATE/TIME	WEIGHT (g)	pH/T(°C)
1	9/26, 1240 TD	10.1292	5.42/20.4
2	9/26, 1440 TD	9.8978	5.39/21.1
3	9/27, 0835 TD	10.1746	5.50/19.7
4	9/28, 0835 TD (a) 10.1539 (b) 10.1265		5.51/19.9
5	9/29, 0707 TD	10.0645	5.55/19.1
6	9/30, 0835 TD (a) 10.0556 (b) 10.0318		5.60/20.5
7	10/2, 0835 TD	10.0766	5.57/20.4
8	10/4, 0835 TD	10.1253	5.61/20.6
9	10/7, 0835 TD (a) 9.8605 (b) 9.8130		5.64/21.1
10	10/10, 1035 TD	10.0267	5.64/20.8
11	10/14, 0935 TD	10.0630	5.62/21.8
12	10/19, 0935 TD (a) 10.0546 (b) 10.0504		5.61/20.8

K3-1*B*	DATE/TIME	WEIGHT (g)	pH/T(°C)
1	9/26, 1242 TD	10.1285	6.04/20.6
2	9/26, 1440 TD	10.1288	6.00/21.1
3	9/27, 0840 TD	10.1774	6.08/19.7
4	9/28, 0840 TD (a) 10.1604 (b) 10.1420		6.06/19.9
5	9/29, 0710 TD	10.0505	6.10/19.1
6	9/30, 0840 TD (a) 10.0230 (b) 10.0391		6.10/20.6
7	10/2, 0840 TD	10.0951	6.10/20.4
8	10/4, 0840 TD	10.1104	6.09/20.6
9	10/7, 0840 TD (a) 10.0352 (b) 9.9675		6.09/21.1
10	10/10, 1040 TD	10.0726	6.14/20.8
11	10/14, 0940 TD	10.0923	6.09/20.9
12	10/19, 0940 TD (a) 10.0398 (b) 10.0306		6.05/20.9

K3-1*C*	DATE/TIME	WEIGHT (g)	pH/T(°C)
1	9/26, 1255 TD	9.5783	5.23/20.6
2	9/26, 1455 TD	10.1086	5.18/20.6
3	9/27, 0855 TD	10.2109	5.23/19.7
4	9/28, 0853 TD (a) 10.0448 (b) 10.0312		5.22/19.9
5	9/29, 0720 TD	10.0651	5.21/19.1
6	9/30, 0854 TD (a) 9.9498 (b) 10.1430		5.23/20.6
7	10/2, 0854 TD	10.0007	5.22/20.5
8	10/4, 0854 TD	10.1032	5.22/20.6
9	10/7, 0854 TD (a) 10.1097 (b) 10.0792		5.22/21.2
10	10/10, 1054 TD	9.9433	5.23/20.9
11	10/14, 0954 TD	10.0049	5.21/21.9
12	10/19, 0954 TD (a) 10.0519 (b) 10.0109		5.21/21.0

K3-2*A*	DATE/TIME	WEIGHT (g)	pH/T(°C)
1	9/26, 1245 TD	10.1038	6.87/20.6
2	9/26, 1445 TD	10.1572	6.83/21.1
3	9/27, 0845 TD	10.1810	6.92/19.7
4	9/28, 0845 TD (a) 10.1408 (b) 10.0989		6.90/19.9
5	9/29, 0714 TD	10.0722	6.93/19.1
6	9/30, 0845 TD (a) 9.9883 (b) 10.0376		6.95/20.6
7	10/2, 0845 TD	10.0294	6.93/20.4
8	10/4, 0845 TD	10.1020	6.90/20.6
9	10/7, 0845 TD (a) 10.0857 (b) 9.8305		6.95/21.2
10	10/10, 1045 TD	9.9254	6.96/20.8
11	10/14, 0945 TD	10.0144	6.94/22.0
12	10/19, 0945 TD (a) 10.0095 (b) 10.0097		6.90/20.9

K3-2XB*	DATE/TIME	WEIGHT(g)	pH/T(°C)
1	TD 9/26; 1250	10.1259	6.75/20.6
2	TD 9/26; 1450	10.1375	6.72/21.1
3	TD 9/27, 0850	10.0897	6.79/19.7
4	TD 9/28, 0850	(a) 10.1705 / (b) 10.1426	6.78/19.9 6.77
5	TD 9/29, 0717	10.0530	6.80/19.1
6	TD 9/30, 0850	(a) 9.9170 (b) 9.9882	6.83/20.6
7	TD 10/2, 0850	10.0320	6.80/20.4
8	TD 10/4, 0850	10.0483	6.81/20.6
9	TD 10/7, 0850	(a) 10.0601 (b) 10.0675	6.80/21.1
10	TD 10/10, 1050	10.0563	6.84/20.9
11	TD 10/14, 0950	9.9841	6.82/22.0
12	TD 10/17, 0950	(a) 10.0174 (b) 10.0432	6.79/21.0

K3-2XC*	DATE/TIME	WEIGHT(g)	pH/T(°C)
1	TD 9/26; 1300	9.9371	6.85/20.6
2	TD 9/26, 1500	10.0675	6.79/21.2
3	TD 9/27, 0900	10.1153	6.87/19.8
4	TD 9/28, 0856	(a) 10.1192 (b) 10.1104	6.83/20.0
5	TD 9/29, 0723	10.0648	6.88/19.1
6	TD 9/30, 0856	(a) 10.0898 (b) 10.1710	6.88/20.6
7	TD 10/2, 0856	9.8983	6.86/20.5
8	TD 10/4, 0856	10.1547	6.86/20.6
9	TD 10/7, 0856	(a) 10.0864 (b) 10.0603	6.85/21.2
10	TD 10/10, 1056	9.9110	6.89/20.9
11	TD 10/14, 0956	10.0223	6.90/22.0
12	TD 10/17, 0956	(a) 9.9990 (b) 10.0180	6.95/21.1

9/28/92 TD reach soln

2 samples should have been taken at 1 on 9/27, but were not. This will be done today.

9/28/92

930 TD EXPERIMENT B-I

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-I-xpH2.00	1.90/20.1	B-I-Cx pH6.0xa	5.08/20.1
2.25	2.13/20.1	x b	6.06/20.1
2.50	2.28/20.1	pH8.0xa	7.78/20.2
2.75	2.70/20.1	x b	7.79/20.2
3.00	2.81/20.1	pH4.5 xa	9.31/20.2
3.25	3.13/20.1	x b	9.31/20.2
3.50	3.33/20.1	pH 2 buffer	1.99/20.2
3.75	3.54/20.1	pH 7 buffer	7.06/20.2
4.00	3.72/20.1	pH 10 buffer	10.08/20.2
4.25	4.28/20.1		
4.50	4.43/20.1		
4.75	4.59/20.1		
5.25	5.32/20.1		
5.00	4.83/20.0		
5.50	5.09/20.1		
5.75	4.92/20.1		
6.00	5.16/20.1		
6.25	5.49/20.0		
6.50	5.11/20.0		
6.75	6.20/20.1		
7.00	6.75/20.1		
7.25	6.91/20.1		
7.50	7.35/20.1		
7.75	7.58/20.1		
8.00	7.79/20.1		
8.25	8.09/20.1		
8.50	8.24/20.1		
8.75	8.65/20.1		
9.00	8.92/20.1		
B-I-Cx pH2.0 xa	1.88/20.2		
x b	1.83/20.2		
pH 4.6 xa	3.73/20.2		
pH 4.0 x b	3.70/20.1		

9/29/92
1245 TO EXPERIMENT B-II

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-II *pH 2.00	2.02 / 20.6	B-II-C *pH 9.5	9.28 / 20.8
2.25	2.19 / 20.6	pH 2 buffer	2.00 / 20.8
2.50	2.35 / 20.6	pH 7 buffer	7.05 / 20.9
2.75	2.64 / 20.6	pH 10 buffer	10.09 / 20.8
3.00	2.74 / 20.6		
3.25	3.11 / 20.8		
3.50	3.48 / 20.8		
3.75	3.55 / 20.8		
4.00	3.64 / 20.8		
4.25	4.29 / 20.8		
4.50	4.24 / 20.8		
4.75	4.24 / 20.8		
5.00	4.39 / 20.8		
5.25	4.73 / 20.7		
5.50	4.82 / 20.6		
5.75	6.46 / 20.6		
6.00	4.30 / 20.7		
6.25	4.69 / 20.7		
6.50	4.59 / 20.7		
6.75	6.20 / 20.7		
7.00	6.59 / 20.7		
7.25	7.00 / 20.6		
7.50	7.13 / 20.7		
7.75	7.56 / 20.7		
8.00	7.85 / 20.7		
8.25	8.01 / 20.6		
8.50	8.38 / 20.6		
8.75	8.65 / 20.6		
9.00	8.91 / 20.7		
B-II-C *pH 2.0	2.96 / 20.8		
4.0	3.61 / 20.8		
6.0	4.29 / 20.8		
8.0	7.56 / 20.9		

1245 TO EXPERIMENT B-III

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-III *pH 2.00	1.90 / 20.9	B-III-C *pH 6.0 *a	5.89 / 20.9
2.25	2.17 / 20.9	*b	5.76 / 20.9
2.50	2.43 / 20.9	pH 8.0 *a	7.85 / 20.9
2.75	2.77 / 20.9	*b	7.75 / 20.9
3.00	2.96 / 20.9	pH 9.5 *a	9.19 / 20.9
3.25	3.22 / 21.0	*b	9.16 / 20.9
3.50	3.36 / 21.0	pH 2 buffer	2.00 / 21.1
3.75	3.75 / 21.0	pH 7 buffer	7.03 / 21.1
4.00	3.97 / 21.0	pH 10 buffer	10.08 / 21.1
4.25	4.09 / 21.0		
4.50	4.26 / 20.9		
4.75	4.52 / 20.9		
5.00	4.72 / 21.0		
5.25	5.49 / 21.0		
5.50	5.41 / 21.0		
5.75	5.59 / 21.0		
6.00	5.95 / 21.0		
6.25	6.06 / 21.0		
6.50	6.30 / 21.0		
6.75	6.67 / 21.0		
7.00	6.55 / 21.0		
7.25	7.01 / 21.0		
7.50	7.29 / 21.0		
7.75	7.49 / 21.1		
8.00	7.72 / 21.1		
8.25	7.95 / 21.1		
8.50	8.27 / 21.1		
8.75	8.55 / 21.1		
9.00	8.85 / 21.1		
B-III-C *pH 2.0 *a	1.89 / 20.9		
*b	1.88 / 20.9		
pH 4.0 *a	3.99 / 20.9		
*b	3.92 / 20.9		

30 Sept 1992 TD EXPERIMENT B-III

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-III-xpH2.00	1.94/20.8	B-III-CxpH4.0xb	3.95/21.1
2.25	2.19/20.8	pH 6.0xa	5.82/21.1
2.50	2.45/20.8	xb	5.82/21.1
2.75	2.79/20.8	pH 8.0xa	7.79/21.1
3.00	2.97/20.8	xb	7.79/21.1
3.25	3.23/20.8	pH 9.5xa	9.26/21.1
3.50	3.38/20.8	xb	9.24/21.1
3.75	3.77/20.8	pH 2 buffer	2.01/21.1
4.00	3.99/20.8	pH 7 buffer	7.05/21.1
4.25	4.11/20.8	pH 10 buffer	10.07/21.1
4.50	4.28/20.9		
4.75	4.55/20.9		
5.00	4.74/20.9		
5.25	5.54/20.9		
5.50	5.49/20.9		
5.75	5.72/20.9		
6.00	5.99/20.9		
6.25	6.06/20.9		
6.50	6.35/20.9		
6.75	6.72/20.9		
7.00	6.99/20.9		
7.25	7.10/21.0		
7.50	7.40/21.0		
7.75	7.60/21.0		
8.00	7.70/21.0		
8.25	8.05/21.0		
8.50	8.28/21.0		
8.75	8.57/21.0		
9.00	8.88/21.0		
B-III-CxpH2.0xa	1.93/21.0		
xb	1.90/21.0		
pH 4.0xa	4.02/21.0		

9/30/1992 TD EXPERIMENT B-II

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-II-xpH2.00	2.00/22.2	B-II-CxpH 8.0	7.57/21.9
2.25	2.17/22.1	9.5	9.29/21.9
2.50	2.35/22.1	pH 2 buffer	1.98/22.2
2.75	2.62/21.8	pH 7 buffer	7.03/22.2
3.00	2.73/21.9	pH 10 buffer	10.05/22.2
3.25	3.10/21.9		
3.50	3.48/21.9		
3.75	3.54/21.8		
4.00	3.62/21.8		
4.25	4.26/21.9		
4.50	4.26/21.8		
4.75	4.21/21.8		
5.00	4.38/21.8		
5.25	4.73/22.1		
5.50	4.81/22.1		
5.75	6.48/22.2		
6.00	4.30/22.0		
6.25	4.67/22.0		
6.50	4.59/22.0		
6.75	6.22/22.0		
7.00	6.58/22.0		
7.25	7.03/22.1		
7.50	7.22/22.1		
7.75	7.50/22.0		
8.00	7.84/22.0		
8.25	8.01/22.0		
8.50	8.36/22.0		
8.75	8.61/22.1		
9.00	8.87/22.0		
B-II-CxpH2.0.	1.97/21.9		
4.0	3.61/21.9		
6.0	4.29/21.9		

EXPERIMENT B-I

REMEASURED THE pH OF ALL SOLUTIONS

SOLUTION	10/20/92 pH/100	SOLUTION	pH/100
B-I * pH 2.00	1.93 / 22.7	B-I-C * pH 4.0 x a	3.74 / 22.3
2.25	2.17 / 22.7	x b	3.72 / 22.3
2.50	2.32 / 22.7	pH 6.0 x a	5.06 / 22.3
2.75	2.72 / 22.7	x b	6.04 / 22.3
3.00	2.83 / 22.7	pH 8.0 x a	7.78 / 22.3
3.25	3.16 / 22.6	x b	7.78 / 22.20
3.50	3.36 / 22.6	pH 9.5 x a	9.30 / 22.4
3.75	3.55 / 22.5	x b	9.29 / 22.4
4.00	3.73 / 22.6	pH 2 buffer	1.98 / 22.7
4.25	4.29 / 22.5	pH 10 buffer	10.04 / 22.7
4.50	4.44 / 22.5		
4.75	4.61 / 22.5		
5.00	4.94 / 22.4		
5.25	5.31 / 22.5		
5.50	5.09 / 22.4		
5.75	4.93 / 22.4		
6.00	5.17 / 22.4		
6.25	5.46 / 22.4		
6.50	5.11 / 22.4		
6.75	6.28 / 22.4		
7.00	6.82 / 22.6		
7.25	6.94 / 22.7		
7.50	7.28 / 22.4		
7.75	7.61 / 22.4		
8.00	7.77 / 22.7		
8.25	8.07 / 22.7		
8.50	8.32 / 22.7		
8.75	8.57 / 22.7		
9.00	8.85 / 22.7		
B-I-C * pH 2.0 x a	1.94 / 22.2		
x b	1.87 / 22.2		

10/2/92 TD EXPERIMENT B-I

Remeasured the pH of all solutions prior to the addition of ~0.200gm of Na-clinoptilbite. The zeolite was not added to the B-I-C * pH 4.0 (a, b) solns.

SOLUTION	pH/100	WEIGHT CDV... NaF ADDED (g)
B-I * pH 2.00	1.92 / 21.0	0.2005
2.25	2.15 / 21.0	0.2006
2.50	2.30 / 21.0	0.2009
2.75	2.70 / 21.0	0.1993
3.00	2.83 / 21.0	0.2003
3.25	3.15 / 21.0	0.2000
3.50	3.36 / 21.0	0.1997
3.75	3.56 / 21.0	0.2004
4.00	3.74 / 21.0	0.2002
4.25	4.30 / 21.1	0.2001
4.50	4.45 / 21.1	0.2004
4.75	4.62 / 21.1	0.2007
5.00	5.35 / 21.1	0.1998
5.25	5.34 / 21.1	0.1994
5.50	5.12 / 21.1	0.2004
5.75	4.95 / 21.1	0.2005
6.00	5.19 / 21.1	0.2002
6.25	5.49 / 21.1	0.1994
6.50	5.13 / 21.1	0.2000
6.75	6.23 / 21.2	0.1997
7.00	6.79 / 21.0	0.1995
7.25	6.97 / 21.0	0.2000
7.50	7.38 / 20.9	0.2008
7.75	7.55 / 21.2	0.1999
8.00	7.95 / 21.0	0.2010
8.25	8.13 / 20.9	0.1993
8.50	8.39 / 20.9	0.1994
8.75	8.61 / 20.9	0.2006
9.00	8.92 / 20.9	0.2009

10/2/92^{TD} EXPERIMENT B-I (cont)

SOLUTION	pH/TCC
B-I-C* pH 2.0 *a	1.92/20.8
*b	1.87/20.9
pH 4.0 *a	3.73/20.9
*b	3.71/20.9
pH 6.0 *a	5.07/20.9
*b	6.03/20.9
pH 8.0 *a	7.80/20.9
*b	7.70/20.9
pH 9.5 *a	9.36/20.9
*b	9.36/20.9

pH 4 buffer // pH 7 buffer 4.03/21.2 // 7.04/21.2

After the clinoptilolite was added, the bottles were swirled, recovered with a kimwipe, and returned to the gyratory shaker set at ~120 rpm.

TD

10/3/92^{TD} EXPERIMENT B-II

Remeasured the pH of all solutions prior to the addition of ~0.200g CDV*...*NaF. The zeolite was not added to B-II-C*phi solutions.

SOLUTION	pH/TCC	WEIGHT CDV*...*NaF ADDED (g)
B-II-C* pH 2.00	1.99/21.6	0.1994
2.25	2.16/21.7	0.2003
2.50	2.33/21.7	0.1994
2.75	2.61/21.4	0.2005
3.00	2.74/21.5	0.1999
3.25	3.11/21.5	0.1995
3.50	3.50/21.5	0.2009
3.75	3.57/21.5	0.1990
4.00	3.65/21.5	0.2007
4.25	4.30/21.5	0.1993
4.50	4.28/21.5	0.1997
4.75	4.24/21.5	^{TD} 0.201 0.1997
5.00	4.42/21.5	^{TD} 0.1995
5.25	4.75/21.7	0.2008
5.50	4.85/21.7	0.1999
5.75	6.61/21.6	0.1995
6.00	4.38/21.9	0.2005
6.25	4.72/21.9	0.2010
6.50	4.80/21.9	0.1996
6.75	6.39/21.9	0.1997
7.00	6.72/21.9	0.2006
7.25	6.86/21.7	0.1995
7.50	7.23/21.8	0.1994
7.75	7.62/21.8	0.2005
8.00	7.94/21.9	0.2007
8.25	8.08/21.8	0.1999
8.50	8.43/21.8	^{TD} 0.200 0.1992
8.75	8.68/21.8	0.2010
9.00	8.93/21.8	0.1994

10/3/12 EXPERIMENT B-II (cont) TD

SOLUTION	pH/T(°C)
B-II-C * pH2.0	1.96/21.6
4.0	3.64/21.6
6.0	4.33/21.6
8.0	7.73/21.6
9.5	9.37/21.6
pH 4 buffer	4.02/21.9
pH 7 buffer	7.07/21.9

The solutions were swirled, recovered w/ a kimwipe and returned to the gyratory shaker set to 120 rpm.

10/5/12 900 TD EXPERIMENT B-III

Remeasured the pH of all solutions prior to the addition of ~0.200g CDV*...*Naf. The CDV*...*Naf was not added to the B-III-C * pH 6.0/6.0(b) solutions.

SOLUTION	pH/T(°C)	WEIGHT CDV*...*Naf ADDED(g)
B-III * pH2.00	1.94/20.9	0.2008
2.25	2.20/20.9	0.2003
2.50	2.45/20.9	0.2001
2.75	2.79/20.9	0.1996
3.00	2.97/20.9	0.2002
3.25	3.23/20.9	0.1998
3.50	3.38/20.9	0.1999
3.75	3.77/20.9	0.2008
4.00	3.99/20.9	0.2008
4.25	4.12/21.0	0.1992
4.50	4.30/21.0	0.2000
4.75	4.56/21.0	0.1995
5.00	4.76/21.0	0.2002
5.25	5.53/21.0	0.2007
5.50	5.51/21.0	0.2005
5.75	5.71/21.0	0.2010
6.00	5.99/21.0	0.1996
6.25	6.05 ^{10/5/12} 5.99/21.0	0.2001
6.50	6.30/21.0	0.1999
6.75	6.69/21.0	0.2001
7.00	6.61/21.0	0.2008
7.25	7.07/21.0	0.1998
7.50	7.36/21.1	0.2002
7.75	7.56/21.1	0.2004
8.00	7.77/21.1	0.2003
8.25	8.04/21.1	0.2009
8.50	8.35/21.1	0.2003
8.75	8.64/21.1	0.1993
9.00	8.92/21.1	0.2007

10/5/92 EXPERIMENT B-II TD

SOLUTION	pH/T(°C)
B-II *C *pH2.0 *a	1.93/21.1
*b	1.98/21.1
*pH4.0 *a	4.02/21.1
*b	3.94/21.1
*pH6.0 *a	5.85/21.2
*b	5.82/21.2
*pH8.0 *a	7.76/21.2
*b	7.73/21.2
*pH9.5 *a	9.36/21.2
*b	9.35/21.2
pH 4 buffer	4.02/21.2
pH 7 buffer	7.05/21.2

The solutions were re-covered with a Kimwipe, swirled, and returned to the gyratory shaker set at ~120 rpm

10/19/92 TD EXPERIMENT K3

Took samples K3-1 *FU *a(b) & K3-2 *FU *a(b)

These samples were weighed and 500 μ L
1.0M HNO_3 was added

SAMPLE	WEIGHT(g)
K3-1 *FU *a	10.0501
*b	10.0306
K3-2 *FU *a	10.0112
*b	10.0934

10/21/92 Experiments B-I \rightarrow B-III TD

5 mL (~5.0g) of HNO_3 0.02M HNO_3 was placed in
labeled centrifuge tubes that will contain the samples
taken from the B-I, B-II, and B-III solutions.

10/21/92
D EXPERI 23 Oct 1992 TD EXPERIMENT B-I

Measured the Final pH of all solutions after taking $\frac{2}{10}$ 5ml
Samples.

SOLUTION	pH/T(°C)	WT. (g) A	WT (g) B
B-I *pH2.00	2.06/23.1	5.0667	5.0497
2.25	2.29/23.1	5.0542	5.0511
2.50	2.45/23.1	5.0296	5.0310
2.75	2.85/23.1	5.0558	4.9505
3.00	2.96/23.1	4.9404	5.0541
3.25	3.32/23.1	4.9996	5.0380
3.50	3.55/23.1	5.0232	5.0033
3.75	3.78/23.2	5.0595	5.0470
4.00	3.99/23.2	5.0588	5.0140
4.25	4.65/23.2	5.0299	4.9975
4.50	4.83/23.2	5.0034	5.0064
4.75	5.03/23.2	4.9501	4.9576
5.00	5.30/23.2	5.0016	5.0056
5.25	5.74/23.2	4.9951	4.9881
5.50	5.53/23.2	4.9843	5.0077
5.75	5.39/23.2	4.9709	5.0061
6.00	5.58/23.2	4.9242	4.9609
6.25	5.81/23.2	4.9993	4.9994
6.50	5.55/23.2	4.9738	4.9757
6.75	6.36/23.2	4.9744	4.8914
7.00	6.83/23.1	5.0229	5.0325
7.25	7.05/23.1	5.0014	5.0129
7.50	7.46/22.9	5.0315	5.0317
7.75	7.66/23.2	5.0556	5.0023
8.00	7.94/23.0	5.0231	5.0382
8.25	8.23/23.0	5.0423	5.0102
8.50	8.48/23.0	5.0254	5.0375
8.75	8.70/23.0	5.0388	5.0704
9.00	8.93/23.0	5.0341	5.0649
- C *pH2.0 *a	1.99/23.0	5.0686	5.0149
b	1.92/23.0	5.0139	5.0580

Switch values TD

B-I (CONT)

<u>SOLUTION</u>	<u>pH/T(°C)</u>	<u>WT(g) A</u>	<u>WT(g) B</u>
B-I-C *pH 4.0 *a	3.78/23.0	4.8829	5.0934
xb	TP 6.1 3.77/23.0	5.0232	5.0324
6.0 *a	5.12/23.0	5.0007	5.0172
b	6.15/22.8	4.9858	4.9996
8.0 *a	7.92/23.0	5.0204	4.9983
b	7.93/23.0	5.0044	5.0170
9.5 *a	9.45/22.8	4.9063	4.9750
b	9.45/22.8	5.0533	5.0065

24 Oct 1992 TO EXPERIMENT B-II

Measured the Final pH of all solutions after 2 - 5ml samples.

<u>SOLUTION</u>	<u>pH/T(°C)</u>	<u>WT(g) A</u>	<u>WT(g) B</u>
B-II *pH 2.00	2.06/22.8	5.0386	5.0535
2.25	2.22/22.7	4.9512	4.9494
2.50	2.39/22.7	4.9813	4.9630
2.75	2.72/22.3	5.0160	5.0546
3.00	2.81/22.4	5.0359	5.0670
3.25	3.20/22.4	4.9666	5.0464
3.50	3.64/22.4	5.0157	5.0406
3.75	3.77/22.3	5.0540	4.9305
4.00	3.90/22.3	5.0335	5.0263
4.25	4.63/22.4	5.0511	5.0481
4.50	4.63/22.3	5.0018	5.0354
4.75	4.56/22.3	5.0391	5.0150
5.00	4.75/22.3	5.0612	5.0361
5.25	5.07/22.7	4.9897	4.9878
5.50	5.14/22.7	4.9441	4.9830
5.75	6.26/22.8	5.0258	5.0556
6.00	4.63/22.5	4.9920	5.0541
6.25	5.03/22.6	5.0382	5.0480
6.50	5.81/22.6	5.0443	5.0664
6.75	6.37/22.6	5.0468	5.0172
7.00	6.63/22.5	5.0122	5.0234
7.25	7.06/22.7	4.9172	4.9683
7.50	7.12/22.6	4.9239	4.9850
7.75	7.50/22.6	5.0036	5.0022
8.00	7.90/22.5	4.9892	5.0509
8.25	8.03/22.6	5.0196	5.0289
8.50	8.34/22.6	5.0102	5.0396
8.75	8.60/22.6	4.9298	5.0360
9.00	8.88/22.6	4.9776	5.0152
-C *pH 2.0	1.96/22.4	5.0232	5.0136
4.0	3.60/22.4	5.0008	5.0457
6.0	4.27/22.4	4.9206	4.9452
8.0	7.66/22.4	5.0801	4.9056
9.5	9.37/22.4	5.0586	5.0570

25 Oct 1992 TD EXPERIMENT B-III

Measured the Final pH after taking 2-5 ml samples.

SOLUTION	pH/T(°C)	WT(g) A	WT(g) B
B-III * pH 2.00	2.00/20.9	4.9897	4.9748
2.25	2.27/20.9	4.9089	5.0008
2.50	2.53/20.9	4.9659	4.9625
2.75	2.88/20.9	4.9696	4.9895
3.00	3.08/20.9	4.9679	4.9089
3.25	3.36/20.9	4.9969	4.9934
3.50	3.53/20.9	4.9991	4.9910
3.75	3.99/20.9	5.0341	5.0334
4.00	4.25/20.9	4.9759	5.0090
4.25	4.40/20.9	5.0253	5.0211
4.50	4.59/20.9	5.0016	5.0410
4.75	4.91/20.9	5.0201	5.0142
5.00	5.12/21.0	5.0192	4.9833
5.25	5.75/21.0	5.0908	5.0657
5.50	5.69/21.0	5.1512	4.9653
5.75	5.88/21.0	4.9110	4.9875
6.00	6.06/21.0	4.9557	4.9438
6.25	6.18/21.0	4.9708	5.0152
6.50	6.38/21.0	4.9294	4.9521
6.75	6.78/21.0	4.9338	4.9634
7.00	6.58/21.0	4.9437	4.9003
7.25	7.02/21.0	4.9549	4.9774
7.50	7.30/21.0	5.1711	4.9945
7.75	7.60/21.0	5.0064	4.9854
8.00	7.84/21.0	4.9635	4.9477
8.25	8.10/21.0	4.9126	4.9729
8.50	8.32/21.0	4.9234	4.9535
8.75	8.59/21.0	4.9345	4.9568
9.00	8.67/21.0	4.9920	5.0067
-C * pH 2.0 x a	1.92/21.1	5.0006	4.9678
b	1.88/21.1	5.0082	4.9970
4.0 x a	4.02/21.1	4.9831	4.9967
b	3.93/21.1	4.9909	4.9906

B-III (CONT)

SOLUTION	pH/T(°C)	WT(g) A	WT(g) B
6.0 x a	5.76/21.1	4.9134	4.9647
b	5.90/21.1	4.9929	4.9231
8.0 x a	7.72/21.1	4.9770	5.0227
b	7.78/21.1	4.9559	4.9675
9.5 x a	9.37/21.1	5.0144	5.0042
b	9.37/21.1	4.9646	5.0171

TD 28 Oct 1992 EXPERIMENT B-III

Remeasured the pH of all solutions.

SOLN	pH/T(°C)	SOLN	pH/T(°C)
B-III * pH 2.00	2.04/23.8	B-III * pH 7.50	7.33/23.9
2.25	2.31/23.8	7.75	7.62/23.9
2.50	2.57/23.8	8.00	7.82/23.9
2.75	2.92/23.8	8.25	8.12/23.9
3.00	3.12/23.8	8.50	8.34/23.9
3.25	3.40/23.8	8.75	8.63/23.9
3.50	3.57/23.8	9.00	8.88/23.9
3.75	4.02/23.8	B-III * pH 2.0 x a	1.94/23.8
4.00	4.27/23.9	b	1.92/23.8
4.25	4.43/23.8	4.0 x a	4.05/23.8
4.50	4.65/23.8	b	3.95/23.8
4.75	5.00/23.8	6.0 x a	6.11/23.8
5.00	5.21/23.9	b	6.09/23.8
5.25	5.81/23.9	8.0 x a	7.80/23.8
5.50	5.82/23.9	b	7.86/23.8
5.75	6.08/23.9	9.5 x a	9.38/23.8
6.00	6.13/23.9	b	9.39/23.8
6.25	6.27/23.9		
6.50	6.44/23.9		
6.75	6.77/23.9		
7.00	6.60/23.9		
7.25	7.03/23.9		

10/28/92 EXPERIMENT B-I

Remeasured the pH of all solutions.

SOLUTION	pH/T(°C)	SOLUTION	pH/T(°C)
B-I- $\text{pH} 2.00$	2.00/22.5	B-I-C* $\text{pH} 0.0 \times a$	5.26/22.5
2.25	2.22/22.5	b	6.07/22.5
2.50	2.38/22.5	8.0 $\times a$	7.83/22.5
2.75	2.80/22.5	b	7.86/22.5
3.00	2.90/22.5	9.5 $\times a$	9.39/22.5
3.25	3.25/22.5	b	9.88/22.5
3.50	3.49/22.5		
3.75	3.74/22.5		
4.00	3.95/22.5		
4.25	4.65/22.5		
4.50	4.89/22.5		
4.75	5.05/22.5		
5.00	5.37/22.5		
5.25	5.75/22.5		
5.50	5.72/22.5		
5.75	5.47/22.5		
6.00	5.61/22.5		
6.25	5.88/22.5		
6.50	5.71/22.5		
6.75	6.37/22.5		
7.00	6.89/22.5		
7.25	7.03/22.5		
7.50	7.42/22.5		
7.75	7.66/22.5		
8.00	7.91/22.5		
8.25	8.17/22.5		
8.50	8.40/22.5		
8.75	8.65/22.5		
9.00	8.90/22.5		
B-I-C* $\text{pH} 2.0 \times a$	1.89/22.5		
b	1.83/22.5		
4.0 $\times a$	3.72/22.5		
b	3.70/22.5		

10/28/92 EXPERIMENT B-II

Remeasured the pH of all solutions

SOLUTION	pH/T(°C)
B-II- $\text{pH} 2.00$	1.07/22.5
2.25	2.24/22.5
2.50	2.41/22.5
2.75	2.70/22.5
3.00	2.80/22.5
3.25	3.21/22.5
3.50	3.64/22.5
3.75	3.77/22.5
4.00	3.90/22.5
4.25	4.67/22.5
4.50	4.64/22.5
4.75	4.55/22.5
5.00	4.78/22.5
5.25	5.29/22.5
5.50	5.29/22.5
5.75	6.54/22.5
6.00	4.73/22.5
6.25	5.15/22.5
6.50	5.91/22.5
6.75	6.46/22.5
7.00	6.70/22.5
7.25	7.15/22.5
7.50	7.30/22.5
7.75	7.64/22.5
8.00	7.95/22.5
8.25	8.06/22.5
8.50	8.37/22.5
8.75	8.65/22.5
9.00	8.90/22.5
B-II-C* $\text{pH} 2.0$	1.94/22.5
4.0	3.62/22.5
6.0	4.31/22.5
8.0	7.70/22.5
9.5	9.38/22.5

10/30/92 ~~Two~~ ^{TD} 10/30/92
K3-1 & K3-2 TD

2 additional samples, labeled 13a or 13b, were taken from each of the K3-1 and K3-2 sample STD solutions in the same manner as before.

SAMPLE E

SOLN ID	DATE/TIME	pH/T(°C)	Wt Sample A (g)	Wt Sample B (g)
K3-1#A#13	10/30, 0935	5.65/21.3	10.0930	10.0884
#B*	10/30, 0940	6.06/21.3	10.0677	10.0521
#C*	10/30, 0954	5.23/21.4	9.9513	9.9658
K3-2#A*	10/30, 0945	6.86/21.3	10.0293	10.0535
#B*	10/30, 0950	6.74/21.4	10.0238	10.0107
#C*	10/30, 0956	6.79/21.4	10.0303	10.0058

TD 10/30/92 EXPERIMENT B-I

TD 10/30/92
Soc Initiated reverse experiments by adjusting the pH either up or down using amounts of acid or base calculated using EQ3. d=drops. NaHCO_3 lot # 897186A.
 HNO_3 prepared 5 Aug 1992, see GL-07-149.

SOLN	pH/T(°C)	Adj. needed	Adj. made
B-I# pH 3.00	2.89/21.9	25d, 1.0M HNO_3	
3.50	3.48/21.9	8d, 1.0M	
4.00	3.94/21.9	25d, 0.1M	
4.50	4.92/21.9	8d, 0.1M	
5.00	5.35/21.9	25d, 0.01M	
5.50	5.58/21.9	8d, 0.01M	
6.00	5.55/21.9	4d, 0.01M	
6.25	5.72/21.9	4d, 0.01M	
6.50	5.58/21.9	0.00154g NaHCO_3	0.00163 g
6.75	6.25/21.9	0.00276	0.0286
7.00	6.80/21.9	0.00495	0.00525
7.25	6.95/21.9	0.00896	0.00932
7.50	7.29/22.0	0.0164	0.0167
8.00	7.79/22.0	0.1599	0.1605

11/2/92 TD EXPERIMENT B-I

Remeasured the pH of the remaining solutions.

SOLUTION	pH/T(°C)
TD# 11/2/92	
*2.00	1.97/20.0
2.25	2.21/20.0
2.50	2.36/20.0
2.75	2.75/20.0
3.25	3.23/20.0
3.75	3.70/20.0
4.25	4.66/20.0
4.75	5.05/19.9
5.25	5.47/20.0
5.75	5.47/20.0
7.75	7.67/20.0
8.25	8.13/20.0
8.50	8.38/20.0
8.75	8.62/20.0
9.00	8.89/20.0
C#20#A	1.83/20.2
b	1.79/20.2
4.0#C	3.66/20.1
b	3.65/20.1
6.0#C	5.21/20.1
b	6.13/20.0
8.0#C	7.77/20.2
b	7.75/20.1
9.5#C	9.38/20.0
b	9.39/20.0

4/2/92 TP EXPERIMENT B-II

Remeasured the pH of all solutions. Some of the solutions had their pH adjusted according to Table I on page 217. This adjustment marked the beginning of the reverse experiment.

SOLN	pH/T(°C)	SOLN	pH/T(°C)
2.00	2.09/20.4	8.75	8.70/20.4
2.25	2.26/20.5	9.00	8.95/20.3
2.50	2.43/20.5	Cx pH 2.0	1.95/21.2
2.75	2.70/21.3	4.0	3.61/21.2
3.00	2.83/21.2	6.0	4.34/21.2
3.25	3.21/21.2	8.0	7.69/21.2
3.50	3.66/21.2	9.5	9.38/21.2
3.75	3.79/21.3		
4.00	3.92/21.3		
4.25	4.69/21.2		
4.50	4.63/21.3		
4.75	4.60/21.3		
5.00	4.89/21.3		
5.25	5.22/20.3		
5.50	5.35/20.5		
5.75	6.62/20.5		
6.00	4.78/20.3		
6.25	5.14/21.3		
6.50	5.81/20.3		
6.75	6.51/20.2		
7.00	6.72/20.2		
7.25	7.11/20.3		
7.50	7.27/20.4		
7.75	7.62/20.3		
8.00	7.92/20.3		
8.25	8.07/20.4		
8.50	8.36/20.3		

The adjustment made is also shown in Table I. The adjustments made with acid were exactly what was given

TABLE I

Solution, either B-II or B-III	Adjustment Needed (B-III)	Adjustment Made (B-III)
3.00	25 drops 1.0M HNO ₃	---
3.50	8 drops	---
4.00	25 drops 0.1M HNO ₃	---
4.50	8 drops	---
5.00	25 drops 0.01M HNO ₃	---
5.50	9 (8) drops	---
6.00	5 (4) drops	---
6.25	5 (4) drops	---
6.50	0.0015 g NaHCO ₃	0.0015 g (0.0019 g)
6.75	0.0028 g	0.0030 g (0.0034 g)
7.00	0.0050 g	0.0051 g (0.0052 g)
7.25	0.0090 g	0.0090 g (0.0091 g)
7.50	0.0164 g	0.0166 g (0.0167 g)
8.00	0.1599 g	0.1599 g (0.1598 g)

The values in parenthesis are those for B-III, the others are for B-II.

4/2/92 TP EXPERIMENT B-III

starting the reverse experiment

Remeasured the pH of all solutions. Adjustments were made according to Table I at above. The adjustments were recorded in the table in parenthesis. Acid adjustments were ~~not~~ exactly what was given

<u>SOLN</u>	<u>pH/T(°C)</u>	<u>SOLN</u>	<u>pH/T(°C)</u>	<u>SOLN</u>	<u>pH/T(°C)</u>
2.00	2.01/21.5	4.75	5.00/21.6	7.75	7.56/21.9
2.25	2.28/21.5	5.00	5.20/21.6	8.00	7.79/21.8
2.50	2.54/21.5	5.25	5.83/21.6	8.25	8.06/21.9
2.75	2.90/21.5	^{11/21/92} 5.75 5.50	5.81/21.6	8.50	9.32/21.9
3.00	3.11/21.5	^{11/21/92} 6.00 5.75	5.97/21.6	8.75	9.62/21.9
3.25	3.39/21.5	^{11/21/92} 6.25 6.00	6.15/21.6	9.00	8.88/21.9
3.50	3.56/21.5	^{11/21/92} 6.50 6.25	6.24/21.7	C-2.0 (a) 1.91/21.5	(b) 1.98/21.5
3.75	4.02/21.6	^{11/21/92} 6.75 6.50	6.41/21.7	4.0 (a) 4.04/21.4	(b) 3.95/21.4
4.00	4.29/21.6	^{11/21/92} 7.00 6.75	6.74/21.7	6.0 (a) 6.05/21.4	(b) 6.06/21.4
4.25	4.44/21.6	^{11/21/92} 7.25 7.00	6.65/21.8	8.0 (a) 7.81/21.4	(b) 7.80/21.4
4.50	4.65/21.6	^{11/21/92} 7.50 7.25	7.05/21.8	9.5 (a) 9.38/21.4	(b) 9.40/21.4
		^{11/21/92} 7.50 7.50	7.36/21.8		

11/9/92 TO EXPERIMENT B-III.

The samples taken on 25 Oct 1992 (GC-07-210) were spiked with a ^{232}U solution, either 2.0187 nCi/g activity at^{TD} (19B) or 0.2047 nCi/g (19C). The amount of spike needed was weighed in a plastic weighing boat, and then washed quantitatively into the centrifuge tube containing the sample. The approximate volume of liquid left^{TD} in the was also noted _{11/9/92}

Identification	Spike#	Wt (g)	Wt(g) used	Vol (mL)
B-III*IUa	19B	0.119	0.1193	19
B-III*IUb	19B	0.119	0.1187	14
B-III-C*pH2*a1	19B	0.107	0.1058	16
B-III-C*pH2*a2	19B	0.107	0.1038	17
B-III-C*pH2*b1	19B	0.107	0.1052	18
B-III-C*pH2*b2	19B	0.107	0.1063	17
B-III-C*pH4*a1	19B	0.107	0.1049	18
B-III-C*pH4*a2	19B	0.107	0.1101	15
B-III-C*pH4*b1	19B	0.107	0.1054	15
B-III-C*pH4*b2	19B	0.107	0.1061	16
B-III-C*pH6*a1	19B	0.095	0.0941	17
B-III-C*pH6*a2	19B	0.095	0.0943	17
B-III-C*pH6*b1	19B	0.095	0.0954	15
B-III-C*pH6*b2	19B	0.095	0.0945	17
B-III-C*pH8*a1	19C	0.714	0.7137	25
B-III-C*pH8*a2	19C	0.714	0.7134	20
B-III-C*pH8*b1	19C	0.714	0.7142	17
B-III-C*pH8*b2	19C	0.714	0.7137	17
B-III-C*pH9.5*a1	19C	0.476	0.4756	17
B-III-C*pH9.5*a2	19C	0.476	0.4752	17
B-III-C*pH9.5*b1	19C	0.476	0.4757	17
B-III-C*pH9.5*b2	19C	0.476	0.4763	20
B-III*pH2.00*a	19C	0.952	0.9518	17
B-III*pH2.00*b	19C	0.952	0.9517	18
B-III*pH2.25*a	19C	0.922	0.9223	15
B-III*pH2.25*b	19C	0.922	0.9216	16
B-III*pH2.50*a	19C	0.893	0.8928	15
B-III*pH2.50*b	19C	0.893	0.8921	16
B-III*pH2.75*a	19C	0.863	0.8625	12
B-III*pH2.75*b	19C	0.863	0.8627	17
B-III*pH3.00*a	19C	0.833	0.8332	15
B-III*pH3.00*b	19C	0.833	0.8319	14
B-III*pH3.25*a	19C	0.786	0.7850	14
B-III*pH3.25*b	19C	0.786	0.7851	14
B-III*pH3.50*a	19C	0.762	0.7612	15
B-III*pH3.50*b	19C	0.762	0.7617	14
B-III*pH3.75*a	19C	0.714	0.7142	15
B-III*pH3.75*b	19C	0.714	0.7136	17
B-III*pH4.00*a	19C	0.417	0.4163	13
B-III*pH4.00*b	19C	0.417	0.4158	14
B-III*pH4.25*a	19C	0.298	0.2983	12
B-III*pH4.25*b	19C	0.298	0.2975	14

+ 2.7320g 19B 11/11/92 TD

EXPERIMENT B-III (CONT 11/9/92)

Identification	Spike#	Wt (g)	wt(g) used	Vol (mL)
B-III*pH4.50*a	19C	0.214	0.2146	13
B-III*pH4.50*b	19C	0.214	0.2127	14
B-III*pH4.75*a	19C	0.095	0.0941	14
B-III*pH4.75*b	19C	0.095	0.0955	14
B-III*pH5.00*a	19C	0.060	0.0601	13
B-III*pH5.00*b	19C	0.060	0.0592	14
B-III*pH5.25*a	19C	0.060	0.0599	15
B-III*pH5.25*b	19C	0.060	0.0597	13
B-III*pH5.50*a	19C	0.060	0.0595	14
B-III*pH5.50*b	19C	0.060	0.0596	14
B-III*pH5.75*a	19C	0.060	0.0600	14
B-III*pH5.75*b	19C	0.060	0.0590	14
B-III*pH6.00*a	19C	0.060	0.0605	14
B-III*pH6.00*b	19C	0.060	0.0605	13
B-III*pH6.25*a	19C	0.060	0.0604	14
B-III*pH6.25*b	19C	0.060	0.0597	14
B-III*pH6.50*a	19C	0.060	0.0599	16
B-III*pH6.50*b	19C	0.060	0.0590	14
B-III*pH6.75*a	19C	0.060	0.0605	14
B-III*pH6.75*b	19C	0.060	0.0588	14
B-III*pH7.00*a	19C	0.060	0.0597	14
B-III*pH7.00*b	19C	0.060	0.0590	14
B-III*pH7.25*a	19C	0.060	0.0596	14
B-III*pH7.25*b	19C	0.060	0.0593	15
B-III*pH7.50*a	19C	0.119	0.1182	15
B-III*pH7.50*b	19C	0.119	0.1193	14
B-III*pH7.75*a	19C	0.238	0.2382	15
B-III*pH7.75*b	19C	0.238	0.2387	17
B-III*pH8.00*a	19C	0.357	0.3572	16
B-III*pH8.00*b	19C	0.357	0.3561	14
B-III*pH8.25*a	19C	0.536	0.5368	14
B-III*pH8.25*b	19C	0.536	0.5352	14
B-III*pH8.50*a	19B	0.095	0.0951	18
B-III*pH8.50*b	19B	0.095	0.0941	14
B-III*pH8.75*a	19B	0.101	0.1003	20
B-III*pH8.75*b	19B	0.101	0.1001	16
B-III*pH9.00*a	19B	0.107	0.1079	17
B-III*pH9.00*b	19B	0.107	0.1072	17

The samples will be allowed to equilibrate for at least 24 hours before proceeding to proceeding. _{11/9/92}

TOTAL 19B USED = 2.0656 g
 " 19C " = 22.4935 g _{11/11/92} TD 23.9206 g

11/11/92 EXPERIMENT B-I

the samples taken on 25 Oct were spiked as before (p.21B) using ¹¹¹In spikes 19B (2.0187 nCi/g) and 19C (0.20147 nCi/g)

Identification	Spike#	Wt (g)	wt used (g)	Approx. Final Vol. (mL)
B-I*IUa	19B	1.188	1.1793	15
B-I*IUb	19B	1.188	1.1796	15
B-I-C*pH2*a1	19B	1.176	1.1764	15
B-I-C*pH2*a2	19B	1.176	1.1754	15
B-I-C*pH2*b1	19B	1.176	1.1756	15
B-I-C*pH2*b2	19B	1.176	1.1761	14
B-I-C*pH4*a1	19B	1.128	1.1278	15
B-I-C*pH4*a2	19B	1.128	1.1273	15
B-I-C*pH4*b1	19B	1.128	1.1278	15
B-I-C*pH4*b2	19B	1.128	1.1283	15
B-I-C*pH6*a1	19B	0.950	0.9501	14
B-I-C*pH6*a2	19B	0.950	0.9496	15
B-I-C*pH6*b1	19B	0.950	0.9490	14
B-I-C*pH6*b2	19B	0.950	0.9490	15
B-I-C*pH8*a1	19B	0.713	0.7133	16
B-I-C*pH8*a2	19B	0.713	0.7127	14
B-I-C*pH8*b1	19B	0.713	0.7115	14
B-I-C*pH8*b2	19B	0.713	0.7129	14
B-I-C*pH9.5*a1	19B	0.475	0.4742	15
B-I-C*pH9.5*a2	19B	0.475	0.4752	16
B-I-C*pH9.5*b1	19B	0.475	0.4747	15
B-I-C*pH9.5*b2	19B	0.475	0.4750	14
B-I*pH2.00*a	19B	0.950	0.9497	15
B-I*pH2.00*b	19B	0.950	0.9504	16
B-I*pH2.25*a	19B	0.921	0.9212	15
B-I*pH2.25*b	19B	0.921	0.9188	15
B-I*pH2.50*a	19B	0.915	0.9141	15
B-I*pH2.50*b	19B	0.915	0.9146	14
B-I*pH2.75*a	19B	0.832	0.8321	15
B-I*pH2.75*b	19B	0.832	0.8316	15
B-I*pH3.00*a	19B	0.832	0.8323	14
B-I*pH3.00*b	19B	0.832	0.8318	14
B-I*pH3.25*a	19B	0.784	0.7831	16
B-I*pH3.25*b	19B	0.784	0.7833	15
B-I*pH3.50*a	19B	0.760	0.7599	15
B-I*pH3.50*b	19B	0.760	0.7591	15
B-I*pH3.75*a	19B	0.742	0.7411	14
B-I*pH3.75*b	19B	0.742	0.7411	15
B-I*pH4.00*a	19B	0.713	0.7127	15
B-I*pH4.00*b	19B	0.713	0.7124	15
B-I*pH4.25*a	19B	0.214	0.2139	16
B-I*pH4.25*b	19B	0.214	0.2132	14

Identification	Spike#	Wt (g)	wt used (g)	Approx. Final Vol. (mL)
B-I*pH4.50*a	19C	1.190	1.1890	15
B-I*pH4.50*b	19C	1.190	1.1898	15
B-I*pH4.75*a	19C	0.595	0.5948	14
B-I*pH4.75*b	19C	0.595	0.5954	15
B-I*pH5.00*a	19C	0.595	0.5940	15
B-I*pH5.00*b	19C	0.595	0.5949	15
B-I*pH5.25*a	19C	0.595	0.5946	14
B-I*pH5.25*b	19C	0.595	0.5955	15
B-I*pH5.50*a	19C	0.595	0.5946	14
B-I*pH5.50*b	19C	0.595	0.5954	15
B-I*pH5.75*a	19C	0.595	0.5942	16
B-I*pH5.75*b	19C	0.595	0.5946	15
B-I*pH6.00*a	19C	0.595	0.5949	14
B-I*pH6.00*b	19C	0.595	0.5948	16
B-I*pH6.25*a	19C	0.595	0.5950	14
B-I*pH6.25*b	19C	0.595	0.5957	14
B-I*pH6.50*a	19C	0.595	0.5951	15
B-I*pH6.50*b	19C	0.595	0.5948	15
B-I*pH6.75*a	19C	0.595	0.5942	14
B-I*pH6.75*b	19C	0.595	0.5951	15
B-I*pH7.00*a	19C	0.595	0.5944	15
B-I*pH7.00*b	19C	0.595	0.5945	15
B-I*pH7.25*a	19C	0.595	0.5957	14
B-I*pH7.25*b	19C	0.595	0.5946	15
B-I*pH7.50*a	19C	1.190	1.1892	17
B-I*pH7.50*b	19C	1.190	1.1896	16
B-I*pH7.75*a	19C	2.381	2.3801	15
B-I*pH7.75*b	19C	2.381	2.3798	25
B-I*pH8.00*a	19B	0.475	0.4747	14
B-I*pH8.00*b	19B	0.475	0.4740	14
B-I*pH8.25*a	19B	0.653	0.6532	14
B-I*pH8.25*b	19B	0.653	0.6524	15
B-I*pH8.50*a	19B	1.010	1.0105	14
B-I*pH8.50*b	19B	1.010	1.0093	15
B-I*pH8.75*a	19B	1.010	1.0096	15
B-I*pH8.75*b	19B	1.010	1.0105	14
B-I*pH9.00*a	19B	1.069	1.0682	15
B-I*pH9.00*b	19B	1.069	1.0690	15

The solutions were allowed to equilibrate for at least 24 hours before proceeding.

11/11/92 - 11/12/92 EXPERIMENT B-II

The B-II samples were spiked as before (p. 218) using spikes 19A (20.187 nCi/g) and 19B (2.0187 nCi/g).

The samples B-II * pH 4.25 * a and ... * pH 7.50 * b were spiked with 19A, the amount needed was obtained by dividing that given by 10. The approximate final vol. was noted.

Identification	Spike#	Wt (g)	wt. used (g)	vol (ml)
B-II*IUa	19A	1.188	1.1868	15
B-II*IUb	19A	1.188	1.1973	15
B-II-C*pH2*a	19A	1.176	1.1760	17
B-II-C*pH2*b	19A	1.176	1.1752	16
B-II-C*pH4*a	19A	1.128	1.1267	16
B-II-C*pH4*b	19A	1.128	1.1267	17
B-II-C*pH6*a	19A	0.950	0.9492	15
B-II-C*pH6*b	19A	0.950	0.9501	15
B-II-C*pH8*a	19A	0.713	0.7127	16
B-II-C*pH8*b	19A	0.713	0.7132	16
B-II-C*pH9.5*a	19A	0.475	0.4748	15
B-II-C*pH9.5*b	19A	0.475	0.4755	15
B-II*pH2.00*a	19A	0.950	0.9501	15
B-II*pH2.00*b	19A	0.950	0.9496	15
B-II*pH2.25*a	19A	0.921	0.9202	15
B-II*pH2.25*b	19A	0.921	0.9203	15
B-II*pH2.50*a	19A	0.921	0.9203	15
B-II*pH2.50*b	19A	0.921	0.9203	15
B-II*pH2.75*a	19A	0.921	0.9211	15
B-II*pH2.75*b	19A	0.921	0.9206	15
B-II*pH3.00*a	19A	0.921	0.9206	15
B-II*pH3.00*b	19A	0.921	0.9215	15
B-II*pH3.25*a	19A	0.921	0.9208	15
B-II*pH3.25*b	19A	0.921	0.9205	15
B-II*pH3.50*a	19A	0.903	0.9023	15
B-II*pH3.50*b	19A	0.903	0.9032	15
B-II*pH3.75*a	19A	0.832	0.8321	15
B-II*pH3.75*b	19A	0.832	0.8323	16
B-II*pH4.00*a	19A	0.653	0.6538	15
B-II*pH4.00*b	19A	0.653	0.6533	15
B-II*pH4.25*a	19B	2.732	2.7330	13
B-II*pH4.25*b	19B	2.732	2.7315	17
B-II*pH4.50*a	19B	2.732	2.7318	17
B-II*pH4.50*b	19B	2.732	2.7313	17
B-II*pH4.75*a	19B	3.089	3.0877	17
B-II*pH4.75*b	19B	3.089	3.0874	17
B-II*pH5.00*a	19B	2.257	2.2558	17
B-II*pH5.00*b	19B	2.257	2.2564	17
B-II*pH5.25*a	19B	1.663	1.6623	16
B-II*pH5.25*b	19B	1.663	1.6621	18
B-II*pH5.50*a	19B	1.544	1.5448	15
B-II*pH5.50*b	19B	1.544	1.5428	15

Identification	Spike#	Wt (g)	wt. used (g)	vol (ml)
B-II*pH5.75*a	19B	0.713	0.7128	15
B-II*pH5.75*b	19B	0.713	0.7120	16
B-II*pH6.00*a	19B	2.613	2.6128	16
B-II*pH6.00*b	19B	2.613	2.6133	16
B-II*pH6.25*a	19B	1.782	1.7815	16
B-II*pH6.25*b	19B	1.782	1.7810	16
B-II*pH6.50*a	19B	0.832	0.8321	15
B-II*pH6.50*b	19B	0.832	0.8315	14
B-II*pH6.75*a	19B	0.594	0.5933	15
B-II*pH6.75*b	19B	0.594	0.5941	16
B-II*pH7.00*a	19B	0.594	0.5938	14
B-II*pH7.00*b	19B	0.594	0.5938	15
B-II*pH7.25*a	19B	1.188	1.1875	15
B-II*pH7.25*b	19B	1.188	1.1883	15
B-II*pH7.50*a	19B	1.782	1.7824	20
B-II*pH7.50*b	19B	1.782	1.7823	15
B-II*pH7.75*a	19A	0.511	0.5114	15
B-II*pH7.75*b	19A	0.511	0.5107	15
B-II*pH8.00*a	19A	0.903	0.9035	14
B-II*pH8.00*b	19A	0.903	0.9019	16
B-II*pH8.25*a	19A	1.033	1.0319	14
B-II*pH8.25*b	19A	1.033	1.0323	15
B-II*pH8.50*a	19A	1.093	1.0929	15
B-II*pH8.50*b	19A	1.093	1.0925	14
B-II*pH8.75*a	19A	1.128	1.1269	15
B-II*pH8.75*b	19A	1.128	1.1274	17
B-II*pH9.00*a	19A	1.164	1.1645	15
B-II*pH9.00*b	19A	1.164	1.1639	13

Sample ... 7.50 * a was accidentally spiked twice. All the spikes using 19A were done by 11/11/92. The samples were allowed to sit for at least 24 hrs. to equilibrate before proceeding.

11/13/92 EXPERIMENTS B-I, B-II, B-III

All samples had 1 mL of an iron ⁵⁹Fe carrier solution added to them. The solution is 24.3 mg Fe per 1 mL of solution. They will be allowed to equilibrate before continuing. The Fe carrier was prepared on 7/21/92 (031/56) by O.C.L.

11/18/92 TD

Two samples taken previously were used to get acquainted with the separation procedure given below. The samples were B-I*In*~~a~~ and B-I*~~pH2.00~~*~~a~~

- Bring the pH up to 7 with the addition of concentrated NH_4OH from the reagent bottle using a pasteur pipet. 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 is strongly exothermic and the solution should be gently swirled or stirred with a glass stirring rod during the addition of the base. If a glass rod is used, after stirring, rinse the rod into the beaker with ultrapure water and set the rod aside.
- The solution can then be centrifuged (labeled 50 ml centrifuge tubes) and the supernate is discarded.
- The $\text{Fe}(\text{OH})_3$ precipitates are washed with 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

- 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 or greater).
- The samples pH will be lowered to 1 using concentrated Nitric acid or any other concentration of Nitric acid so that the volume of solution is a minimum. Next, saturate the solution with Ammonium Nitrate and shake until all is dissolved. This reaction is highly endothermic.
- Add 30-40 mL of 8N Ammonium Nitrate - 0.1 N Nitric acid into anion exchange column and elute into plastic waste beaker.
- Add the sample into the column and elute Fe into a glass beaker (250 ml) using 80 ml, added in 20 ml increments using squirt bottle, of 8 N NH_4NO_3 - 0.1 N HNO_3 . Test for the presence of Fe after 70 and 80 ml have eluted 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.
- Next add 70-80 mL of 8N HCL acid and elute the Thorium into the same beaker. Dispose of solution in beaker down the sink with liberal flushing.
- Add no more than 5 mL of 0.1 N Nitric acid and drain into waste beaker. 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 near dryness.

C. Solvent extraction and plating

- The dried U eluate is taken up with two mls of 0.001 N HNO_3 (pH 3) issued from a squirt bottle. Using a new pasteur pipet carefully and completely wash the beaker with the acid. Transfer the acid solution to a glass centrifuge tube (7 or 12 mls). Repeat this step twice more with 0.5 - 1 ml of the acid.
- Add 1-2 ml of the 0.4 M TTA in benzene solution to the centrifuge tube.
- Homogenize and extract the U using the same pasteur pipet used in step C1.
- Solution is centrifuged for 1-2 minutes, making sure that the tube is covered.
- 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.
- The TTA solution is carefully separated using a clean pasteur pipet, taking care not to include any of the HNO_3 solution.
- The TTA was evaporated drop-wise on heated steel plates that have been placed on mounting ring, making sure that spattering is avoided.
- Pass the plate through the flame of a propane flame inside the fume to burn off the organic deposit.
- The TTA extraction is repeated (Steps 2- 7).
- Place plates in labeled glassine stamp envelopes.

D. Cleaning up

- 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.
- The used anion exchange resin should be rinsed with water from a squirt bottle into the used resin bottle (see instructions above).
- All glassware should then be washed usingalconox. The glassware should be rinsed with the single pass water. The glassware is then ready for the fuming nitric acid bath.

The acidification in step B-2 was accomplished using 0.25 ml 1.0M HNO_3 added to the 22 ml of $\text{Fe}(\text{OH})_3$, and raising the total volume to 5ml with 0.1 N HNO_3 . Ammonium Nitrate (NH_4NO_3) is saturated at 118.3g in 100 mL H_2O (from CPC). Therefore 5.9g NH_4NO_3 was added to our 5ml solution.

11/19/92 TD

The $U(232,233)$ was washed off the columns and extracted as done in section C on pg. 225.

11/20/92 TD

Prepared 0.1N HNO_3 by mixing 1987 ml of H_2O with 12.7 ml of conc. HNO_3 in a 2L acid bottle. Also prepared 1000 mL 8.0N NH_4NO_3 -0.1N HNO_3 by dissolving 640.5g NH_4NO_3 in 0.1N HNO_3 in a 1000 ml volumetric flask and diluting to the mark with H_2O .

11/21/92 TD

Samples B-I* $pH 2.25 \times a$ through $pH 4.00 \times a$ (only $\times a$ samples) were run through steps A-5 \rightarrow A-7 and B1-B6 on pg. 224

11/23/92 TD

prepared 100 mL 0.4 M TTA in benzene by dissolving 8.71g TTA (Thenoyltrifluoroacetone Lot # 904128) in 100 mL benzene in a volumetric flask.

11/23/92 TD

The B-I* $pH 2.25-4.00 \times a$ samples were all heated to dryness. B-I* $pH 2.25, 3.75$, and 4 were all plated onto a stainless steel planchet according to section C on page 225

Samples B-I* $pH 4.25 \times a$ — B-I*... $6.75 \times a$ (only "a" samples) were run through steps A5-A7 and B1-B6 on page 224

11/25/92 TD

Samples B-I* $pH 2.50 \times a$ — B-I* $pH 3.00 \times a$ were plated according to steps ~~ETD~~^{4/25/92} in section C on page 225.

11/27/92 TD

Samples B-I* $pH 7.00-8.75 \times a$ were run through steps ~~ETD~~ A5-A7 and B1-B6 on page 224

Sample B-I* $pH 3.25 \times a$ was plated according to section C on page 225.

11/30/92 TD

Samples from B-I with label of 3.50, 4.25, 4.50-6.00 were plated onto a stainless steel planchet according to the steps on page 225, section C

12/1/92 TD

Samples from B-I with $pH_i = 6.25-7.25$ were plated according to the steps on page 225, section C

12/3/92 TD

Samples B-I* $pH_i \times a$, $i = 7.50-7.75$ were plated according to p. 225, section C. Tube broke for 7.75, 7.50 dropped onto plastic tray while hot

12/4/92 TD

Samples B-I* $pH 8.00 \times a$ through B-I* $pH 8.75 \times a$ were plated according to section C page 225.

TD 12/10/92 B-I* $pH 9.0 \times a$, B-I* $C \times pH 2.0 \times a$ (bl), B-I-C* $pH 4.0 \times a$ (bl) and B-I* $pH 1.0$ B-I-C* $pH 6.0 \times a$ were run through steps A5-A7, B1-B6 on page 224.

12/14/92 TD

Samples B-I-C * pH 4.0 * a1 (or b1) → B-I-C * pH 9.5 * b1 were run through steps A5-7 and B1-6 on pg 224

1L of 8N NH_4NO_3 / 0.1N HNO_3 was prepared by dissolving 640.5 g NH_4NO_3 (lot # 922457) in 0.1N HNO_3 in a 1L volumetric flask.

Samples B-I * pH 9.00 * a, B-I-C * pH 2.0 * a1 (and b1), and B-I-C * pH 4.0 * a1 were all plated onto a stainless steel planchet as in sec C on page 225.

12/15/92 TD

Samples B-I-C * pH 4.0 * b1 and B-I-C * pH 6.0 * a1 were plated onto stainless steel planchets according to the procedure in sec C p 225.

12/16/92 TD

Samples B-I-C * pH i * a1 (b1), $i = 6, 8, 9.5$ were plated according to section C on page 225. The centrifuge tube with the sample $i = 8.0$ * a1 was broken, and some of the sample lost and possibly contaminated.

12/21/92 TD 0800

Prepared 250 mL 0.01N NaOH by dissolving 0.1g NaOH in 250 mL deionized H_2O in a volumetric flask.

wt NaOH used = 0.1004g NaOH TD 12/21/92
lot # 914599A

250 mL 0.1N NaOH was taken from a pre-made commercial solution, lot # 910651-24.

12/21/92 TD 0900

Prepared 1000 mL of 0.02% Crystal violet solution by dissolving 0.2001g of the Crystal violet powder in 1000 mL deionized H_2O in a Volumetric flask. This solution was placed in a clean, labeled 1000 mL PP bottle. ~125 mL was placed into a clean, labeled 125 mL PP bottle for easier use. This is a pH indicator from 0-1.8

lot # 915464

12/21/92 TD 1000

Prepared 1000 mL of 0.1% Benzopurpurin-4B solution by dissolving 1.0013g Benzopurpurin-4B in 1000 mL deionized H_2O in a volumetric flask. This solution was transferred to a 1000 mL PP bottle. ~125 mL was transferred to a 125 mL PP bottle.

lot # 572207.

This is a pH indicator from 2.2-4.2.

1200 TD

The solutions with pH ranging from 0 to 4.0, $\Delta\text{pH} = 0.25$ were prepared. ~3 mL of each solution was taken and mixed with 2 drops of the solution^{TD} indicator solution prepared above that covers the appropriate pH range for the solution (the solution pH was in the range of the indicator). A 0.1% Methyl Orange solution, lot # 905561-18, was also used to test solutions between pH 2.75 & - pH 4.00.

The purpose of this test is to confirm an indicator (pH) that will be used in the plating procedure on p. 225. It is necessary that the pH be between 3-3.5 for U to be plated rather than Th, which prefers lower pH. These results are on the next page.

pH	Color		
	Crystal Violet	Benzopurpurin	Methyl Orange
0	yellow		
0.25	green-yellow		
0.50	green		
0.75	blue-green		
1.00	green-blue		
1.25	light blue		
1.50	blue		
1.75	blue		
2.00	blue-violet		
2.25		purple	
2.50		purple	
2.75		reddish-purple	
3.00		reddish-purple	red
3.25		purplish-red	red-orange-red
3.50		purplish-red	orange
3.75		red	yellow-orange
4.00		red	orange-yellow

It was easy to distinguish the different colors visually, but harder to do so ^{to initial} with words in the table. Methyl Orange has a greater color difference and it is easier to notice the changes than the Benzopurpurin, so it will most likely be the indicator.

12/22/92 TD 0900

Prepared 3 solutions for plating, one with ^{233}U , one with ^{232}U , and one blank. These were prepared as a regular sample. 1 mL of the Fe carrier was added.

Wt. ^{233}U = 1.0005 g (Spike 234)

Wt. ^{232}U = 0.2008 g (Spike 19A)

These will be allowed to equilibrate at least overnight.

12/22/92 TD 1000

Began sampling the Reverse experiments. 2-5 mL samples were taken from each solution and placed into a preweighed centrifuge tubes containing ~5 g 0.02N HNO_3 . The tubes were labeled B-I *...* R a (orb). The results were entered into table 1 below. The pH of all solutions was also recorded ^{12/22/92} and recorded.

TABLE 1: SAMPLE WEIGHTS AND SOLUTION pH FOR THE REVERSE SORPTION EXPERIMENTS

Sample i	B-I			B-II			B-III		
	pH/T(°C)	Wt. A (g)	Wt. B (g)	pH/T(°C)	Wt. A (g)	Wt. B (g)	pH/T(°C)	Wt. A (g)	Wt. B (g)
3.00	1.96/19.3	5.0399	4.0416	1.85/20.8	5.0427	5.0598	1.87/20.6	5.0727	5.0669
3.50	2.36/19.3	5.0434	5.0382	2.36/20.8	5.0781	5.0551	2.31/20.7	5.0728	4.9477
4.00	2.94/19.4	5.0481	5.0630	2.88/20.9	5.0469	5.0490	2.88/20.7	5.0000	4.9931
4.50	3.62/19.4	5.0555	5.0466	3.50/20.9	5.0229	5.0468	3.44/20.7	4.9988	5.0065
5.00	4.11/19.4	5.0569	5.0226	4.02/20.9	5.0234	5.0391	4.17/20.7	5.0447	5.0101
5.50	4.79/19.5	5.0453	5.0359	4.77/20.9	5.0393	5.0318	4.99/20.7	5.0003	5.0017
6.00	5.15/19.5	5.0416	5.0230	4.60/21.0	5.0415	5.0280	5.39/20.7	4.9991	5.0063
6.25	6.06/19.6	5.0291	5.0491	4.93/21.0	5.0339	5.0402	5.75/20.7	5.0030	5.0114
6.50	7.00/19.6	5.0347	5.0435	7.26/21.1	5.0394	5.0573 ^{TD}	7.35/20.7	5.0176	5.0287
6.75	7.45/19.7	5.0617	5.0595	7.64/21.1	5.0447	5.0460	7.45/20.7	5.0069	5.0190
7.00	7.72/19.7	5.0404	5.0472	7.76/21.2	5.0400	4.9338	7.64/20.7	5.0246	5.0253
7.25	8.01/19.8	5.0319	5.0512	8.04/21.2	5.0465	5.0445	8.00/20.8	5.0345	5.0112
7.50	8.42/19.8	5.0629	5.0418	8.39/21.3	5.0454	5.0411	8.26/20.8	4.9997	4.6076
8.00	9.19/19.8	5.0577	5.0563	9.23/21.3	5.0425	5.0466	9.18/20.8	5.0176	5.0182

12/22/92 1200 TD

Sampled the B-II reverse experiments as done earlier today with the B-I solutions. The tubes were labeled B-II *...* R a (orb). The results are in Table 1 above.

12/28/92 1400 TD

Took samples from the B-III reverse samples. Samples were taken in the same manner as the previous samples, page 231. Results of weighing and pH measurements were recorded in table 1 on page 231.

12/29/92 TD

After counting several of the B-I*...^{TD 12/29/92} plates, it was determined by Bret Leslie that the extraction and plating procedure needed to be modified. The Alpha spectra showed too much ²²⁸Th to ²³²U be present. The amount of 8N HCl used to wash Th off the column, was increased to 100 mL. Also, prior to ^{TD 12/29/92} the Benzene-TTA extraction, the solution pH will be adjusted, if necessary, to ~3.0-3.5 using a 0.1% methyl orange indicator solution. The ²³³U, ²³²U, and blank were then run through the plating procedure found on page 224, with the modification above (100 mL 8N HCl) used.

12/30/92 TD

The three samples run through the columns were plated according to section C on page 225. The pH of the solutions ~~was~~^{TD 12/30/92} was checked with the 0.1% methyl orange indicator solution and compared with the color of the solutions prepared in the test on 12/21/92. The pH was adjusted with 0.1 N NaOH prepared on 12/21/92. The spectra were acquired and Bret Leslie determined the modifications were adequate.

1/4/93 TD

Run samples through the ^{TD 1/4/93} separation procedure found on page 224, only 100 mL 8N HCl was used. The samples were B-I*^{TD 1/4/93} Iu*b, B-I*^{TD 1/4/93} phi*b; i = 2.00-3.50, ΔpH = 0.25.

1/5/93 TD

Prepared 1 L 8N NH₄NO₃/0.1N HNO₃ by dissolving 640.5g NH₄NO₃ in 0.1N HNO₃ in a volumetric flask and diluting to the mark.

Prepared 2 L 8N HCl by dissolving 1322g ^{TD 1/5/93} conc HCl in 678 mL H₂O.

Samples B-I*^{TD 1/5/93} Iu*b, B-I*^{TD 1/5/93} phi*b; i = 2, 2.25, 3.00, were plated according to section C on page 225, prior to addition of TTA/Benzene, the pH of the solution was adjusted using 0.1N NaOH with 0.1% methyl orange as an indicator. After the organic was burned off and the plate cooled, the U was ^{TD 1/5/93} counted immediately on the α-spec system. The results were saved onto a disk for future ~~data~~ calculations.

1/6/93 TD

Samples B-I*^{TD 1/6/93} phi*b, i = 2.50, 2.75, 3.25, and 3.50 were plated and counted in the same manner as the previous days samples. Print outs of the spectra are kept in a binder entitled α-Spectrometry, ROE data, Peak data and Spectra.

1/8/93 TD

Samples B-I*^{TD 1/8/93} phi*b; i = 3.75 - 4.50, ΔpH = 0.25 were run through steps A^{TD 1/8/93} 7 and B1-6 on page 224.

1/19/93 TD

Prepared 2L 0.1N HNO_3 by mixing diluting:
12.7 mL conc HNO_3 to 2000 mL in a volumetric flask using DI H_2O .

2/4/93 TD

Prepared 2L 8N NH_4NO_3 / 0.1N HNO_3 by dissolving
1,281g NH_4NO_3 in 2L 0.1N HNO_3 in a 2L volumetric flask.

Prepared 2L 0.1N HNO_3 by diluting 12.7 mL conc. HNO_3
with DI water in a 2L volumetric flask.

1/15/93 TD

Samples 3.75 & 4.00 were plated and counted with α -spec.

1/18/93 TD

Samples 4.25 - 4.75 were counted using α -spectrometry

1/22/93 TD

Samples B-I * pH 5.5 - 5.50 were processed
and counted by α -spectrometry using
the procedure on pp. 224-225.

TD 1/23/93 Sample B-I * pH 6.50 was processed and counted
using the procedure on p. 224-5.

TD 1/29/93 Samples B-I * pH 5.75 - 6.25 were processed
and counted according to the procedure on p. 224-225.

TD 2/1/93 Samples B-I * pH 6.75 (& 7.00) were counted
after being run through the procedure on
pg. 224-5

TD

2/16/93 TD

Samples B-I * pH (7.25, 7.50) were processed and counted
using α -spectrometry.

TD 2/18/93 Samples B-I * pH 7.75 (8.00) were processed and counted
using α -spectrometry.

TD 3/12/93 Samples B-I * pH 8.50 - 8.75 were processed and counted by
 α -spectrometry according to the procedure on pp. 224-5.

TD 3/9/93

the K3-1 and K3-2 samples, and also the B-I * pH * R * a (or b)
samples were prepared for liquid scintillation counting
(LSC) according to the following procedure.

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. Plot away!

The results of the counting can be found below.

Protocol #: 5 Name: U-233 3% 2 sigma 04-Mar-93 08:48
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	19.03 1.45	3.006 3.65	28.32 1.19	136.02 B
2	6.75	4.82 78.23	655.512 3.01	659.68 3.06	714.77
3	6.58	6.19 63.38	672.678 3.01	678.06 3.06	718.80
4	6.67	27.59 19.19	664.010 3.01	692.52 3.00	693.91
5	6.67	2.26 158.9	663.861 3.01	666.88 3.06	719.46
6	6.37	1.76 180.3	528.416 3.02	530.58 3.08	722.92
7	10.79	2.28 123.7	409.042 3.02	411.90 3.10	719.03
8	14.00	20.90 16.22	314.484 3.03	335.25 3.04	675.91
9	19.98	0.00 0.00	219.516 3.04	218.33 3.22	722.19
10	41.94	14.61 12.41	162.955 3.09	117.22 3.19	635.38
11	16.84	21.76 14.36	260.689 3.04	283.97 3.04	667.41
12	8.59	0.00 0.00	514.456 3.02	513.01 3.10	721.70
13	7.05	2.24 155.4	627.774 3.01	629.41 3.07	718.86
14	6.67	0.76 456.7	663.711 3.01	666.28 3.06	722.73
15	6.55	3.26 113.6	675.925 3.01	678.85 3.06	711.88
16	7.46	4.02 87.67	593.375 3.01	597.55 3.07	717.61
17	6.50	4.35 87.41	681.302 3.01	686.14 3.06	720.40
18	6.65	4.88 77.97	666.317 3.01	671.08 3.06	714.68
19	7.18	3.11 113.2	616.493 3.01	620.01 3.07	717.45
20	8.24	3.54 93.85	536.557 3.02	541.10 3.07	711.27
21	10.53	6.23 49.94	419.311 3.02	424.95 3.09	708.22
22	14.03	0.21 1117.	313.744 3.03	314.44 3.15	716.42
23	19.93	2.04 101.7	220.075 3.04	222.71 3.19	714.16
24	41.73	0.57 245.8	103.512 3.09	104.44 3.43	715.30
25	17.16	16.28 17.70	256.027 3.04	272.03 3.08	677.00
26	8.67	0.69 439.1	509.566 3.02	510.32 3.09	721.34
27	7.01	8.50 46.75	631.373 3.01	639.15 3.05	710.77
28	6.57	166.81 6.38	673.858 3.01	641.08 2.74	579.15
29	6.60	5.08 75.75	670.630 3.01	677.74 3.05	707.52
30	4.55	2.94 149.6	974.357 3.01	979.81 3.04	711.54
31	4.62	9.54 52.23	961.713 3.01	972.33 3.03	710.71
32	4.79	10.40 47.73	926.221 3.01	938.28 3.03	706.82
33	4.83	10.16 48.47	918.319 3.01	930.08 3.03	707.21
34	4.96	319.68 5.17	894.171 3.01	1215.23 2.61	526.58
35	5.21	413.60 4.41	850.161 3.01	1264.96 2.49	485.62
36	5.95	218.11 5.79	744.389 3.01	963.78 2.68	554.31
37	6.83	2.64 135.6	648.092 3.01	652.21 3.06	712.16
38	6.74	165.09 6.33	656.786 3.01	822.57 2.73	572.77
39	6.93	5.06 73.84	638.408 3.01	643.68 3.06	709.88
40	7.97	142.45 6.32	554.961 3.02	697.40 2.74	571.72
41	8.21	183.89 5.41	538.407 3.02	724.78 2.64	537.83
42	8.59	220.90 4.79	515.038 3.02	736.29 2.58	503.51
43	9.28	3.27 95.12	475.873 3.02	479.65 3.09	715.38
44	9.95	2.88 103.6	443.627 3.02	446.95 3.09	716.64
45	10.19	1.28 221.6	433.206 3.02	435.27 3.10	715.32
46	10.76	107.08 6.40	410.191 3.02	519.17 2.75	570.44
47	11.52	2.58 106.7	383.018 3.02	385.13 3.11	713.17
48	12.01	185.30 4.45	367.102 3.02	552.19 2.52	482.18
49	12.30	84.14 6.89	358.457 3.02	442.82 2.80	585.90

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
50	13.44	10.65 28.02	327.723 3.03	339.24 3.08	701.08
51	13.81	0.81 298.9	318.862 3.03	318.96 3.15	715.79
52	5.73	6.10 68.84	772.736 3.01	782.32 3.04	714.22
53	5.41	8.14 55.18	818.990 3.01	828.61 3.04	713.04
54	6.42	57.29 12.05	689.673 3.01	749.56 2.94	688.86
55	5.74	147.17 7.32	771.558 3.01	920.28 2.79	605.18
56	7.54	4.04 86.80	586.516 3.02	592.10 3.06	716.40
57	9.22	92.03 7.55	479.098 3.02	573.20 2.82	604.67
58	8.09	7.05 51.10	546.685 3.02	556.72 3.06	710.36
59	10.05	132.81 5.66	439.282 3.02	573.67 2.70	554.36
60	10.86	5.00 59.76	406.294 3.02	412.84 3.09	707.56
61	11.26	3.61 78.87	391.665 3.02	395.83 3.10	709.48
62	12.43	109.93 5.87	354.677 3.03	465.48 2.71	549.66
63	13.91	2.68 93.88	316.620 3.03	321.43 3.12	706.76
64	14.49	1.05 225.9	303.757 3.03	303.22 3.16	714.32
65	14.92	1.41 167.3	294.916 3.03	296.95 3.15	710.43
66	15.56	115.41 5.10	282.598 3.03	397.84 2.63	509.54
67	16.25	114.81 5.01	270.471 3.03	385.89 2.62	502.81
68	17.16	3.46 66.64	255.868 3.04	259.27 3.16	704.90
69	16.90	105.94 5.14	259.953 3.04	366.89 2.64	512.99
70	18.01	103.01 5.06	243.801 3.04	346.97 2.63	503.62
71	18.68	0.99 211.4	234.895 3.04	235.65 3.19	711.96
72	4.84	433.24 4.46	916.209 3.01	1351.84 2.50	489.80
73	4.62	400.88 4.76	959.981 3.01	1360.42 2.55	507.81
74	4.77	14.09 37.46	929.908 3.01	946.94 3.02	703.26
75	4.64	273.21 5.81	956.046 3.01	1231.38 2.68	554.37
76	4.60	336.62 5.22	965.472 3.01	1301.03 2.61	530.92
77	4.77	2.98 144.5	929.908 3.01	934.36 3.04	711.07
78	4.59	5.59 83.08	967.364 3.01	974.29 3.03	708.32
79	4.69	318.28 5.33	945.608 3.01	1263.58 2.63	536.05
80	4.59	363.76 5.02	966.711 3.01	1335.51 2.58	519.32
81	4.72	10.42 48.04	940.214 3.01	954.73 3.02	704.17
82	5.08	13.45 37.66	872.585 3.01	888.21 3.02	698.91
83	4.65	3.98 112.1	954.413 3.01	959.64 3.04	707.92
84	4.72	308.30 5.40	940.003 3.01	1248.80 2.63	538.34
85	4.72	346.86 5.08	986.519 3.01	1287.98 2.59	522.39
86	4.73	5.70 80.35	938.220 3.01	945.46 3.04	707.75
87	4.85	238.49 6.11	915.138 3.01	1153.33 2.71	567.01
88	4.73	281.18 5.67	937.375 3.01	1216.71 2.67	548.23
89	4.56	362.33 5.05	972.213 3.01	1336.15 2.59	519.03
90	4.71	7.51 63.36	942.429 3.01	949.39 3.04	707.66
91	4.80	266.38 5.79	924.702 3.01	1192.10 2.68	553.53
92	5.07	8.19 56.71	873.917 3.01	883.32 3.04	705.78
93	4.88	3.30 129.8	910.109 3.01	913.48 3.04	707.32
94	4.34	13.46 40.72	1021.19 3.01	1037.81 3.02	696.87
95	4.46	304.73 5.59	894.079 3.01	1300.60 2.65	545.37
96	4.59	482.67 4.43	966.711 3.01	1433.55 2.49	484.55
97	4.51	421.99 4.69	984.355 3.01	1408.04 2.53	499.49
98	4.72	404.70 4.68	940.003 3.01	1348.59 2.53	500.13
99	5.01	5.12 85.96	885.417 3.01	892.84 3.04	707.50
100	5.79	175.61 6.61	764.870 3.01	941.97 2.75	581.10
101	6.47	7.09 56.84	684.011 3.01	693.63 3.05	699.93
102	6.59	115.26 7.84	674.653 3.01	788.37 2.82	608.31
103	7.26	7.41 51.63	609.115 3.02	618.10 3.05	702.49
104	8.04	5.34 65.36	550.228 3.02	556.26 3.07	702.39
105	7.78	130.52 6.72	567.982 3.01	698.25 2.77	581.50

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	FLAG
106	8.95	98.96	7.34	493.977	3.02	593.13	2.81	592.39	
107	8.80	120.86	6.26	450.463	3.02	572.90	2.73	561.53	
108	10.75	7.29	43.09	410.389	3.02	418.93	3.08	698.99	
109	10.62	63.45	8.80	416.015	3.02	479.49	2.89	619.17	
110	12.24	4.25	65.22	360.474	3.02	365.55	3.10	703.63	
111	12.28	132.03	5.32	358.883	3.03	491.39	2.65	522.80	
112	13.02	2.32	111.1	338.469	3.03	339.80	3.13	707.45	
113	13.21	0.65	378.7	334.011	3.02	336.10	3.13	716.74	
114	13.81	110.44	5.55	318.790	3.03	445.59	2.63	531.53	
115	13.80	125.10	5.17	319.168	3.03	445.59	2.63	531.53	
116	4.53	495.76	4.30	978.230	3.01	1477.84	2.47	478.88	
117	4.70	480.12	4.29	944.015	3.01	1426.57	2.47	472.41	
118	4.56	318.69	5.40	873.090	3.01	1293.83	2.63	540.62	
119	4.87	11.15	44.72	909.520	3.01	919.73	3.03	707.39	
120	5.89	6.43	64.78	752.172	3.01	759.11	3.05	708.42	
121	6.74	206.19	5.61	656.638	3.01	863.67	2.68	543.89	
122	6.80	247.00	5.07	651.700	3.01	899.77	2.60	519.99	
123	7.48	180.83	5.72	591.112	3.02	773.15	2.68	548.16	
124	8.47	3.75	87.72	521.905	3.02	527.29	3.07	711.13	
125	8.16	3.88	86.60	542.092	3.02	545.94	3.07	710.94	
126	9.51	3.26	84.34	464.607	3.02	468.94	3.08	710.50	
127	10.21	3.10	95.36	432.351	3.02	435.05	3.10	709.24	
128	10.88	130.60	5.68	405.450	3.02	536.57	2.69	543.92	
129	11.42	178.51	4.66	386.136	3.02	585.11	2.55	492.90	
130	11.84	149.55	5.05	372.332	3.02	521.43	2.61	511.31	
131	12.40	99.43	6.22	355.462	3.03	454.26	2.75	562.18	
132	13.42	105.93	5.77	328.365	3.03	434.35	2.70	543.32	
133	13.50	1.78	140.3	328.179	3.03	329.53	3.13	709.71	
134	14.50	0.41	562.2	303.684	3.03	304.44	3.15	731.82	
135	14.54	1.12	212.1	302.771	3.03	303.87	3.15	718.04	
136	4.95	252.48	5.87	895.580	3.01	1151.48	2.68	563.42	
137	3.63	582.07	4.42	1223.72	3.00	1808.59	2.49	488.89	
138	4.61	4.18	107.6	962.721	3.01	967.12	3.04	671.07	
139	4.89	10.41	47.20	906.605	3.01	919.74	3.03	705.41	
140	4.84	7.21	64.73	915.382	3.01	927.88	3.03	709.51	
141	4.91	202.15	6.64	902.493	3.01	1105.28	2.75	585.04	
142	4.90	426.07	4.47	903.933	3.01	1330.45	2.50	490.24	
143	4.98	8.48	55.55	891.773	3.01	901.20	3.03	711.12	
144	5.02	10.85	45.06	883.448	3.01	896.58	3.03	708.45	
145	4.51	3.36	132.8	983.690	3.01	986.76	3.04	711.85	
146	4.84	290.68	5.50	915.382	3.01	1209.69	2.64	546.35	
147	4.85	320.14	5.23	914.107	3.01	1237.04	2.61	530.23	
148	4.84	304.31	5.37	915.796	3.01	1222.50	2.63	538.53	
149	4.71	7.08	66.62	942.005	3.01	949.60	3.04	707.80	
150	4.83	311.40	5.31	917.077	3.01	1230.48	2.62	536.25	
151	4.87	325.32	5.17	909.930	3.01	1235.95	2.61	528.02	
152	4.81	284.75	5.80	922.150	3.01	1189.35	2.68	557.40	
153	4.72	241.98	6.15	939.367	3.01	1182.06	2.71	569.25	
154	4.80	147.84	7.98	925.536	3.01	1072.51	2.82	617.87	
155	4.76	288.64	5.59	930.607	3.01	1217.48	2.66	548.03	
156	4.39	267.98	6.04	1011.57	3.01	1282.84	2.69	564.20	
157	4.22	3.00	152.4	1051.02	3.01	1056.28	3.04	711.15	

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED

90	13A	112	12A	135	13B
91	13B	113	12B	136	K3-2*C*11
92	K3-1*FUA	114	13A	137	K3-2*C*12
93	K3-1*FUB	115	13B	138	K3-2*C*1
94	K3-2*IUA	116	K3-2*B*11	139	2
95	K3-2*IUB	117	K3-2*B*12	140	3
96	K3-2*A*11	118	K3-2*B*1	141	4A
97	K3-2*A*12	119	2	142	4B
98	K3-2*A*1	120	3	143	5
99	2	121	4A	144	6A
100	3	122	4B	145	6B
101	4A	123	5	146	7
102	4B	124	6A	147	8
103	5	125	6B	148	9A
104	6A	126	7	149	9B
105	6B	127	8	150	10
106	7	128	9A	151	11
107	8	129	9B	152	12A
108	9A	130	10	153	12B
109	9B	131	11	154	13A
110	10	132	12A	155	13B
111	11	133	12B	156	K3-2*FUA
		134	13A	157	K3-2*FUB

BOTTLE LABELED
K3-1*A*6A HAS
K3-1*B*6A SOLN
K3-1*B*6A HAS
K3-1*A*6A SOLN

The sample weights are:

B-I*PHI*R*a

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE (g)
3	7.3533	8.3482	0.9949
3.5	7.3608	8.3502	0.9894
4	7.472	8.4655	0.9935
4.5	7.3327	8.3269	0.9942
5	7.3475	8.2843	0.9368
5.5	7.3885	8.3792	0.9907
6	7.2972	8.2879	0.9907
6.25	7.3223	8.3128	0.9905
6.5	7.3983	8.3901	0.9918
6.75	7.3807	8.3809	1.0002
7	7.4082	8.3929	0.9847
7.25	7.3865	8.3783	0.9918
7.5	7.4519	8.4444	0.9925
8	7.3663	8.3565	0.9902

B-I*PHI*R*b

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE (g)
3	7.3751	8.3647	0.9896
3.5	7.4035	8.3957	0.9922
4	7.3866	8.3746	0.988
4.5	7.4463	8.4282	0.9819
5	7.3453	8.3405	0.9952
5.5	7.3647	8.3566	0.9919
6	7.3625	8.3517	0.9892
6.25	7.4305	8.4236	0.9931
6.5	7.3625	8.3502	0.9877
6.75	7.3081	8.2928	0.9847
7	7.3462	8.3367	0.9905
7.25	7.3985	8.3918	0.9933
7.5	7.3598	8.3554	0.9956
8	7.368	8.3602	0.9922

K3-1*B*I

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE (g)
11	7.3492	8.3427	0.9935
12	7.3444	8.3385	0.9941
1	7.3592	8.3572	0.9980
2	7.3306	8.3277	0.9971
3	7.4172	8.4101	0.9929
4A	7.3054	8.2994	0.9940
4B	7.3528	8.3446	0.9918
5	7.3824	8.3741	0.9917
6A	7.4060	8.4018	0.9958
6B	7.3754	8.3773	1.0019
7	7.3755	8.3697	0.9942
8	7.3778	8.3660	0.9882
9A	7.4431	8.4372	0.9941
9B	7.3334	8.3292	0.9958
10	7.4125	8.4058	0.9933
11	7.3125	8.3066	0.9941
12A	7.4280	8.4236	0.9956
12B	7.4442	8.4347	0.9905
13A	7.3883	8.3840	0.9957
13B	7.3238	8.3114	0.9876

NOTE: K3-1*B*6A
CONTAINS K3-1*A*6A
SOLUTION

K3-1*A*I

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE (g)
IUA	7.4165	8.4144	0.9979
IUB	7.3752	8.3695	0.9943
11	7.3542	8.3491	0.9949
12	7.3953	8.3856	0.9903
1	7.3310	8.3280	0.9970
2	7.3480	8.3341	0.9861
3	7.3614	8.3504	0.9890
4A	7.3484	8.3429	0.9945
4B	7.2860	8.2802	0.9942
5	7.4059	8.3982	0.9923
6A	7.3444	8.3404	0.9960
6B	7.3887	8.3730	0.9843
7	7.3396	8.3356	0.9960
8	7.4112	8.4033	0.9921
9A	7.3800	8.3801	1.0001
9B	7.3735	8.3644	0.9909
10	7.3417	8.3292	0.9875
11	7.3615	8.3490	0.9875
12A	7.4033	8.3941	0.9908
12B	7.3256	8.3165	0.9909
13A	7.4610	8.4495	0.9885
13B	7.3694	8.3548	0.9854

NOTE: K3-1*A*6A
CONTAINS K3-1*B*6A
SOLUTION

K3-1*C*I

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE (g)
11	7.3237	8.3112	0.9875
12	7.4424	8.4365	0.9941
1	7.3756	8.3659	0.9903
2	7.2803	8.2739	0.9936
3	7.3742	8.3673	0.9931
4A	7.3480	8.3317	0.9837
4B	7.2881	8.2777	0.9896
5	7.4722	8.4595	0.9873
6A	7.3830	8.3708	0.9878
6B	7.3981	8.3867	0.9886
7	7.3404	8.3312	0.9908
8	7.3459	8.3387	0.9928
9A	7.4368	8.4211	0.9843
9B	7.3195	8.3136	0.9941
10	7.4057	8.3987	0.9930
11	7.4110	8.4017	0.9907
12A	7.3780	8.3686	0.9906
12B	7.3250	8.3158	0.9908
13A	7.4117	8.4081	0.9964
13B	7.3811	8.3646	0.9835
FUA	7.4105	8.4024	0.9919
FUB	7.3842	8.3727	0.9885

K3-2*A*1

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE(g)
IUA	7.3458	8.3364	0.9480
IUB	7.3386	8.3265	0.9518
11	7.3884	8.3819	1.0291
12	7.3747	8.3643	1.0187
1	7.3528	8.3378	0.8949
2	7.3456	8.3372	1.0202
3	7.4429	8.4300	1.0461
4A	7.3170	8.3105	0.9180
4B	7.3839	8.3733	1.0330
5	7.3925	8.3826	0.9493
6A	7.3403	8.3261	0.9680
6B	7.4333	8.4282	1.1202
7	7.3581	8.3512	0.9577
8	7.3080	8.2974	0.9522
9A	7.3935	8.3829	1.0242
9B	7.3452	8.3410	0.9990
10	7.3587	8.3505	0.9973
11	7.3420	8.3307	0.9887
12A	7.3532	8.3491	0.9959
12B	7.3869	8.3772	0.9903
13A	7.3082	8.2974	0.9892
13B	7.3802	8.3719	0.9917

K3-2*B*1

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE(g)
11	7.3254	8.3146	0.9892
12	7.3458	8.3367	0.9909
1	7.3976	8.3901	0.9925
2	7.4146	8.4052	0.9906
3	7.3808	8.3751	0.9943
4A	7.3601	8.3525	0.9924
4B	7.4151	8.4160	1.0009
5	7.4167	8.4146	0.9979
6A	7.4618	8.4541	0.9923
6B	7.4278	8.4148	0.9870
7	7.4043	8.4008	0.9965
8	7.3632	8.3583	0.9951
9A	7.4226	8.4214	0.9988
9B	7.3477	8.3403	0.9926
10	7.3352	8.3281	0.9929
11	7.3808	8.3746	0.9938
12A	7.3107	8.3036	0.9929
12B	7.3194	8.3137	0.9943
13A	7.3918	8.3813	0.9895
13B	7.3674	8.3606	0.9932

K3-2*C*1

I	WT. VIAL (g)	WT. SAMPLE + VIAL (g)	WT. SAMPLE(g)
11	7.3758	8.3696	0.9938
12	7.4048	8.3917	0.9869
1	7.3488	8.3394	0.9906
2	7.3481	8.3428	0.9947
3	7.3300	8.3210	0.9910
4A	7.3374	8.3307	0.9933
4B	7.4556	8.4484	0.9928
5	7.3895	8.3759	0.9864
6A	7.3067	8.2981	0.9914
6B	7.3845	8.3709	0.9864
7	7.4209	8.4165	0.9956
8	7.3639	8.3568	0.9929
9A	7.3543	8.3477	0.9934
9B	7.3585	8.3533	0.9948
10	7.4395	8.4363	0.9968
11	7.4221	8.4157	0.9936
12A	7.3356	8.3267	0.9911
12B	7.3253	8.3115	0.9862
13A	7.4293	8.4222	0.9929
13B	7.3672	8.3573	0.9901
FUA	7.3976	8.3862	0.9886
FUB	7.4206	8.4167	0.9961

3 March 1993 TD

Samples B-I * pH 8.50 (8.75) * b were processed and counted by α -Spec according to p224-225

15 March 1993 TD

Samples B-I * pH 8.25 (9.00) and B-I-C * pH 2.0 * a2 were processed and counted by α -Spec.

23 March 1993 TD

Samples B-I-C * pH 2.0 * b2 & B-I-C * pH 4.0 * a2 were processed and counted by alpha Spec

25 March 1993 TD

Sample B-I-C * pH 4.0 * b2 was processed & counted by α -Spec
The solution B-I-C * pH 6.0 * b2 was spilled and the solution lost.

30 March 1993 TD

prepared 100 ml 0.4M TTA-benzene by dissolving 6.71g TTA in a volumetric flask w/100 ml benzene.

Lot # 904729 . TTA

920979 benzene

The solution was stored in the volumetric flask that was covered w/Al foil.

Sample B-I-C * pH 6.0 * a2 was processed and counted by Alpha spectrometry.

2 April 1993 TD

SAMPLES B-I-C * pH 8.0 * a2 AND B-I-C * pH 9.5 * a2 WERE PROCESSED AND COUNTED USING ALPHA-SPEC.

6 April 1993 TD

Sample B-I-C * pH 8.0 * b2 was processed and counted by α -spectrometry.

6 April 1993 TD

1 L 0.1 M NaNO_3 was prepared by dissolving 8.4994 g NaNO_3 in ^{400 ml} 1 kg H_2O .

8 April 1993 TD

2 L 8N NH_4NO_3 / 0.1N HNO_3 were prepared by dissolving 2 640.5g portions of NH_4NO_3 in 0.1N HNO_3 in separate 1L volumetric flasks and diluting to the mark with 0.1N HNO_3 .

Prepared 4L 0.1N HNO_3 by ^{TD 4/5/92} in 2 2L portions by diluting 12.7 mL conc HNO_3 to 2000 mL with DI H_2O .

2 L 8N HCl were prepared by diluting 1322 mL concentrated HCl to 2000 mL with deionized H_2O .

9 April 1993 TD

Began Experiments W1-50 and W1-5.
The procedure can be found on the following pages.

The 50 ppb solution was made by diluting 40.00 g of ^{233}U stock soln (500 ppb) to 400g using 0.1M NaNO_3 .

The 5ppb solution was made by diluting 4.02g ^{100g} to 401.33g using 0.1M NaNO_3 ^{233}U stock soln (500 ppb).

TD

URANIUM SORPTION CONTROL TEST W1:
Uranium loss to container walls: Polypropylene vs Teflon

WRITTEN BY: R.T. PABALAN
REVISION NO.: 1

DATE WRITTEN: April 6, 1993
DATE REVISED: April 8, 1993

OBJECTIVE:

- To investigate the potential loss of uranium from solution to the container walls
- To compare uranium loss to polypropylene bottles versus uranium loss to teflon bottles

EQUIPMENT:

Gyratory shaker or constant temperature shaker bath
Liquid scintillation counter
ORION pH/mV/ISE/ $^{\circ}\text{C}$ meter
Combination pH electrode - *vinex electrode w/ 0.1N HNO_3 then DI H_2O*
Automatic temperature compensator probe
Analytical balance

SUPPLIES:

- pH buffer (pH = 4,7,9)
- 6 60-ml polypropylene (PP) bottle
- 6 60-ml teflon (FEP) bottles
- 2 500-ml FEP bottles
- reagent grade NaHCO_3
- 500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike
- 1 L 0.10 M NaNO_3 stock solution

PROCEDURE:

1. Solution W1-50

- Initial ΣU \approx 50 ppb
- Initial pH \approx 7.0; adjustment made with NaHCO_3
- Initial volume \approx 50 ml
- Ionic strength = 0.1 M NaNO_3
- Initial $[\text{Na}^+] = 0.1 \text{ M } \text{NaNO}_3 + [\text{NaHCO}_3]$ added
- $\text{pCO}_2 = \text{atmospheric} = 10^{-3.48} \text{ bar}$

a) In a pre-cleaned 500-ml teflon (FEP) bottle, prepare 400 g of 50 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 400 g by carefully taring 0.10 M NaNO_3 solution

The solutions were stored in 500ml Teflon (FEP) bottles labeled W1-50 or W1-5 stock soln.

into the bottle on a Mettler 4600 balance.

Within the same day, analyze 5 samples (sample names W1-50-IU i , where i is 1,2,3,4, or 5) to determine the starting U concentration using standard liquid scintillation methods (5 ml Ultima + 0.5 ml 0.02 N HNO₃ + 0.5 g sample). If possible transfer the 0.5 g sample into the scintillation vial by pouring directly from the 500-ml FEP bottle instead of using a pipet. Note that the exact weight of the sample must be measured.

Adjust the pH of the remaining solution to about 7.0 by adding 0.0027 g NaHCO₃ to the solution. Mix well, then place the bottle (covered with Kimwipe) on a gyratory shaker (~100-120 rpm). Monitor the pH for several days until it remains constant.

b) Into each of 3 60-ml PP bottles labeled W1-50-PP i and 3 60-ml FEP bottles labeled W1-50-FEP i [where i is the mixture number 1,2 or 3], tare 50±2 g of the 50 ppb uranium solution. Cover each bottle with a Kimwipe, then place the bottles on a gyratory shaker.

Within the same day, use the remaining solution to analyze the initial U concentration by standard liquid scintillation counting procedures (5 ml Ultima cocktail + 0.5 ml 0.02 N HNO₃ + 0.5 g sample). Analyze 5 samples (sample names W1-50-IU i , where i is 6,7,8,9, or 10).

c) After periods of one, two and three weeks, analyze three samples from each PP and FEP bottle by liquid scintillation counting.

After taking the last set of samples, measure the pH of each solution.

d) Compare uranium losses for PP and FEP bottles.

2. Solution W1-5

- Initial EU ≈ 5 ppb
- Initial pH ≈ 7.0; adjustment made with NaHCO₃
- Initial volume ≈ 50 ml
- Ionic strength = 0.1 m NaNO₃
- Initial [Na⁺] = 0.1 m NaNO₃ + [NaHCO₃] added
- pCO₂ = atmospheric = 10^{-3.48} bar

a) In a pre-cleaned 500-ml teflon (FEP) bottle, prepare 400 g of 5 ppb U solution by diluting 4 g of a 500 ppb stock solution (in 0.1 m NaNO₃ matrix; prepared previously from commercial 50 ppm ²³³U spike) to a total of 400 g by carefully taring 0.10 m NaNO₃ solution into the bottle on a Mettler 4600 balance.

Within the same day, analyze 5 samples (sample names W1-5-IU i , where i is 1,2,3,4, or 5) to determine the starting U concentration using standard liquid scintillation methods (5 ml Ultima + 0.5 ml 0.02 N HNO₃ + 0.5 g sample). If possible transfer the 0.5 g sample into the scintillation vial by pouring directly from the 500-ml FEP bottle instead of using a pipet. Note

10 5 samples were taken from each stock solution, acidified w/0.5 ml 0.02 M HNO₃ and prepared for liquid scintillation counting according to

that the exact weight of the sample must be measured.

Adjust the pH to about 7.0 by adding 0.0027 g NaHCO₃ to the solution. Mix well, then place the bottle (covered with Kimwipe) on a gyratory shaker (~100-120 rpm). Monitor the pH for several days until it remains constant.

b) Into each of 3 60-ml PP bottles labeled W1-5-PP i and 3 60-ml FEP bottles labeled W1-5-FEP i [where i is the mixture number 1,2 or 3], tare 50±2 g of the 50 ppb uranium solution. Cover each bottle with a Kimwipe, then place the bottles on a gyratory shaker.

Within the same day, use the remaining solution to analyze the initial U concentration by standard liquid scintillation counting procedures (5 ml Ultima cocktail + 0.5 ml 0.02 N HNO₃ + 0.5 g sample). Analyze 5 samples (sample names W1-5-IU i , where i is 6,7,8,9, or 10).

c) After periods of one, two and three weeks, analyze three samples from each PP and FEP bottle by liquid scintillation counting.

After taking the last set of samples, measure the pH of each solution.

d) Compare uranium losses for PP and FEP bottles.

PREPARATION:

1. Preclean:
 - 6 60-ml PP bottles
 - 6 60-ml FEP bottles
 - 2 500-ml FEP bottles
2. Prepare:
 - 1 L 0.10 m NaNO₃ stock solution

the procedure on p. 235. 0.5 mL samples (5 from each stock solution) were taken with a 500 µL fixed volume pipet. (EXCEPT FOR W1-50-IU1 & ... - IU2 - these were poured directly from the bottles.). The sample weights and vial numbers can be found in Table 1 on the next page.

The pH of the solutions was adjusted with 0.0027 g NaHCO₃.

Wt. used, W1-50 0.0027g
Wt. used, W1-5 0.0028g

The 60 ml bottles were covered with a kimwipe and placed on a gyratory shaker; the pH will be monitored periodically until constant.

TABLE 1- Wt. of samples taken for LiSA

VIAL #	CONTENTS	WT. VIAL + ACID (g)	WT. VIAL + SAMPLE (g)	WT. SAMPLE (g)
1	BACKGROUND	7.8480		
2	W1-50-IU1	7.9020	8.4010	0.4990
3	W1-50-IU2	7.8856	9.1854	1.2998
4	W1-50-IU3	7.8626	8.3627	0.5001
5	W1-50-IU4	7.8084	8.3108	0.5024
6	W1-50-IU5	7.8821	8.3835	0.5014
7	W1-5-IU1	7.8466	8.3484	0.5018
8	W1-5-IU2	7.9312	8.4330	0.5018
9	W1-5-IU3	7.8674	8.3691	0.5017
10	W1-5-IU4	7.9218	8.4220	0.5002
11	W1-5-IU5	7.8306	8.3265	0.4959

The sample in Vial 3 was taken by pouring directly from the bottle. All others were taken with a 500 μ L Eppendorf pipet

13 April 1993 TD 1416

The pH of ^WMET-50 and ^WMET-5 were measured ^{TD 4/13/93}

SAMPLE	pH/T(°C)
^{4/13/93} TD ^W MET-50	6.40/22.7
^{4/13/93} TD ^W MET-5	6.79/22.7

The electrode was rinsed with 0.1N HNO₃ and then deionized H₂O to try and minimize cross contamination.

2L 1.0N HCl was prepared by mixing 1834 mL Ultrapure Water and 166 mL concentrated HCl in the 2L storage bottle

Lot # FL-05-1290

2L 2.0N HNO₃ was prepared by mixing 1750 mL Ultrapure H₂O and 253 mL concentrated HNO₃ in the 2L storage bottle

2L 1.0N HNO₃ was prepared by mixing 1873 mL Ultrapure H₂O and 127 mL concentrated HNO₃ in the 2L storage bottle.

Lot # FL-04-0390

15 April 1993 TD 1310

The pH of ^WMET-50 and ^WMET-5 was remeasured ^{TD 4/15/93}

SAMPLE	pH/T(°C)	pH/T(°C)
^{4/15/93} TD ^W MET-50	6.23/20.5	6.40/20.6
^{4/15/93} TD ^W MET-5	7.23/21.4°	6.76/20.7

The electrode was rinsed first with 0.1N HNO₃ and then with Ultrapure H₂O to minimize cross contamination.

NOTE: There was some instability with the temperature readings from the automatic temperature compensator probe, with temperature rising as high as ~55°C. Therefore each pH was measured twice to ^{TD 4/15/93}verify the readings. The meter was recalibrated between readings. Since there was a ^{TD 4/15/93}large difference, the solutions will be allowed to equilibrate for a while longer.

15 April 1993 ID

Experiment W2 (see below) was begun.

URANIUM SORPTION CONTROL TEST W2:Uranium loss to Teflon container or to Eppendorf pipet or to syringe filter: pH dependenceWRITTEN BY: R.T. PABALAN
REVISION NO.: 0DATE WRITTEN: April 8, 1993
DATE REVISED:

OBJECTIVE:

- To investigate the potential loss of uranium from solution to the Teflon container walls or to the Eppendorf pipet or the syringe filter
- To investigate the pH dependence of these uranium losses

EQUIPMENT:

Gyratory shaker or constant temperature shaker bath
Liquid scintillation counter
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance

SUPPLIES:

- pH buffer (pH = 2,4,7,9)
7 60-ml teflon (FEP) bottles (to contain experimental solutions)
1 500-ml FEP bottles (to contain W2-50-IU solution)
reagent grade NaHCO₃ (for adjusting solution pH)
stock solution of 0.1 M HNO₃ (for adjusting solution pH)
stock solution of 0.01 M HNO₃ (for adjusting solution pH)
stock solution of 0.02 M HNO₃ (for acidifying liquid scintillation samples)
500 ppb U stock solution prepared from 50 ppm ²³³U commercial spike
0.5 L 0.10 M NaNO₃ stock solution
1 0.5 ml Eppendorf pipet (for taking 0.5 ml samples and for adding 0.5 ml HNO₃ solution to liquid scintillation samples)

PROCEDURE:

1. Solution W2-50

- Initial $\Sigma U \approx 50$ ppb
- Initial pH ≈ 3 to 9, every 1 pH unit; adjustments made with HNO₃ or NaHCO₃
- Initial volume ≈ 50 ml
- Ionic strength = 0.1 M NaNO₃
- Initial [Na⁺] = 0.1 M NaNO₃ + [NaHCO₃] added
- pCO₂ = atmospheric = 10^{-3.48} bar

a) In a pre-cleaned 500-ml teflon (FEP) bottle labeled W2-50, prepare 400 g of 50 ppb U solution by diluting 40 g of a 500 ppb stock solution (in 0.1 M NaNO₃ matrix; prepared previously from commercial 50 ppm ²³³U spike) to a total of 400 g by carefully taring 0.10 M NaNO₃ solution into the bottle on a Mettler 4600 balance.

Within the same day, analyze 5 samples (sample names W2-50-IU_i, where *i* is 1,2,3,4 or 5) to determine the starting U concentration using standard liquid scintillation methods (5 ml Ultima + 0.5 ml 0.02 N HNO₃ + ~0.5 g sample). If possible, transfer the ~0.5 g samples into the scintillation vials by pouring directly from the 500-ml bottle instead of using a pipet. Note that the exact weight of the sample must be measured.

b) Tare 50 ± 2 g of the solution into each of 7 teflon (FEP) bottles labeled W2-50-pH_i (where *i* is the target initial pH of the solution equal to 3, 4, 5, 6, 7, 8, or 9). Adjust the pH of each solution by adding HNO₃ or NaHCO₃ in the amounts given in Table 1. Mix well, then place the bottles (covered with Kimwipe) on a gyratory shaker (~100-120 rpm).

c) After periods of one, two and three weeks, analyze samples taken from each bottle by three different methods:

a) Direct pouring from the teflon bottle.

b) Using 0.5 ml Eppendorf pipet

c) Using a syringe filter (DynaGard 0.2 μ m, 0.8 cm²). Use new filters for each sample. Use 1 ml capacity polypropylene syringe. Leave the filter on during both sample withdrawal and sample dispensing steps.

Take two samples using each method. Use standard liquid scintillation methods to analyze ²³³U concentrations (5 ml Ultima scintillation cocktail + 0.5 ml 0.02 N HNO₃ + ~0.5 g sample). Note that the exact weight of the sample must be measured.

After taking each set of samples, measure the pH of each experimental solution W2-50-pH_i. Take care to minimize the measurement time and the pH electrode surface in contact with the solution (to minimize adsorption of U onto the glass electrode).

Note: If significant losses are observed due to the filtration step, additional samples may be taken with the syringe filter on only during the sample withdrawal step. A larger surface area filter may also be used to check if larger surface areas will cause larger uranium losses.

d) Compare uranium losses as a function of pH for the three methods used.

e) Take 5 additional samples (sample names W2-50-IU_i, where *i* is equal to 6,7,8,9, or 10). from the solution leftover from a) and analyze the U concentration by liquid scintillation counting.

PREPARATION:

- 1. Preclean:
 - 7 60-ml FEP bottles
 - 1 500-ml FEP bottles
- 2. Prepare:
 - 0.5 L 0.10 m NaNO₃ stock solution
 - stock solution of 0.1 m HNO₃
 - stock solution of 0.01 m HNO₃
 - stock solution of 0.02 m HNO₃

Table 1. Amount of reagent grade HNO₃ or NaHCO₃ to add to 50 ml 0.1 m NaNO₃ 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 assuming the solutions are in equilibrium with atmospheric CO₂(g).

Solution	Volume HNO ₃ Needed, Drops (.0394 mL/drop)	Molarity of HNO ₃ Used
3.00	15 (0.605 mL)	0.1
4.00	2 (0.06 mL)	0.1
5.00	2 (0.055 mL)	0.01
Solution pH	Amount of NaHCO ₃ to be Added, Grams	
6.00	0.000059	
7.00	0.000332	
8.00	0.003082	
9.00	0.036353	

500g 0.1 m NaNO₃ was prepared by dissolving
4.2495 g NaNO₃ in 500g H₂O.

wt. NaNO₃ used: 4.2497g
lot # 7808 KCCL

19 April 1993 2015 TD

The pH of both W1-50(s) were remeasured

sample	pH/T(°C)
W1-50	6.56/-TD 6.44/24.2
W1-5	6.78/24.2 TD 4/19/93

20 April 1993
The following are the results of the Liquid Scintillation Analysis
of the W1-50 & W1-5 initial solutions. The column S#
corresponds with the vial numbers on p.248, Table 1.

Protocol #: 5 Name:U-233 3% 2 sigma 15-Apr-93 21:44
Region A: LL-UL= 0.0-100. Ler= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Ler= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Ler= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS	FLAG
1	999.98	18.64 1.47	2.916 3.70	27.69 1.20	136.46	B
2	8.19	3.34 98.35	540.307 3.01	545.33 3.07	733.65	
3	3.22	6.83 82.44	1380.31 3.00	1388.46 3.02	676.68	
4	8.28	1.78 177.6	534.040 3.02	536.92 3.08	737.27	
5	8.17	1.56 202.3	541.148 3.02	543.42 3.08	730.97	
6	8.21	2.92 111.2	538.740 3.02	541.86 3.07	736.28	
7	90.67	0.54 176.4	46.097 3.20	46.80 3.94	727.55	
8	89.63	0.18 545.4	46.666 3.20	46.99 3.95	740.51	
9	90.30	0.06 1642.	46.320 3.20	46.33 3.97	740.47	
10	88.43	0.74 132.5	47.350 3.19	48.45 3.89	732.29	
11	88.91	0.00 0.00	47.067 3.19	46.82 3.97	748.02	

W1-50 and W1-5 Initial U(233) concentrations (before pH adjustment)

SAMPLE NAME	CPMB	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
W1-50-IU1	540.307	0.4990	1082.7796	1.25210121E+14	2.07887E-10	48.4458
W1-50-IU2	1380.310	1.2998	1061.9403	1.22800317E+14	2.03886E-10	47.5134
W1-50-IU3	534.040	0.5001	1067.8664	1.234856E+14	2.05023E-10	47.7786
W1-50-IU4	541.148	0.5024	1077.1258	1.24556332E+14	2.06801E-10	48.1929
W1-50-IU5	538.740	0.5014	1074.4715	1.24249393E+14	2.06292E-10	48.0741
W1-5-IU1	46.097	0.5018	91.8633	10622858349418	1.76372E-11	4.1102
W1-5-IU2	46.666	0.5018	92.9972	10753981988719	1.78549E-11	4.1609
W1-5-IU3	46.320	0.5017	92.3261	10676375370285	1.7726E-11	4.1309
W1-5-IU4	47.350	0.5002	94.6621	10946510071573	1.81745E-11	4.2354
W1-5-IU5	47.067	0.4959	94.9123	10975436324312	1.82225E-11	4.2466

TD 6/15/94
* See verification on page 142-143 of GC-011. (CNWRA controlled
RD 6/14/94 143-144 wpy 091)

20 April 1993 TD

The pH of the W1-50 & W1-5 solutions were remeasured

SAMPLE	pH/T(°C)
W1-50	6.96/21.6 ^{TD 4/20/93} 6.80/21.7
W1-5	6.45/21.7

The pH has stabilized enough to proceed. 50 ± 2 g samples were poured out into bottles labeled W1-50(or 5)-FEP-i or W1-50(or 5)-PP-i, $i = 1, 2, 3$. These were covered with a kimwipe and placed on a gyratory shaker at ~ 120 rpm.

Sample	Wt. Soln. (g)
W1-50-FEP-1	50.96
2	49.80
3	49.73
- PP-1	50.02
2	50.79
3	50.38
W1-5-FEP-1	50.47
2	49.68
3	49.69
- PP-1	49.40
2	50.52
3	49.94

5 0.5 ml samples were taken from each sample for LSA. The sample weights are on the following page. Each was acidified with 500 μ L 0.02 N HNO_3 . 5 ml LSA "cocktail" was added to each. Samples were taken with a 500 μ L fixed volume Eppendorf pipet.

VIAL #	SAMPLE NAME	WT. VIAL+ACID (g)	WT. VIAL, ACID, SAMPLE (g)	WT. SAMPLE (g)
1	BACKGRND	7.8617	N.A.	
2	W1-50-IU6	7.9088	8.4127	0.5039
3	W1-50-IU7	7.8929	8.3988	0.5059
4	W1-50-IU8	7.8523	8.3582	0.5059
5	W1-50-IU9	7.9122	8.4175	0.5053
6	W1-50-IU10	7.8614	8.3915	0.5301
7	W1-5-IU6	7.8439	8.3449	0.5010
8	W1-5-IU7	7.8307	8.3357	0.5050
9	W1-5-IU8	7.9415	8.4458	0.5043
10	W1-5-IU9	8.0041	8.5073	0.5032
11	W1-5-IU10	7.8893	8.3931	0.5038

22 APRIL 1993 TD

EXPERIMENT W2

400g of 50ppb ^{233}U was prepared by diluting 40.30g of the 500ppb ^{233}U stock solution to 401.38g using 0.1M NaNO_3 .

7 50g aliquots were taken and placed in pre-cleaned, labeled FEP bottles. The pH was adjusted according to Table 1 on pg. 252. The weights of the NaHCO_3 used were as follows:

W2-50- pH6.00	0.00006 g
W2-50- pH7.00	0.00051 g
W2-50- pH8.00	0.0035 g
W2-50- pH9.00	0.0366 g

NaHCO_3 lot #: 897186A

The bottles were covered ^{TD 4/22/93} and with a kimwipe and placed on a gyratory shaker.

5 - 0.5 ml samples of the initial solution were taken for liquid scintillation analysis. They were acidified with 0.5 ml 0.02M HNO_3 and 5 ml "cocktail" was added to each.

The weights of the samples are as follows:

VIAL #	SAMPLE NAME	WT VIAL/ACID (g)	WT VIAL/ACID/SAMPLE (g)	WT SAMPLE (g)
12	W2-50-IU-1	7.9126	8.4103	0.4977
13	W2-50-IU-2	7.8987	8.4000	0.5013
14	W2-50-IU-3	7.8631	8.3654	0.5023
15	W2-50-IU-4	7.9387	8.4416	0.5029
16	W2-50-IU-5	7.9174	8.4198	0.5024

these were placed behind the W1-50(5)-IU i
Samples prepared previously (i=6-10).

26 April 1993 TD

The samples taken were counted by liquid Scintillation. The
data from the counting are presented below.

Protocol #: 5 Name: U-233 3% 2 sigma 25-Apr-93 11:28
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.87 1.46	2.836 3.76	27.73 1.20	130.73 B
2	8.71	2.14 146.0	507.497 3.02	509.24 3.08	696.55
3	8.80	1.81 170.3	502.278 3.02	505.11 3.08	694.41
4	8.64	4.97 67.10	512.210 3.01	518.45 3.07	690.59
5	8.77	4.73 69.63	504.005 3.02	511.04 3.07	693.21
6	8.50	4.30 77.03	521.164 3.01	526.27 3.07	687.66
7	88.29	0.00 0.00	47.509 3.19	47.67 3.94	700.10
8	88.84	0.31 316.5	47.186 3.19	47.69 3.93	695.63
9	85.22	0.05 1849.	49.323 3.18	49.23 3.92	703.85
10	86.92	0.65 152.0	48.291 3.18	49.09 3.89	692.77
11	90.33	0.00 0.00	46.372 3.19	46.64 3.96	698.73
12	8.40	1.48 210.8	526.212 3.02	529.53 3.08	695.34
13	8.19	1.52 208.9	540.265 3.01	543.45 3.07	697.04
14	8.03	0.93 340.3	550.589 3.02	555.83 3.07	692.60
15	8.31	0.00 0.00	532.182 3.02	532.32 3.09	694.33
16	8.02	2.82 117.0	551.403 3.02	555.94 3.07	693.71

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

The column S# corresponds to the vial number
in the tables on p. 255 and 256. The # of counts
were converted to ppb U and those values can
be found in the table below.

SAMPLE NAME	CPMB	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
W1-50-IU6	507.497	0.5039	1007.1383	1.16463143E+14	1.93364E-10	45.0615
W1-50-IU7	502.278	0.5059	992.8405	1.14809774E+14	1.90619E-10	44.4218
W1-50-IU8	512.210	0.5059	1012.4728	1.17080012E+14	1.94388E-10	45.3002
W1-50-IU9	504.005	0.5053	997.4372	1.15341324E+14	1.91501E-10	44.6274
W1-50-IU10	521.164	0.5301	983.1428	1.13688357E+14	1.88757E-10	43.9879
W1-5-IU6	47.509	0.5010	94.8283	10965729999118	1.82064E-11	4.2428
W1-5-IU7	47.186	0.5050	93.4376	10804910411135	1.79394E-11	4.1806
W1-5-IU8	49.323	0.5043	97.8049	11309929582291	1.87779E-11	4.3760
W1-5-IU9	48.291	0.5032	95.9678	11097494829994	1.84252E-11	4.2938
W1-5-IU10	46.372	0.5038	92.0445	10643808421676	1.76719E-11	4.1183
W2-50-IU1	526.212	0.4977	1057.2875	1.2226228E+14	2.02992E-10	47.3053
W2-50-IU2	540.265	0.5013	1077.7279	1.24625959E+14	2.06917E-10	48.2198
W2-50-IU3	550.589	0.5023	1096.1358	1.26754602E+14	2.10451E-10	49.0434
W2-50-IU4	532.182	0.5029	1058.2263	1.22370837E+14	2.03173E-10	47.3473
W2-50-IU5	551.403	0.5024	1097.5378	1.26916731E+14	2.1072E-10	49.1061

TD 6/4/99
See verification
on pages
115-119 of
U-233-1149
616-11 (CWRA
controlled copy
061)

27 April 1992 TD

A week has passed since the W1-50 and W1-5 solutions
were transferred into the PP and PEP bottles. 3 samples
will be taken from each for LS analyzation. The samples
will be labeled W1-50(5)-FEP(1PP)-Laborc, i=1,2,3.
Each sample was 500uL. They were each acidified with
500uL 0.02N HNO₃. 5mL scintillation cocktail was added
to each. Table 1 on the following page contains the sample
weights.

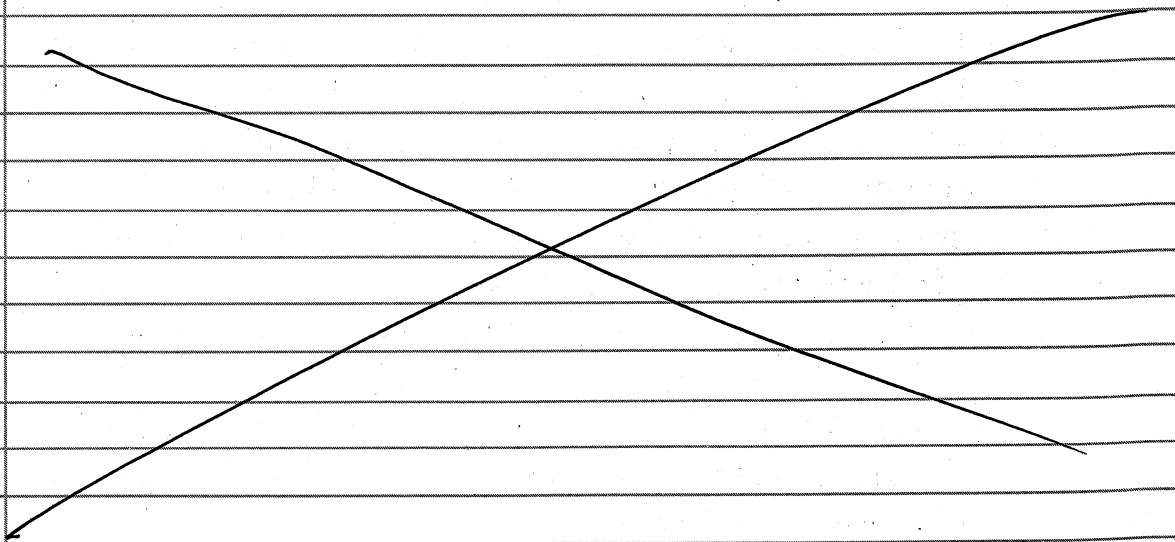


TABLE 1. SAMPLE WEIGHTS FOR THE FIRST SAMPLING OF W1

VIAL #	SAMPLE	WT. VIAL & ACID (g)	WT. VIAL, ACID, & SAMPLE (g)	WT. SAMPLE (g)
1	BLANK	---	---	---
2	W1-50-FEP-1	7.8892	8.3950	0.5058
3	W1-50-FEP-1	7.8683	8.3727	0.5044
4	W1-50-FEP-1	7.8884	8.3930	0.5046
5	W1-50-FEP-2	7.8766	8.3839	0.5073
6	W1-50-FEP-2	7.9126	8.4166	0.5040
7	W1-50-FEP-2	7.8618	8.3655	0.5037
8	W1-50-FEP-3	7.8682	8.3709	0.5027
9	W1-50-FEP-3	7.8644	8.3678	0.5034
10	W1-50-FEP-3	7.8995	8.4025	0.5030
11	W1-50-PP-1A	7.7716	8.2749	0.5033
12	W1-50-PP-1B	7.8544	8.3581	0.5037
13	W1-50-PP-1C	7.9155	8.4190	0.5035
14	W1-50-PP-2A	7.8606	8.3637	0.5031
15	W1-50-PP-2B	7.7482	8.2533	0.5051
16	W1-50-PP-2C	7.8542	8.3595	0.5053
17	W1-50-PP-3A	7.9819	8.4858	0.5039
18	W1-50-PP-3B	7.8598	8.3642	0.5044
19	W1-50-PP-3C	7.8152	8.3193	0.5041
20	W1-5-FEP-1A	7.8850	8.3938	0.5088
21	W1-5-FEP-1B	7.9382	8.4433	0.5051
22	W1-5-FEP-1C	7.9459	8.4504	0.5045
23	W1-5-FEP-2A	7.8543	8.3587	0.5044
24	W1-5-FEP-2B	7.8209	8.3250	0.5041
25	W1-5-FEP-2C	7.8606	8.3624	0.5018
26	W1-5-FEP-3A	7.9171	8.4237	0.5066
27	W1-5-FEP-3B	7.8649	8.3699	0.5050
28	W1-5-FEP-3C	7.9110	8.4151	0.5041
29	W1-5-PP-1A	8.0065	8.5102	0.5037
30	W1-5-PP-1B	7.8208	8.3238	0.5030
31	W1-5-PP-1C	7.8996	8.4024	0.5028
32	W1-5-PP-2A	7.7603	8.2653	0.5050
33	W1-5-PP-2B	7.8940	8.3972	0.5032
34	W1-5-PP-2C	7.8920	8.3945	0.5025
35	W1-5-PP-3A	7.8071	8.3072	0.5001
36	W1-5-PP-3B	7.8989	8.4021	0.5032
37	W1-5-PP-3C	7.9295	8.4353	0.5058

29 April 1993 TD

A week has passed since the W2-50 experiment was started. 6 samples were taken from each bottle, 2 by pouring directly from the experimental bottle into the LS vial; 2 using an Eppendorf pipet (500 μ L) and 2 using a 1ml syringe with a filter attached (see procedure on pg 251 for type of filter). The samples were labeled W2-pHi-P(or E or S)A(or B) i = 3.00-9.00, Δ pH \leq 1. P=pouring
E=Eppendorf
S=Syringe

The samples were prepared for LSA as before. pH was also measured. All data can be found in the table on the following page.

P=pouring, E=using Eppendorf, S=syringe. T for pH measurements=21.3 C

VIAL #	SAMPLE NAME	pH	WT VIAL (g)	WT VIAL+SAMPLE (g)	WT SAMPLE (g)
49	W2-50-pH3.00Pa	2.97	7.8881	8.4734	0.5853
50	W2-50-pH3.00Pb		7.9283	8.6962	0.7679
51	W2-50-pH3.00Ea		7.8273	8.3332	0.5059
52	W2-50-pH3.00Eb		7.8741	8.3818	0.5077
53	W2-50-pH3.00Sa		7.9064	8.3524	0.4460
54	W2-50-pH3.00Sb		7.8765	8.3418	0.4653
55	W2-50-pH4.00Pa	3.67	7.8890	8.2625	0.3735
56	W2-50-pH4.00Pb		7.8576	8.7812	0.9236
57	W2-50-pH4.00Ea		7.9079	8.4134	0.5055
58	W2-50-pH4.00Eb		7.9700	8.4763	0.5063
59	W2-50-pH4.00Sa		7.8629	8.3032	0.4403
60	W2-50-pH4.00Sb		7.9678	8.4153	0.4475
61	W2-50-pH5.00Pa	4.01	7.8709	8.5940	0.7231
62	W2-50-pH5.00Pb		7.8949	8.6212	0.7263
63	W2-50-pH5.00Ea		7.8798	8.3811	0.5013
64	W2-50-pH5.00Eb		7.8843	8.3881	0.5038
65	W2-50-pH5.00Sa		8.0030	8.4506	0.4476
66	W2-50-pH5.00Sb		7.8639	8.2895	0.4256
67	W2-50-pH6.00Pa	4.36	7.8713	8.2864	0.4151
68	W2-50-pH6.00Pb		7.8686	8.4190	0.5504
69	W2-50-pH6.00Ea		7.8955	8.4010	0.5055
70	W2-50-pH6.00Eb		7.8039	8.3100	0.5061
71	W2-50-pH6.00Sa		7.8725	8.3256	0.4531
72	W2-50-pH6.00Sb		7.8731	8.3206	0.4475
73	W2-50-pH7.00Pa	6.47	7.9608	9.0824	1.1216
74	W2-50-pH7.00Pb		7.9806	8.4241	0.4435
75	W2-50-pH7.00Ea		7.8703	8.3770	0.5067
76	W2-50-pH7.00Eb		7.9304	8.4373	0.5069
77	W2-50-pH7.00Sa		7.7936	8.2320	0.4384
78	W2-50-pH7.00Sb		7.9009	8.3580	0.4571
79	W2-50-pH8.00Pa	7.71	7.8893	8.4751	0.5858
80	W2-50-pH8.00Pb		7.9608	8.5779	0.6171
81	W2-50-pH8.00Ea		7.9057	8.4109	0.5052
82	W2-50-pH8.00Eb		7.9036	8.4093	0.5057
83	W2-50-pH8.00Sa		7.8795	8.3291	0.4496
84	W2-50-pH8.00Sb		7.9388	8.3876	0.4488
85	W2-50-pH9.00Pa	8.89	7.9058	8.4494	0.5436
86	W2-50-pH9.00Pb		7.8712	8.4604	0.5892
87	W2-50-pH9.00Ea		7.9443	8.4500	0.5057
88	W2-50-pH9.00Eb		7.7654	8.2702	0.5048
89	W2-50-pH9.00Sa		7.9589	8.3974	0.4385
90	W2-50-pH9.00Sb		7.9261	8.3777	0.4516

3 May 1993 TD

The samples prepared previously (27, 29 April 1993) have finished counting by liquid scintillation. The results of the counting, as well as the results of the ppb calculations follow on TD are on the following pages.
5/3/92

LSA results for first samplings of W1 & W2

Protocol #: 5 Name: U-233 3% 2 sigma 30-Apr-93 10:23
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.91 1.45	2.764 3.80	27.71 1.20	129.76 B
2	9.27	0.00 0.00	476.848 3.02	477.25 3.09	896.53
3	9.08	0.00 0.00	486.773 3.02	487.26 3.09	892.12
4	9.16	0.00 0.00	482.389 3.02	482.66 3.09	892.57
5	8.75	2.81 112.7	505.579 3.02	510.11 3.07	892.47
6	9.13	0.00 0.00	484.092 3.02	484.12 3.09	898.10
7	9.36	1.93 155.6	472.129 3.02	475.49 3.08	892.45
8	9.14	0.00 0.00	484.107 3.02	484.98 3.09	895.56
9	9.09	2.43 126.4	486.125 3.02	489.23 3.08	892.15
10	8.86	1.30 234.0	499.155 3.02	500.73 3.09	896.53
11	10.00	1.09 280.1	441.736 3.02	442.29 3.10	893.72
12	10.11	1.86 154.5	437.097 3.02	439.65 3.09	892.71
13	10.12	4.02 75.24	436.663 3.02	441.06 3.09	890.49
14	12.84	0.00 0.00	343.342 3.02	343.08 3.13	899.58
15	12.43	0.24 1041.	354.999 3.02	356.04 3.12	898.72
16	12.78	0.11 2301.	345.045 3.02	346.07 3.13	897.00
17	15.66	1.91 121.6	281.144 3.03	284.74 3.14	897.85
18	15.30	0.64 358.5	287.824 3.03	288.04 3.16	898.56
19	15.40	0.00 0.00	285.937 3.03	286.77 3.15	897.56
20	108.06	0.72 124.3	38.370 3.23	39.47 4.08	886.46
21	108.37	0.39 229.0	38.244 3.23	39.15 4.10	896.39
22	104.31	0.08 1066.	39.840 3.22	40.59 4.07	703.48
23	96.34	0.22 418.6	43.364 3.20	43.61 4.02	894.96
24	93.68	0.00 0.00	44.685 3.19	44.14 4.04	709.37
25	95.84	0.24 391.1	43.605 3.20	44.46 3.97	892.94
26	94.43	0.47 200.6	44.297 3.20	44.82 3.98	891.88
27	93.21	1.03 94.04	44.924 3.19	46.39 3.91	880.32
28	94.53	0.00 0.00	44.247 3.20	44.87 3.98	897.81
29	127.99	0.42 195.3	31.957 3.28	32.77 4.32	888.12
30	128.96	0.15 534.4	31.696 3.28	31.97 4.38	899.14
31	127.27	0.00 0.00	32.154 3.27	32.32 4.37	703.72
32	123.33	0.47 178.0	33.302 3.26	33.79 4.29	885.10
33	121.02	0.00 0.00	33.957 3.26	34.11 4.30	899.49
34	126.17	0.11 772.3	32.466 3.27	32.58 4.37	897.48
35	133.84	0.54 149.8	30.447 3.29	31.35 4.37	890.27
36	133.58	0.42 191.8	30.512 3.29	30.60 4.45	887.23
37	129.87	0.38 214.7	31.462 3.28	32.02 4.36	887.91
(11 missing vials)					
49	6.96	2.21 157.9	635.885 3.01	640.25 3.06	891.76
50	5.33	2.67 151.1	831.195 3.01	835.14 3.05	883.12
51	8.24	1.24 253.5	536.799 3.01	538.79 3.08	893.79
52	8.02	1.79 180.1	551.974 3.01	553.96 3.08	895.09
53	9.65	0.00 0.00	458.065 3.02	456.74 3.10	704.73
54	8.88	0.00 0.00	498.024 3.02	498.30 3.09	701.21
55	11.02	3.51 81.72	400.593 3.02	404.59 3.10	701.67
56	4.46	2.17 200.9	994.770 3.01	999.64 3.04	870.88
57	7.98	1.27 251.7	554.379 3.01	556.12 3.08	896.65
58	8.32	0.00 0.00	531.972 3.01	532.74 3.08	701.76
59	9.91	0.00 0.00	445.874 3.02	446.86 3.10	704.36

S# corresponds to the vial number in the Tables on 258, 259.

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
60	9.47	1.79 165.9	466.613 3.02	471.02 3.08	701.09
61	5.73	0.64 579.8	773.501 3.01	775.08 3.05	684.02
62	5.93	0.65 556.9	746.983 3.01	749.86 3.05	684.43
63	8.46	1.66 188.6	523.477 3.01	526.90 3.07	696.90
64	8.31	3.11 104.9	532.013 3.02	536.79 3.07	692.48
65	10.39	0.92 302.0	425.147 3.02	428.49 3.09	698.55
66	10.31	0.59 469.9	428.371 3.02	430.00 3.10	697.56
67	9.90	2.81 105.9	446.125 3.02	448.65 3.09	696.92
68	7.68	0.62 513.1	576.142 3.01	577.36 3.08	688.97
69	8.49	3.47 93.86	521.264 3.01	525.88 3.07	691.91
70	8.53	0.00 0.00	518.221 3.02	520.35 3.08	693.58
71	10.22	4.09 73.71	432.265 3.02	437.45 3.09	691.50
72	10.06	1.17 242.2	438.985 3.02	440.48 3.10	695.02
73	4.23	3.55 130.0	1048.77 3.01	1051.96 3.04	656.71
74	10.60	1.84 145.2	416.764 3.02	420.02 3.10	698.81
75	9.37	1.48 200.6	471.836 3.02	474.21 3.09	691.37
76	9.44	3.44 89.71	468.105 3.02	471.65 3.08	690.40
77	12.00	2.84 95.21	367.903 3.02	371.29 3.11	691.03
78	12.71	3.20 82.85	347.118 3.02	349.94 3.12	692.43
79	6.99	1.55 221.4	633.431 3.01	634.80 3.07	688.14
80	6.68	5.34 71.49	663.254 3.01	668.84 3.05	685.27
81	8.10	2.70 121.5	545.878 3.02	547.47 3.08	688.39
82	7.98	3.27 102.2	554.253 3.01	556.75 3.07	689.43
83	10.52	0.96 287.9	419.669 3.02	420.48 3.11	692.60
84	9.26	2.47 123.3	477.258 3.02	479.74 3.09	696.74
85	7.72	2.08 159.3	573.272 3.01	576.69 3.07	690.19
86	6.86	0.04 7746.	645.924 3.01	645.03 3.07	687.14
87	7.82	0.91 349.8	565.650 3.01	568.32 3.07	692.56
88	8.05	2.33 139.6	549.534 3.01	552.78 3.07	685.51
89	9.63	2.48 120.5	459.230 3.02	462.73 3.09	693.11
90	9.18	2.88 107.4	481.332 3.02	483.94 3.09	690.24

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED

C14 CHI SQUARE IPA DATA PROCESSED

H3 IPA DATA PROCESSED

H3 CHI SQUARE IPA DATA PROCESSED

BKG IPA DATA PROCESSED

W1-50 AND W1-5: U(233) concentrations at the first sampling time (27 April 1993)

SAMPLE NAME	CPMB	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
W1-50-FEP-1A	476.848	0.5058	942.7600	1.0901858E+14	1.81004E-10	42.1811
W1-50-FEP-1B	486.773	0.5044	965.0535	1.1159656E+14	1.85284E-10	43.1785
W1-50-FEP-1C	482.389	0.5046	955.9830	1.1054766E+14	1.83543E-10	42.7727
W1-50-FEP-2A	505.579	0.5073	996.6075	1.1524539E+14	1.91342E-10	44.5903
W1-50-FEP-2B	484.092	0.5040	960.5000	1.1107E+14	1.8441E-10	42.9748
W1-50-FEP-2C	472.192	0.5037	937.4469	1.0840419E+14	1.79984E-10	41.9433
W1-50-FEP-3A	484.107	0.5027	963.0137	1.1136068E+14	1.84892E-10	43.0873
W1-50-FEP-3B	486.125	0.5034	965.6834	1.1166939E+14	1.85405E-10	43.2067
W1-50-FEP-3C	499.155	0.5030	992.3559	1.1475373E+14	1.90526E-10	44.4001
W1-50-PP-1A	441.736	0.5033	877.6793	1.014928E+14	1.68509E-10	39.2692
W1-50-PP-1B	437.097	0.5037	867.7725	1.003472E+14	1.66607E-10	38.8260
W1-50-PP-1C	436.663	0.5035	867.2552	1.0028738E+14	1.66507E-10	38.8028
W1-50-PP-2A	343.342	0.5031	682.4528	7.891726E+13	1.31026E-10	30.5344
W1-50-PP-2B	354.999	0.5051	702.8291	8.1273534E+13	1.34939E-10	31.4460
W1-50-PP-2C	345.045	0.5053	682.8518	7.8963397E+13	1.31103E-10	30.5522
W1-50-PP-3A	281.144	0.5039	557.9361	6.4518438E+13	1.0712E-10	24.9632
W1-50-PP-3B	287.824	0.5044	570.6265	6.5985925E+13	1.09557E-10	25.5310
W1-50-PP-3C	285.937	0.5041	567.2228	6.5592328E+13	1.08903E-10	25.3787
W1-5-FEP-1A	38.370	0.5088	75.4127	8720554118327	1.44788E-11	3.3741
W1-5-FEP-1B	38.244	0.5051	75.7157	8755588175054	1.45369E-11	3.3877
W1-5-FEP-1C	39.840	0.5045	78.9693	9131824243602	1.51616E-11	3.5333
W1-5-FEP-2A	43.364	0.5044	85.9715	9941539510989	1.6506E-11	3.8465
W1-5-FEP-2B	44.685	0.5041	88.6431	1.0250486E+13	1.70189E-11	3.9661
W1-5-FEP-2C	43.605	0.5018	86.8972	1.0048588E+13	1.66837E-11	3.8880
W1-5-FEP-3A	44.297	0.5066	87.4398	1.0111335E+13	1.67879E-11	3.9122
W1-5-FEP-3B	44.924	0.5050	88.9584	1.0286945E+13	1.70794E-11	3.9802
W1-5-FEP-3C	44.247	0.5041	87.7743	1.0150011E+13	1.68521E-11	3.9272
W1-5-PP-1A	31.957	0.5037	63.4445	7336576271309	1.21809E-11	2.8386
W1-5-PP-1B	31.696	0.5030	63.0139	7286783364478	1.20983E-11	2.8194
W1-5-PP-1C	32.154	0.5028	63.9499	7395016093095	1.2278E-11	2.8613
W1-5-PP-2A	33.302	0.5050	65.9446	7625675550198	1.26609E-11	2.9505
W1-5-PP-2B	33.957	0.5032	67.4821	7803475429005	1.29561E-11	3.0193
W1-5-PP-2C	32.466	0.5025	64.6090	7471229948296	1.24045E-11	2.8907
W1-5-PP-3A	30.447	0.5001	60.8818	7040233083395	1.16889E-11	2.7240
W1-5-PP-3B	30.512	0.5032	60.6359	7011798518415	1.16417E-11	2.7130
W1-5-PP-3C	31.462	0.5058	62.2025	7192947438265	1.19425E-11	2.7831

TO 6/14/94
 See verification
 on pages 142-143
 of GC-011.
 (CINRA CONTROLLED)
 COPY 081

W2-50: U(233) concentrations at the first sampling time (29 April 1993)

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
W2-50-pH3.00-PA	635.885	0.5853	1086.4258	1.2563176E+14	2.08587E-10	48.6090
W2-50-pH3.00-PB	831.195	0.7679	1082.4261	1.2516925E+14	2.07819E-10	48.4300
W2-50-pH3.00-EA	536.799	0.5059	1061.0773	1.2270052E+14	2.0372E-10	47.4748
W2-50-pH3.00-EB	551.974	0.5077	1087.2050	1.2572187E+14	2.08736E-10	48.6438
W2-50-pH3.00-SA	458.065	0.4460	1027.0516	1.1876586E+14	1.97187E-10	45.9524
W2-50-pH3.00-SB	498.024	0.4653	1070.3288	1.2377035E+14	2.05496E-10	47.8888
W2-50-pH4.00-PA	400.593	0.3735	1072.5382	1.2402583E+14	2.0592E-10	47.9876
W2-50-pH4.00-PB	994.770	0.9236	1077.0572	1.245484E+14	2.06788E-10	48.1898
W2-50-pH4.00-EA	554.379	0.5055	1096.6944	1.268192E+14	2.10558E-10	49.0684
W2-50-pH4.00-EB	531.972	0.5063	1050.7051	1.2150111E+14	2.01729E-10	47.0107
W2-50-pH4.00-SA	445.874	0.4403	1012.6596	1.1710161E+14	1.94424E-10	45.3085
W2-50-pH4.00-SB	466.613	0.4475	1042.7106	1.2057664E+14	2.00194E-10	46.6531
W2-50-pH5.00-PA	773.501	0.7231	1069.7013	1.2369778E+14	2.05376E-10	47.8607
W2-50-pH5.00-PB	746.983	0.7263	1028.4772	1.1893072E+14	1.97461E-10	46.0162
W2-50-pH5.00-EA	523.477	0.5013	1044.2390	1.2075338E+14	2.00487E-10	46.7214
W2-50-pH5.00-EB	532.013	0.5038	1056.0004	1.2211344E+14	2.02745E-10	47.2477
W2-50-pH5.00-SA	425.147	0.4476	949.8369	1.0983694E+14	1.82363E-10	42.4977
W2-50-pH5.00-SB	428.371	0.4256	1006.5108	1.1639058E+14	1.93244E-10	45.0334
W2-50-pH6.00-PA	446.125	0.4151	1074.7410	1.2428056E+14	2.06343E-10	48.0862
W2-50-pH6.00-PB	576.142	0.5504	1046.7696	1.2104601E+14	2.00973E-10	46.8347
W2-50-pH6.00-EA	521.264	0.5055	1031.1850	1.1924384E+14	1.97981E-10	46.1374
W2-50-pH6.00-EB	518.221	0.5061	1023.9498	1.1840718E+14	1.96592E-10	45.8137
W2-50-pH6.00-SA	432.265	0.4531	954.0168	1.1032029E+14	1.83165E-10	42.6847
W2-50-pH6.00-SB	438.985	0.4475	980.9721	1.1343734E+14	1.8834E-10	43.8907
W2-50-pH7.00-PA	1048.770	1.1216	935.0660	1.0812886E+14	1.79527E-10	41.8368
W2-50-pH7.00-PB	416.764	0.4435	939.7159	1.0866657E+14	1.80419E-10	42.0449
W2-50-pH7.00-EA	471.836	0.5067	931.1940	1.0768112E+14	1.78783E-10	41.6636
W2-50-pH7.00-EB	468.105	0.5069	923.4662	1.0678749E+14	1.77299E-10	41.3178
W2-50-pH7.00-SA	367.903	0.4384	839.1948	9.7042543E+13	1.6112E-10	37.5473
W2-50-pH7.00-SB	347.118	0.4571	759.3918	8.7814311E+13	1.45798E-10	33.9768
W2-50-pH8.00-PA	633.431	0.5858	1081.3093	1.2504011E+14	2.07604E-10	48.3800
W2-50-pH8.00-PB	663.254	0.6171	1074.7918	1.2428643E+14	2.06353E-10	48.0884
W2-50-pH8.00-EA	545.878	0.5052	1080.5186	1.2494867E+14	2.07453E-10	48.3447
W2-50-pH8.00-EB	554.253	0.5057	1096.0115	1.2674023E+14	2.10427E-10	49.0378
W2-50-pH8.00-SA	419.669	0.4496	933.4275	1.0793939E+14	1.79212E-10	41.7635
W2-50-pH8.00-SB	477.258	0.4488	1063.4091	1.2297016E+14	2.04168E-10	47.5791
W2-50-pH9.00-PA	573.272	0.5436	1054.5843	1.2194968E+14	2.02473E-10	47.1843
W2-50-pH9.00-PB	645.924	0.5892	1096.2729	1.2677046E+14	2.10477E-10	49.0495
W2-50-pH9.00-EA	565.650	0.5057	1118.5485	1.2934636E+14	2.14754E-10	50.0462
W2-50-pH9.00-EB	549.534	0.5048	1088.6173	1.2588518E+14	2.09007E-10	48.7070
W2-50-pH9.00-SA	459.230	0.4385	1047.2748	1.2110443E+14	2.0107E-10	46.8573
W2-50-pH9.00-SB	481.332	0.4516	1065.8370	1.2325092E+14	2.04634E-10	47.6878

TD 6/14/94
See Verification
on pages 142-143-144
of GC-N.
(CNWRA controlled copy)
031

SAMPLE WTS FOR SECOND SAMPLING OF W1-50 & W1-5

VIAL #	SAMPLE	WT. VIAL & ACID (g)	WT. VIAL, ACID, & SAMPLE (g)	WT. SAMPLE (g)
1	BLANK	-	-	-
2	W1-50-FEP-1D	7.9427	8.4462	0.5035
3	W1-50-FEP-1E	8.0581	8.5649	0.5068
4	W1-50-FEP-1F	8.0883	8.5906	0.5023
5	W1-50-FEP-2D	8.0232	8.5310	0.5078
6	W1-50-FEP-2E	8.0453	8.5476	0.5023
7	W1-50-FEP-2F	7.8461	8.3520	0.5059
8	W1-50-FEP-3D	7.8237	8.3304	0.5067
9	W1-50-FEP-3E	7.8910	8.3984	0.5074
10	W1-50-FEP-3F	7.9444	8.4490	0.5046
11	W1-50-PP-1D	7.9948	8.4979	0.5031
12	W1-50-PP-1E	8.0092	8.5145	0.5053
13	W1-50-PP-1F	8.0707	8.5153	0.4446
14	W1-50-PP-2D	7.8625	8.3666	0.5041
15	W1-50-PP-2E	7.8690	8.3765	0.5075
16	W1-50-PP-2F	8.0097	8.5107	0.5010
17	W1-50-PP-3D	7.9066	8.4099	0.5033
18	W1-50-PP-3E	7.8403	8.3447	0.5044
19	W1-50-PP-3F	7.8686	8.3738	0.5052
20	W1-5-FEP-1D	7.8113	8.3169	0.5056
21	W1-5-FEP-1E	7.8146	8.3197	0.5051
22	W1-5-FEP-1F	7.8132	8.3180	0.5048
23	W1-5-FEP-2D	7.8453	8.3481	0.5028
24	W1-5-FEP-2E	7.8382	8.3413	0.5031
25	W1-5-FEP-2F	7.8181	8.3220	0.5039
26	W1-5-FEP-3D	7.7732	8.2787	0.5055
27	W1-5-FEP-3E	7.8477	8.3538	0.5061
28	W1-5-FEP-3F	7.7573	8.2626	0.5053
29	W1-5-PP-1D	7.8287	8.3370	0.5083
30	W1-5-PP-1E	7.8730	8.3797	0.5067
31	W1-5-PP-1F	7.7821	8.2865	0.5044
32	W1-5-PP-2D	7.7889	8.2936	0.5047
33	W1-5-PP-2E	7.7973	8.3008	0.5035
34	W1-5-PP-2F	7.8246	8.3295	0.5049
35	W1-5-PP-3D	7.7850	8.2941	0.5091
36	W1-5-PP-3E	7.7687	8.2756	0.5069
37	W1-5-PP-3F	7.8277	8.3339	0.5062

4 MAY 1993 TD

The 2nd week samples were taken for W1-50 and W1-5.
The samples were acidified with 0.5 ml 0.02 M HNO₃
and 5ml "cocktail" was added to each 0.5ml sample.
The samples were weighed (see Table on following page)
and placed in the LS analyzer until at least
5 May 1993.

7 MAY 1993 TD

The second set of samples for W2-50 were taken.
They were taken ^{TD 5/19/93} by pouring (P), and Eppendorf
500 µL Pipet (E) and a 1ml Syringe with filter
(S). They were acidified with 500 µL 0.02 M HNO₃
and all got 5 ml of "cocktail." The weights can
be found on the following page.

WEIGHTS FOR 7 MAY 1993 SAMPLING.

P=pouring, E=using Eppendorf, S=syringe. T for pH measurements=22.9 C

VIAL #	SAMPLE NAME	pH	WT VIAL (g)	WT VIAL+SAMPLE (g)	WT SAMPLE (g)
2	W2-50-pH3.00P-C	2.95	7.8287	8.5508	0.7221
3	W2-50-pH3.00P-D		7.7629	8.3809	0.6180
4	W2-50-pH3.00E-C		7.8461	8.3494	0.5033
5	W2-50-pH3.00E-D		7.8275	8.3314	0.5039
6	W2-50-pH3.00S-C		7.7771	8.2281	0.4510
7	W2-50-pH3.00S-D		7.8015	8.2532	0.4517
8	W2-50-pH4.00P-C	3.72	7.7321	8.5121	0.7800
9	W2-50-pH4.00P-D		7.7829	8.4465	0.6636
10	W2-50-pH4.00E-C		7.7538	8.2591	0.5053
11	W2-50-pH4.00E-D		7.7352	8.2388	0.5036
12	W2-50-pH4.00S-C		7.7994	8.2389	0.4395
13	W2-50-pH4.00S-D		7.8216	8.2863	0.4647
14	W2-50-pH5.00P-C	4.05	7.8072	8.6454	0.8382
15	W2-50-pH5.00P-D		7.8547	8.4925	0.6378
16	W2-50-pH5.00E-C		7.8144	8.3191	0.5047
17	W2-50-pH5.00E-D		7.8277	8.3306	0.5029
18	W2-50-pH5.00S-C		7.8485	8.3042	0.4557
19	W2-50-pH5.00S-D		7.8369	8.2871	0.4502
20	W2-50-pH6.00P-C	4.35	7.8205	8.4857	0.6652
21	W2-50-pH6.00P-D		7.7825	8.1965	0.4140
22	W2-50-pH6.00E-C		7.7837	8.2866	0.5029
23	W2-50-pH6.00E-D		7.8154	8.3182	0.5028
24	W2-50-pH6.00S-C		7.8289	8.2940	0.4651
25	W2-50-pH6.00S-D		7.7218	8.1841	0.4623
26	W2-50-pH7.00P-C	6.43	7.7163	8.4990	0.7827
27	W2-50-pH7.00P-D		7.8225	8.6564	0.8339
28	W2-50-pH7.00E-C		7.7348	8.2352	0.5004
29	W2-50-pH7.00E-D		7.8148	8.3153	0.5005
30	W2-50-pH7.00S-C		7.7608	8.1889	0.4281
31	W2-50-pH7.00S-D		7.7824	8.3260	0.5436
32	W2-50-pH8.00P-C	7.73	7.8174	8.8407	1.0233
33	W2-50-pH8.00P-D		7.8021	8.7032	0.9011
34	W2-50-pH8.00E-C		7.7621	8.2634	0.5013
35	W2-50-pH8.00E-D		7.8338	8.3364	0.5026
36	W2-50-pH8.00S-C		7.7812	8.2185	0.4373
37	W2-50-pH8.00S-D		7.7701	8.3082	0.5381
38	W2-50-pH9.00P-C	8.91	7.8003	8.1551	0.3548
39	W2-50-pH9.00P-D		7.8163	8.4059	0.5896
40	W2-50-pH9.00E-C		7.8512	8.3534	0.5022
41	W2-50-pH9.00E-D		7.7815	8.2844	0.5029
42	W2-50-pH9.00S-C		7.7901	8.2488	0.4587
43	W2-50-pH9.00S-D		7.8036	8.3079	0.5043

10 May 1993 TO

The W2-50 and W2-5 samples were ^{to 5/10/93} have finished counting by liquid ~~scintillation~~ scintillation. The print out below shows the results of the counting.

Protocol #: 5 Name: U-233 3% 2 sigma 07-May-93 15:54
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL= 100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time = 999.89 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.70	1.46	2.928	3.70
2	9.05	1.08	275.5	488.232	3.02
3	9.04	2.78	112.1	488.865	3.02
4	8.74	3.50	91.52	505.882	3.02
5	8.75	0.96	314.9	505.186	3.02
6	9.04	0.00	0.00	489.218	3.02
7	8.80	1.53	199.5	502.867	3.02
8	8.88	1.57	193.4	498.085	3.02
9	8.86	0.71	417.7	499.216	3.02
10	8.89	1.43	210.8	496.959	3.02
11	9.86	1.38	207.8	448.289	3.02
12	9.91	1.78	182.0	445.811	3.02
13	11.13	2.50	110.9	396.533	3.02
14	12.37	1.83	141.4	356.490	3.02
15	12.17	1.68	155.2	362.314	3.02
16	12.42	1.27	201.4	355.043	3.02
17	15.30	0.00	0.00	287.726	3.03
18	15.34	0.00	0.00	286.902	3.03
19	15.20	0.00	0.00	289.440	3.03
20	107.28	0.00	0.00	38.506	3.24
21	108.28	0.47	187.8	38.114	3.24
22	107.69	0.00	0.00	38.339	3.24
23	93.88	0.26	362.7	44.420	3.21
24	95.05	0.15	613.6	43.826	3.21
25	93.32	1.47	66.12	44.693	3.21
26	94.23	0.60	157.0	44.233	3.21
27	96.40	0.96	98.68	43.172	3.21
28	94.79	0.00	0.00	43.955	3.21
29	124.42	0.93	89.99	32.790	3.28
30	124.48	1.37	61.75	32.772	3.28
31	123.96	0.80	104.3	32.922	3.28
32	120.13	0.06	1357.	34.074	3.27
33	118.03	1.06	81.68	34.723	3.27
34	999.98	3.13	12.87	0.000	0.00
35	131.31	0.47	173.8	30.916	3.30
36	132.42	1.20	68.24	30.632	3.31
37	128.70	0.97	85.20	31.602	3.30

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED
SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

The S# corresponds to vial number at on page 263.

RESULTS OF SECOND SAMPLING TIME (W1)

TD
6/14/94
for verification
ice pages
TD 6/15/94
143-144
of GC-11.
CNWRA
controlled copy
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SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W1-50-FEP-1D	488.232	0.5035	969.6763	1.12131118E+14	1.861715E-10	43.3853	2.8970
W1-50-FEP-1E	488.665	0.5068	964.2167	1.11499781E+14	1.851233E-10	43.1411	3.4437
W1-50-FEP-1F	505.882	0.5023	1007.1312	1.16462319E+14	1.933626E-10	45.0612	-0.8538
W1-50-FEP-2D	505.186	0.5078	994.8523	1.15042416E+14	1.910052E-10	44.5118	0.3758
W1-50-FEP-2E	489.218	0.5023	973.9558	1.12625993E+14	1.889932E-10	43.5768	2.4684
W1-50-FEP-2F	502.867	0.5059	994.0047	1.14944406E+14	1.908424E-10	44.4739	0.4607
W1-50-FEP-3D	498.085	0.5067	982.9978	1.13671592E+14	1.887292E-10	43.9814	1.5629
W1-50-FEP-3E	499.216	0.5074	983.8707	1.13772531E+14	1.888968E-10	44.0204	1.4755
W1-50-FEP-3F	496.959	0.5046	984.8573	1.13886619E+14	1.890862E-10	44.0646	1.3767
W1-50-PP-1D	448.289	0.5031	891.0535	1.03039359E+14	1.710765E-10	39.8676	10.7702
W1-50-PP-1E	445.811	0.5053	882.2699	1.02023652E+14	1.693901E-10	39.4746	11.6498
W1-50-PP-1F	396.533	0.4446	891.8871	1.03135757E+14	1.712365E-10	39.9049	10.6867
W1-50-PP-2D	356.490	0.5041	707.1811	81776786296722	1.357742E-10	31.6408	29.1831
W1-50-PP-2E	362.314	0.5075	713.9192	82555964224321	1.370678E-10	31.9422	28.5084
W1-50-PP-2F	355.043	0.5010	708.6687	81948802881073	1.360598E-10	31.7073	29.0342
W1-50-PP-3D	287.726	0.5033	571.6789	66107626205085	1.097586E-10	25.5781	42.7523
W1-50-PP-3E	286.902	0.5044	568.7986	65774549598328	1.092056E-10	25.4492	43.0407
W1-50-PP-3F	289.440	0.5052	572.9216	66251328700337	1.099972E-10	25.6337	42.6278
W1-5-FEP-1D	38.506	0.5056	76.1590	8806852572053	1.462204E-11	3.4075	19.6777
W1-5-FEP-1E	38.114	0.5051	75.4583	8725825951888	1.448751E-11	3.3762	20.4167
W1-5-FEP-1F	38.339	0.5048	75.9489	8782553817751	1.458169E-11	3.3981	19.8993
W1-5-FEP-2D	44.420	0.5028	88.3453	10216042012045	1.696172E-11	3.9528	6.8253
W1-5-FEP-2E	43.826	0.5031	87.1119	10073419080411	1.672492E-11	3.8976	8.1260
W1-5-FEP-2F	44.693	0.5039	88.6942	10256390179381	1.702871E-11	3.9684	6.4573
W1-5-FEP-3D	44.233	0.5055	87.5035	10118697679024	1.68001E-11	3.9151	7.7131
W1-5-FEP-3E	43.172	0.5061	85.3033	9864276020576	1.637768E-11	3.8166	10.0335
W1-5-FEP-3F	43.955	0.5053	86.9879	10059082526404	1.670112E-11	3.8920	8.2568
W1-5-PP-1D	32.790	0.5083	64.5091	7459688488368	1.238534E-11	2.8863	31.9644
W1-5-PP-1E	32.772	0.5067	64.6773	7479135939067	1.241763E-11	2.8938	31.7870
W1-5-PP-1F	32.922	0.5044	65.2696	7547628534748	1.253134E-11	2.9203	31.1623
W1-5-PP-2D	34.074	0.5047	67.5134	7807090241538	1.296213E-11	3.0207	28.7959
W1-5-PP-2E	34.723	0.5035	68.9633	7974751344242	1.32405E-11	3.0856	27.2668
W1-5-PP-2F	0.000	0.5049	0.0000	0	0	0.0000	100.0000
W1-5-PP-3D	30.916	0.5091	60.7268	7022303356523	1.165915E-11	2.7170	35.9535
W1-5-PP-3E	30.632	0.5069	60.4301	6987992772830	1.160218E-11	2.7038	36.2665
W1-5-PP-3F	31.602	0.5062	62.4299	7219245535432	1.198613E-11	2.7932	34.1573

The W1-5-PP-2F shows zero counts because no scintillating "cocktail" was added. It will be added and counted along with the W2.

11 MAY 1993. TD W2-50

The W2-50 samples have been counted using Liquid Scintillation Counting. The raw data, as well as the calculated concentrations can be found on following pages (2)

The last entry is ^{TD 5/11/93} W1-5-PP-2F That was not counted previously.

Protocol #: 5 Name: U-233 3% 2 sigma 10-May-93 17:08
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.95 1.45	2.937 3.69	27.88 1.20	135.30 B
2	5.84	3.65 108.1	758.193 3.01	763.39 3.05	683.89
3	6.61	2.68 135.4	669.983 3.01	672.42 3.06	683.81
4	8.10	1.79 179.7	546.199 3.01	547.18 3.08	696.36
5	8.14	2.91 112.9	543.132 3.02	546.81 3.07	696.25
6	9.46	3.56 86.96	466.936 3.02	471.27 3.08	695.35
7	9.11	2.78 111.5	484.879 3.02	488.15 3.08	697.37
8	4.82	8.85 54.38	919.470 3.01	929.59 3.03	688.13
9	6.08	2.43 154.9	729.135 3.01	733.47 3.05	683.16
10	8.09	0.00 0.00	546.383 3.02	547.03 3.08	692.97
11	8.07	2.98 111.0	548.116 3.02	552.05 3.07	690.67
12	10.17	1.79 180.0	434.133 3.02	437.70 3.09	695.44
13	9.27	0.46 626.5	477.214 3.02	477.73 3.09	695.46
14	5.07	2.94 141.7	873.789 3.01	878.83 3.04	673.62
15	6.50	2.89 127.1	681.986 3.01	684.27 3.06	686.32
16	8.35	0.93 334.3	529.278 3.02	531.40 3.08	694.41
17	8.61	3.69 88.11	513.323 3.02	517.53 3.08	682.59
18	9.57	1.53 192.4	461.640 3.02	464.39 3.09	694.33
19	9.67	0.07 3782.	457.146 3.02	457.23 3.10	695.07
20	6.26	1.17 306.3	706.967 3.01	710.46 3.06	684.19
21	9.92	0.00 0.00	445.450 3.02	444.20 3.11	701.21
22	8.27	1.80 197.5	535.152 3.01	538.14 3.08	689.94
23	8.33	0.00 0.00	531.157 3.02	531.42 3.08	688.51
24	9.63	1.50 194.7	459.057 3.02	460.90 3.09	691.68
25	9.73	4.17 74.22	454.206 3.02	459.99 3.08	687.05
26	6.24	6.69 60.76	709.242 3.01	717.95 3.05	668.41
27	5.94	4.95 81.21	745.211 3.01	750.74 3.05	667.79
28	10.61	1.97 143.2	415.913 3.02	418.87 3.10	688.79
29	10.23	1.38 205.4	431.569 3.02	432.43 3.10	688.25
30	13.32	2.82 91.25	330.997 3.03	334.43 3.12	688.26
31	11.10	5.28 56.20	397.423 3.02	402.66 3.09	679.17
32	3.95	1.05 430.8	1124.91 3.00	1128.32 3.03	658.74
33	4.53	12.39 42.51	979.403 3.01	993.53 3.02	659.52
34	8.23	2.80 116.7	537.039 3.02	541.26 3.07	689.52
35	8.01	4.64 74.18	552.369 3.01	558.14 3.07	686.78
36	10.26	2.88 101.8	430.494 3.02	433.82 3.09	690.33
37	7.69	5.62 63.76	575.476 3.01	583.69 3.06	682.48
38	11.54	4.44 64.39	382.158 3.02	387.28 3.10	693.88
39	6.90	6.12 62.47	641.121 3.01	645.45 3.06	681.37
40	7.97	1.62 198.6	554.905 3.02	556.69 3.08	691.42
41	7.96	1.40 229.5	555.857 3.01	557.42 3.08	686.44
42	8.75	0.82 369.0	505.292 3.02	508.69 3.08	695.32
43	7.78	1.36 239.3	568.528 3.02	570.71 3.07	689.92
44	120.92	0.58 146.5	33.815 3.28	35.27 4.21	674.70

SYSTEM NORMALIZED
 C14 IPA DATA PROCESSED
 C14 CHI SQUARE IPA DATA PROCESSED
 H3 IPA DATA PROCESSED
 H3 CHI SQUARE IPA DATA PROCESSED
 BKG IPA DATA PROCESSED

corrections
to sample
names made
by TD 8/14/93

6/14/93 TO
see verification
on pages 142-144
43 of GC-11.
TD
6/15/94
CNRKA controlled
copy 081

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-pH3.00-PF	758.193	0.7221	1049.9634	1.21417647E+14	2.0159E-10	46.9785	2.5432
W2-50-pH3.00-PF	669.983	0.6180	1084.1149	1.25364535E+14	2.08143E-10	48.5056	-0.6248
W2-50-pH3.00-EF	546.199	0.5033	1085.2354	1.25494114E+14	2.083581E-10	48.5557	-0.7288
W2-50-pH3.00-EF	543.132	0.5039	1077.8567	1.24640855E+14	2.069415E-10	48.2256	-0.0439
W2-50-pH3.00-SF	466.936	0.4510	1035.3348	1.1972372E+14	1.987776E-10	46.3230	3.9029
W2-50-pH3.00-SF	484.879	0.4517	1073.4536	1.2413169E+14	2.060961E-10	48.0286	0.3648
W2-50-pH4.00-PF	919.470	0.7800	1178.8077	1.36314591E+14	2.263234E-10	52.7423	-9.4139
W2-50-pH4.00-PF	729.135	0.6636	1098.7568	1.27057689E+14	2.109542E-10	49.1607	-1.9838
W2-50-pH4.00-EF	546.383	0.5053	1081.3042	1.25039511E+14	2.076034E-10	48.3798	-0.3639
W2-50-pH4.00-EF	548.116	0.5036	1088.3956	1.25850541E+14	2.089649E-10	48.6971	-1.0221
W2-50-pH4.00-SF	434.133	0.4395	987.7884	1.14225562E+14	1.896489E-10	44.1957	8.3160
W2-50-pH4.00-SF	477.214	0.4847	1026.9292	1.18751715E+14	1.971637E-10	45.9470	4.6831
W2-50-pH5.00-PF	873.789	0.8382	1042.4588	1.20547526E+14	2.001453E-10	46.6418	3.2416
W2-50-pH5.00-PF	681.986	0.6378	1069.2788	1.23648921E+14	2.052946E-10	47.8418	0.7523
W2-50-pH5.00-EF	529.278	0.5047	1048.6962	1.21269035E+14	2.013432E-10	46.9210	2.6625
W2-50-pH5.00-EF	513.323	0.5029	1020.7258	1.18034366E+14	1.959727E-10	45.6694	5.2588
W2-50-pH5.00-SF	461.640	0.4557	1013.0349	1.17145009E+14	1.944961E-10	45.3253	5.9727
W2-50-pH5.00-SF	457.146	0.4502	1015.4287	1.17421823E+14	1.949557E-10	45.4324	5.7505
W2-50-pH6.00-PF	706.967	0.6652	1062.7886	1.22898416E+14	2.040485E-10	47.5514	1.3547
W2-50-pH6.00-PF	445.450	0.4140	1075.9662	1.24422238E+14	2.065785E-10	48.1410	0.1316
W2-50-pH6.00-EF	535.152	0.5029	1064.1320	1.23053764E+14	2.043064E-10	47.6115	1.2300
W2-50-pH6.00-EF	531.157	0.5028	1056.3982	1.22159438E+14	2.028216E-10	47.2655	1.9478
W2-50-pH6.00-SF	459.057	0.4651	987.0071	1.14135215E+14	1.894989E-10	44.1608	8.3885
W2-50-pH6.00-SF	454.206	0.4623	982.4919	1.13613087E+14	1.886321E-10	43.9587	8.8076
W2-50-pH7.00-PF	709.242	0.7827	906.1479	1.0478485E+14	1.739745E-10	40.5430	15.8937
W2-50-pH7.00-PF	745.211	0.8339	893.6455	1.0339098E+14	1.715741E-10	39.9836	17.0541
W2-50-pH7.00-EF	415.913	0.5004	831.1611	9611354130496J	1.595775E-10	37.1879	22.8538
W2-50-pH7.00-EF	431.569	0.5005	862.2757	99711567731966	1.655513E-10	38.5800	19.9658
W2-50-pH7.00-SF	330.997	0.4281	773.1768	89408377700887	1.484449E-10	34.5936	28.2357
W2-50-pH7.00-SF	397.423	0.5436	731.0946	84542081110552	1.403654E-10	32.7107	32.1417
W2-50-pH8.00-PF	1124.910	1.0233	1099.2964	1.27120089E+14	2.110578E-10	49.1848	-2.0339
W2-50-pH8.00-PF	979.403	0.9801	1020.0948	1.17961374E+14	1.958515E-10	45.6412	5.3174
W2-50-pH8.00-EF	537.039	0.5013	1071.2926	1.238816E+14	2.056812E-10	47.9319	0.5654
W2-50-pH8.00-EF	552.369	0.5026	1099.0231	1.27088484E+14	2.110053E-10	49.1726	-2.0085
W2-50-pH8.00-SF	430.484	0.4373	984.4363	1.13837935E+14	1.890054E-10	44.0457	8.6271
W2-50-pH8.00-SF	575.478	0.5381	1069.4592	1.23669786E+14	2.053292E-10	47.8498	0.7355
W2-50-pH9.00-PF	382.158	0.3548	1077.1082	1.24554301E+14	2.067978E-10	48.1921	0.0256
W2-50-pH9.00-PF	641.121	0.5896	1087.3630	1.25742446E+14	2.087705E-10	48.6518	-0.9281
W2-50-pH9.00-EF	554.905	0.5022	1104.9482	1.27773654E+14	2.121429E-10	49.4377	-2.5585
W2-50-pH9.00-EF	555.857	0.5029	1105.3032	1.27814707E+14	2.12211E-10	49.4536	-2.5914
W2-50-pH9.00-SF	505.292	0.4587	1101.5740	1.27383466E+14	2.11495E-10	49.2867	-2.2453
W2-50-pH9.00-SF	568.528	0.5043	1127.3607	1.3036538E+14	2.164459E-10	50.4405	-4.6387

W1-5-PP-2F	33.8150	0.5049	66.9737	7.7447E+12	1.2859E-11	2.9965
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12 MAY 1993 TO

The final set of W1-50 and W1-5 samples were taken and weighed and prepared for counting by Liquid Scintillation. The pH of each solution was also measured.

SAMPLE	pH	T (DEGREES C)
W1-50-FEP-1	6.56	20.3
W1-50-FEP-2	6.28	20.3
W1-50-FEP-3	6.47	20.3
W1-50-PP-1	6.52	20.3
W1-50-PP-2	6.52	20.3
W1-50-PP-3	6.48	20.3
W1-5-FEP-1	6.84	20.3
W1-5-FEP-2	6.87	20.3
W1-5-FEP-3	6.88	20.4
W1-5-PP-1	6.88	20.4
W1-5-PP-2	6.89	20.4
W1-5-PP-3	6.89	20.4

The weights can be found on the following page.

IHP should
be FEP
ID

VIAL #	SAMPLE	WT. VIAL & ACID (g)	WT. VIAL, ACID, & SAMPLE (g)	WT. SAMPLE (g)
55	W1-50-IHP-1G	7.8041	8.3042	0.5001
56	W1-50-IHP-1H	7.8215	8.3229	0.5014
57	W1-50-IHP-1I	7.8401	8.3416	0.5015
58	W1-50-IHP-2G	7.8605	8.3642	0.5037
59	W1-50-IHP-2H	7.7793	8.2957	0.5164
60	W1-50-IHP-2I	7.8134	8.3180	0.5046
61	W1-50-IHP-3G	7.8125	8.3142	0.5017
62	W1-50-IHP-3H	7.7939	8.2968	0.5029
63	W1-50-IHP-3I	7.8290	8.3321	0.5031
64	W1-50-PP-1G	7.7894	8.2878	0.4984
65	W1-50-PP-1H	7.7985	8.2999	0.5014
66	W1-50-PP-1I	7.7487	8.2510	0.5023
67	W1-50-PP-2G	7.8074	8.3056	0.4982
68	W1-50-PP-2H	7.8477	8.3477	0.5000
69	W1-50-PP-2I	7.8252	8.3265	0.5013
70	W1-50-PP-3G	7.7809	8.2353	0.4544
71	W1-50-PP-3H	7.7931	8.2958	0.5027
72	W1-50-PP-3I	7.7928	8.2941	0.5013
73	W1-5-IHP-1G	7.8457	8.3374	0.4917
74	W1-5-IHP-1H	7.8131	8.3057	0.4926
75	W1-5-IHP-1I	7.7953	8.2880	0.4927
76	W1-5-IHP-2G	7.7339	8.2276	0.4937
77	W1-5-IHP-2H	7.8153	8.3096	0.4943
78	W1-5-IHP-2I	7.7412	8.2363	0.4951
79	W1-5-IHP-3G	7.8032	8.2961	0.4929
80	W1-5-IHP-3H	7.8076	8.3021	0.4945
81	W1-5-IHP-3I	7.7722	8.2671	0.4949
82	W1-5-PP-1G	7.7747	8.2711	0.4964
83	W1-5-PP-1H	7.7807	8.2832	0.5025
84	W1-5-PP-1I	7.7330	8.2347	0.5017
85	W1-5-PP-2G	8.1438	8.6239	0.4801
86	W1-5-PP-2H	8.0815	8.5758	0.4943
87	W1-5-PP-2I	7.7817	8.2750	0.4933
88	W1-5-PP-3G	8.0877	8.5857	0.4980
89	W1-5-PP-3H	8.0544	8.5509	0.4965
90	W1-5-PP-3I	7.7802	8.2787	0.4985

1000 g 0.1 M NaNO₃ was prepared by dissolving 8.499 g NaNO₃ in 1000g H₂O.

Wt. NaNO₃ Used: 8.5001 g (lot # 7808 KCCL)
Wt. Nanopure H₂O: 1000.0g

This solution will be used in the K3A procedure on the next page.

KINETICS EXPERIMENT K3*A

(Initial pH ~6.5; 0.1M NaNO₃; equil. with atmospheric CO₂)BASED ON: Kinetics Experiment K3
(GC-07-151)

DATE WRITTEN: 30 July 1992

WRITTEN BY: Bobby Pabalan

REVISED BY: Todd Dietrich

OBJECTIVE:

Determine the time it takes to reach sorption equilibrium between ²³³U solutions and clinoptilolite.

K3*A Solution K3*A (3 Bottles)

- Initial ΣU = 50 ppb
- Initial ΣU = 0.1 + x (where 0.1M is from NaNO₃ and x is from NaHCO₃)
- Initial pH = 6.5

STEP 1

In precleaned 250 mL Teflon (FEP) bottles, prepare 250 g of 50 ppb U solution by diluting 25 g of a 500 ppb ²³³U stock solution to a total of 250 g by taring 0.1M NaNO₃ solution into the plastic bottle on the Mettler 4600 balance. The bottles will be labeled K3*A*A, K3*A*B, and K3*A*C with ...*C being a control.

STEP 2

Take 2 500 µL samples of each solution. **MAKE SURE THE WEIGHT OF THE SAMPLE IS KNOWN AND RECORDED.** Add 500 µL 0.02 M HNO₃ and 5 mL scintillation cocktail to each and count within the same day as sampling.To each K3*A solution add 0.0032 g of NaHCO₃ to adjust the pH. Cover the bottles with a kimwipe and place on a gyratory shaker set at ~120 rpm and monitor the pH periodically until it reaches equilibrium with atmospheric CO₂. When this occurs, proceed to the next step.

STEP 3

Take 2 500 µL samples of each solution. **MAKE SURE THE WEIGHT OF THE SAMPLE IS KNOWN AND RECORDED.** Add 500 µL 0.02 M HNO₃ and 5 mL scintillation cocktail to each and count within the same day as sampling. This is the initial ²³³U concentration.Into K3*A*A and K3*A*B add 0.5 g Na-Clinoptilolite. **DO NOT** add to the bottle labeled K3*A*C as this is the control. Return to the gyratory shaker set at 120 rpm.

STEP 4

At the times below, take a 500 µL sample of each solution and prepare for liquid scintillation counting. **MAKE SURE THE WEIGHT OF THE SAMPLE IS KNOWN AND RECORDED.** Add 500 µL 0.02 M HNO₃ but NOT the cocktail. The cocktail will be added at the end right before counting. Label the samples K3*A*A-1, where 1 is the sampling time. Every third sampling time take two samples. Measure and record the pH and temperature of each solution at each sampling time.

Use the following Δtimes: 2, 4, 22, 46, 70, 94, 142, 190, 238, 310, 382, and 454 hours. These times can be adjusted as necessary.

STEP 5

Add the scintillation cocktail to all samples and count using LSA. Convert counts to ppb U and plot vs time. Plot pH vs time.

STEP 1	WT. ²³³ U spike used:	FINAL WEIGHT
	K3*A*A 26.04g	250.01 g
	*B 25.82g	249.99 g
	*C 25.88g	250.00 g

STEP 2

2 500 µL samples were taken from each solution and weighed.

SAMPLE	WT. VIAL(g)	WT. VIAL + SAMPLE(g)	WT. SAMPLE(g)
K3*A*A*U1	7.8230	8.3206	0.4976
U2	7.8628	8.3601	0.4973
*B*U1	7.7422	8.2397	0.4975
U2	7.7918	8.2865	0.4947
*C*U1	7.7947	8.2907	0.4960
U2	7.8250	8.3205	0.4955

5mL scintillation "cocktail" was added and the samples counted with LSA.

0.0032g of NaHCO₃ was added to each solution.

SAMPLE	WT. USED(g)
K3*A*A	0.0037
B	0.0037
C	0.0037

(Lot # 897186A)

The bottles were covered with a kim wipe and placed on a gyratory shaker set to 120rpm. The pH will be monitored periodically until constant.

13 MAY 1993 TD

The third set of W2-50 samples was taken. The samples were prepared for liquid scintillation analysis, and the sample weights, as well as pH, can be found on the following page. The solutions will have their pH adjusted either upward or downward to test reversibility.

SAMPLE WEIGHTS FOR W2-50, THIRD SAMPLING.

P=pouring, E=using Eppendorf, S=syringe. T for pH measurements=22.9 C

VIAL #	SAMPLE NAME	pH	WT VIAL (g)	WT VIAL+SAMPLE (g)	WT SAMPLE (g)
2	W2-50-pH3.00P-E	2.96	7.7758	8.5811	0.8053
3	W2-50-pH3.00P-F		7.8189	8.8956	1.0767
4	W2-50-pH3.00E-E		7.7850	8.2829	0.4979
5	W2-50-pH3.00E-F		7.8319	8.3280	0.4961
6	W2-50-pH3.00S-E		7.7998	8.2338	0.4340
7	W2-50-pH3.00S-F		7.7856	8.2179	0.4323
8	W2-50-pH4.00P-E	3.7	7.8101	8.5555	0.7454
9	W2-50-pH4.00P-F		7.7981	8.7022	0.9041
10	W2-50-pH4.00E-E		7.7687	8.2618	0.4931
11	W2-50-pH4.00E-F		7.8261	8.3202	0.4941
12	W2-50-pH4.00S-E		7.8178	8.2831	0.4653
13	W2-50-pH4.00S-F		7.8008	8.1835	0.3827
14	W2-50-pH5.00P-E	4.06	7.8100	8.7426	0.9326
15	W2-50-pH5.00P-F		7.7835	8.3690	0.5855
16	W2-50-pH5.00E-E		7.8298	8.3244	0.4946
17	W2-50-pH5.00E-F		7.8279	8.3222	0.4943
18	W2-50-pH5.00S-E		7.7918	8.2660	0.4742
19	W2-50-pH5.00S-F		7.7749	8.3169	0.5420
20	W2-50-pH6.00P-E	4.36	7.7922	8.4527	0.6605
21	W2-50-pH6.00P-F		7.7860	8.4482	0.6622
22	W2-50-pH6.00E-E		7.7713	8.2668	0.4955
23	W2-50-pH6.00E-F		7.8074	8.3032	0.4958
24	W2-50-pH6.00S-E		7.7639	8.1994	0.4355
25	W2-50-pH6.00S-F		7.7709	8.2549	0.4840
26	W2-50-pH7.00P-E	6.41	7.7900	8.5700	0.7800
27	W2-50-pH7.00P-F		7.7936	8.4698	0.6762
28	W2-50-pH7.00E-E		7.8009	8.2959	0.4950
29	W2-50-pH7.00E-F		7.7851	8.2790	0.4939
30	W2-50-pH7.00S-E		7.7838	8.2161	0.4323
31	W2-50-pH7.00S-F		7.8068	8.2701	0.4633
32	W2-50-pH8.00P-E	7.71	7.8335	9.2278	1.3943
33	W2-50-pH8.00P-F		7.7927	8.8457	1.0530
34	W2-50-pH8.00E-E		7.7823	8.2777	0.4954
35	W2-50-pH8.00E-F		7.8207	8.3160	0.4953
36	W2-50-pH8.00S-E		7.7729	8.1151	0.3422
37	W2-50-pH8.00S-F		7.8048	8.2401	0.4353
38	W2-50-pH9.00P-E	8.86	7.7818	8.6178	0.8360
39	W2-50-pH9.00P-F		7.8306	8.8492	1.0186
40	W2-50-pH9.00E-E		7.7802	8.2744	0.4942
41	W2-50-pH9.00E-F		7.7443	8.2381	0.4938
42	W2-50-pH9.00S-E		7.8199	8.2844	0.4645
43	W2-50-pH9.00S-F		7.7998	8.2389	0.4391

14 MAY 1993 TD

The W2-50 solutions, ^{5/14/93} with pHi between 4 and 8 had their pH adjusted. The adjustments were 1/2 the value needed previously (see p.214). 1/2 the volume was used, so 1/2 the adjustment.

pHi	Adjustment Needed	Adjustment made
4.00	12 drops, 0.1 M HNO ₃	12 drops, 0.1 M
5.00	12 drops, 0.01 M HNO ₃	12 drops, 0.01 M
6.00	2 drops, 0.01 M HNO ₃	2 drops, 0.01 M
7.00	0.0024g NaHCO ₃	0.0026g
8.00	0.0800g NaHCO ₃	0.0796g

The acid used was previously prepared.
lot # NaHCO₃: 897196A.

14 MAY 1993 TD

The recounting of the second W2-50 set has finished
the results are below and on the next page.

Protocol #: 5 Name: U-233 3% 2 sigma 13-May-93 07:17
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	19.27 1.44	2.992 3.86	28.40 1.19	135.67 B
2	5.64	1.83 212.4	786.015 3.01	789.33 3.05	708.71
3	6.62	2.78 131.6	668.062 3.01	673.86 3.06	705.99
4	8.18	1.51 212.1	540.529 3.02	542.26 3.08	715.40
5	7.95	4.88 71.68	556.253 3.02	562.54 3.07	715.16
6	9.24	1.07 277.8	478.393 3.02	480.04 3.09	718.87
7	9.16	0.00 0.00	482.707 3.02	482.84 3.10	720.33
8	4.93	2.23 187.9	899.239 3.01	901.21 3.05	694.77
9	6.15	0.89 408.4	720.097 3.01	720.87 3.06	706.26
10	8.06	0.00 0.00	548.497 3.02	546.78 3.09	716.93
11	8.20	2.07 156.6	539.203 3.02	542.57 3.08	713.31
12	10.43	0.96 292.6	423.374 3.02	425.10 3.10	716.71
13	9.17	3.08 101.7	481.741 3.02	485.67 3.08	714.56
14	5.03	0.00 0.00	881.899 3.01	883.73 3.05	698.55
15	8.59	4.10 92.21	671.970 3.01	677.97 3.05	706.24
16	8.35	0.49 634.2	529.703 3.02	530.40 3.09	717.89
17	8.33	1.25 251.2	530.982 3.02	533.78 3.08	709.18
18	9.46	3.45 90.11	466.987 3.02	471.81 3.08	714.59
19	9.77	5.19 61.22	452.074 3.02	457.78 3.08	711.65
20	6.43	1.88 193.8	688.454 3.01	692.28 3.06	706.11
21	10.09	0.00 0.00	437.543 3.02	437.70 3.11	724.06
22	8.59	2.03 155.7	514.936 3.02	519.21 3.08	713.09
23	8.58	0.00 0.00	515.073 3.02	516.24 3.09	716.26
24	9.51	3.23 95.66	464.726 3.02	469.29 3.08	715.26
25	10.01	1.91 153.4	441.064 3.02	444.93 3.09	714.65
26	6.28	1.17 308.8	707.072 3.01	708.98 3.06	696.17
27	5.90	3.10 126.0	751.415 3.01	755.84 3.05	695.50
28	10.11	0.61 463.6	436.573 3.02	437.77 3.10	714.32
29	8.99	0.00 0.00	442.353 3.02	442.67 3.10	717.37
30	13.59	0.89 276.0	324.234 3.03	325.17 3.14	719.06
31	11.09	2.10 133.0	397.729 3.02	403.70 3.09	711.29
32	3.95	4.27 114.5	1122.58 3.01	1126.79 3.04	683.04
33	4.51	4.45 103.2	983.926 3.01	991.33 3.03	682.54
34	8.05	3.21 104.5	549.306 3.02	552.59 3.08	719.35
35	8.17	3.13 106.3	541.184 3.02	544.92 3.08	713.45
36	10.40	1.59 178.8	424.796 3.02	426.98 3.10	717.85
37	7.82	2.98 113.7	565.422 3.02	567.89 3.08	707.81
38	11.25	2.42 115.5	392.208 3.02	395.78 3.10	722.51
39	6.82	6.83 57.46	648.621 3.01	656.35 3.05	703.48
40	8.03	0.65 485.2	551.055 3.01	552.42 3.08	719.98
41	8.01	2.07 157.9	552.563 3.01	555.99 3.07	713.81
42	8.71	2.89 111.0	507.456 3.02	511.44 3.08	716.35
43	7.99	2.00 163.5	553.829 3.01	556.58 3.08	711.13

Results of concentration Analysis - W2

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-pH3.00-PC	786.015	0.7221	1088.5127	1.25873084E+14	2.089874E-10	48.7023	-1.0330
W2-50-pH3.00-PD	669.062	0.6180	1082.6246	1.25192201E+14	2.078569E-10	48.4389	-0.4865
W2-50-pH3.00-EC	540.529	0.5033	1073.9698	1.2419138E+14	2.061952E-10	48.0517	0.3169
W2-50-pH3.00-ED	556.253	0.5039	1103.8956	1.27651932E+14	2.119408E-10	49.3906	-2.4608
W2-50-pH3.00-SC	478.393	0.4510	1060.7384	1.22661327E+14	2.036549E-10	47.4597	1.5450
W2-50-pH3.00-SD	482.707	0.4517	1068.6451	1.23575847E+14	2.051729E-10	47.8134	0.8111
W2-50-pH4.00-PC	899.239	0.7800	1152.8705	1.33315276E+14	2.213436E-10	51.5818	-7.0065
W2-50-pH4.00-PD	720.087	0.6636	1085.1371	1.25482745E+14	2.083393E-10	48.5513	-0.7197
W2-50-pH4.00-EC	548.497	0.5053	1085.4878	1.25523299E+14	2.084066E-10	48.5670	-0.7522
W2-50-pH4.00-ED	538.203	0.5036	1070.6970	1.23812919E+14	2.055669E-10	47.9052	0.6206
W2-50-pH4.00-SD	423.374	0.4395	963.3083	1.11394741E+14	1.849488E-10	43.1004	10.5882
W2-50-pH4.00-SC	481.741	0.4647	1036.6710	1.1987823E+14	1.990341E-10	46.3828	3.7788
W2-50-pH5.00-PC	881.889	0.8382	1052.1224	1.21664998E+14	2.020007E-10	47.0742	2.3447
W2-50-pH5.00-PD	671.970	0.6378	1053.5746	1.21832948E+14	2.022795E-10	47.1391	2.2099
W2-50-pH5.00-EC	529.703	0.5047	1049.5403	1.21366412E+14	2.015049E-10	46.9586	2.5843
W2-50-pH5.00-ED	530.982	0.5029	1055.8401	1.22094907E+14	2.027144E-10	47.2405	1.9996
W2-50-pH5.00-SC	466.987	0.4557	1024.7885	1.18501855E+14	1.967489E-10	45.8503	4.8836
W2-50-pH5.00-SD	452.074	0.4502	1004.1626	1.16119037E+14	1.927927E-10	44.9283	6.7962
W2-50-pH6.00-PC	688.454	0.6652	1034.9579	1.19680135E+14	1.987052E-10	46.3062	3.9378
W2-50-pH6.00-PD	437.543	0.4140	1056.8671	1.2221367E+14	2.029116E-10	47.2864	1.9043
W2-50-pH6.00-EC	514.936	0.5029	1023.9332	1.18405262E+14	1.965885E-10	45.8129	4.9611
W2-50-pH6.00-ED	515.073	0.5028	1024.4093	1.1846032E+14	1.966799E-10	45.8342	4.9169
W2-50-pH6.00-SC	464.726	0.4651	989.1959	1.15544696E+14	1.918391E-10	44.7061	7.2572
W2-50-pH6.00-SD	441.064	0.4623	954.0645	1.10325805E+14	1.831742E-10	42.6868	11.4462
W2-50-pH7.00-PC	707.072	0.7827	903.3755	1.0446425E+14	1.734422E-10	40.4189	16.1510
W2-50-pH7.00-PD	751.415	0.8339	901.0853	1.04199413E+14	1.730025E-10	40.3164	16.3636
W2-50-pH7.00-EC	436.573	0.5004	872.4480	1.00887871E+14	1.675044E-10	39.0352	19.0216
W2-50-pH7.00-ED	442.353	0.5005	883.8222	1.0220315E+14	1.696881E-10	39.5441	17.9659
W2-50-pH7.00-SC	324.234	0.4281	757.3791	87581567009578	1.454119E-10	33.8867	29.7020
W2-50-pH7.00-SD	397.729	0.5436	731.6575	84607175171087	1.404735E-10	32.7359	32.0894
W2-50-pH8.00-PC	1122.580	1.0233	1097.0194	1.26856788E+14	2.106206E-10	49.0829	-1.8225
W2-50-pH8.00-PD	983.926	0.9601	1024.8055	1.18506134E+14	1.96756E-10	45.8519	4.8802
W2-50-pH8.00-EC	549.306	0.5013	1095.7630	1.26711497E+14	2.103794E-10	49.0267	-1.7059
W2-50-pH8.00-ED	541.194	0.5026	1076.7887	1.24517351E+14	2.067364E-10	48.1778	0.0552
W2-50-pH8.00-SC	424.786	0.4373	971.4064	1.12331181E+14	1.865037E-10	43.4628	9.8365
W2-50-pH8.00-SD	565.422	0.5381	1050.7749	1.21509182E+14	2.01742E-10	47.0139	2.4698
W2-50-pH9.00-PC	392.208	0.3548	1105.4340	1.27829833E+14	2.122361E-10	49.4594	-2.6036
W2-50-pH9.00-PD	648.621	0.5896	1100.1035	1.27213416E+14	2.112127E-10	49.2209	-2.1088
W2-50-pH9.00-EC	551.055	0.5022	1097.2820	1.26887144E+14	2.10671E-10	49.0947	-1.8469
W2-50-pH9.00-ED	552.563	0.5029	1098.7532	1.27057279E+14	2.109535E-10	49.1605	-1.9835
W2-50-pH9.00-SC	507.456	0.4587	1106.2917	1.27929009E+14	2.124008E-10	49.4978	-2.6832
W2-50-pH9.00-SD	553.829	0.5043	1098.2134	1.2699485E+14	2.108498E-10	49.1364	-1.9334

6/14/94 TD
for verification
see pages 147-149
of GC-11.
CNWRA CONTROLLED
(not sig)

55	9.19	1.82	187.1	480.686	3.02	483.68	3.09	716.02
56	9.24	2.91	106.8	478.089	3.02	481.77	3.09	714.93
57	9.32	2.40	127.6	473.939	3.02	477.71	3.09	715.01
58	9.31	1.56	192.1	474.452	3.02	475.47	3.10	717.36
59	8.77	1.82	171.0	503.963	3.02	506.15	3.09	716.48

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS	FLAG
60	9.13	1.10	273.0	483.755	3.02	487.48 3.08 715.79
61	9.10	0.00	0.00	485.469	3.02	484.57 3.10 722.78
62	8.89	2.10	148.3	497.008	3.02	500.96 3.08 711.85
63	8.96	5.17	64.14	492.990	3.02	497.94 3.08 713.96
64	9.95	0.00	0.00	443.641	3.02	443.96 3.10 718.69
65	9.97	2.79	107.0	442.845	3.02	446.22 3.09 711.90
66	9.66	0.00	0.00	457.153	3.02	455.66 3.11 716.85
67	12.31	1.77	148.9	358.096	3.03	380.79 3.12 715.32
68	11.85	3.09	89.38	372.113	3.02	376.49 3.11 711.73
69	12.08	1.01	258.5	364.972	3.02	366.63 3.12 717.14
70	17.16	1.76	126.5	255.982	3.04	258.43 3.17 716.94
71	14.83	0.55	422.5	296.873	3.03	298.50 3.15 719.94
72	15.06	2.31	104.5	292.360	3.03	296.50 3.14 712.66
73	110.40	0.17	507.9	37.271	3.25	38.01 4.18 722.12
74	111.88	0.02	578.4	36.729	3.26	37.21 4.21 718.68
75	109.17	0.00	0.00	37.715	3.25	37.97 4.20 724.91
76	95.32	0.00	0.00	43.630	3.22	44.16 4.02 719.82
77	95.60	0.64	148.4	43.493	3.22	44.72 3.98 709.95
78	95.52	0.43	221.3	43.532	3.22	44.32 4.01 712.97
79	97.16	0.00	0.00	42.757	3.22	43.00 4.06 715.16
80	95.96	0.69	137.6	43.329	3.22	45.13 3.95 705.54
81	94.00	0.00	0.00	44.295	3.21	44.79 4.01 718.74
82	123.82	0.00	0.00	32.899	3.29	32.77 4.41 737.52
83	119.64	0.54	158.0	34.169	3.28	35.06 4.26 711.68
84	119.64	0.00	0.00	34.161	3.28	34.87 4.28 716.31
85	119.32	0.63	136.7	34.261	3.28	36.24 4.17 692.17
86	117.95	0.01	5918.	34.702	3.27	35.82 4.23 713.04
87	114.84	0.16	534.9	35.705	3.27	36.70 4.20 718.50
88	120.23	0.15	575.7	33.979	3.28	35.48 4.22 710.03
89	124.31	0.00	0.00	32.757	3.29	33.57 4.32 719.04
90	124.24	0.25	342.4	32.777	3.29	33.71 4.31 716.08
91	7.93	1.91	171.6	557.411	3.02	561.64 3.07 720.28
92	8.02	3.42	98.71	551.372	3.02	556.51 3.07 714.03
93	8.18	0.00	0.00	540.529	3.02	540.67 3.09 721.03
94	8.03	2.64	125.4	550.433	3.02	554.91 3.07 719.28
95	8.21	1.07	296.0	539.273	3.01	545.41 3.07 715.88
96	7.85	0.85	376.4	563.505	3.02	567.65 3.07 717.59

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

The S# refers to the vial # on page 269.

The S# 1-43 are for W2-SD and are on page 273.

The results of the calculations are on the following TD
5/17/93
Following page.

17 MAY 1993 Exp. K3xA

The pH of all K3xA solutions was measured

K3xAxA
B
C
pH/T(°C)
6.93/21.5
6.93/21.4
6.94/21.4

17 MAY 1993 TD

The W1-5, W1-50, AND WTD K3xA SAMPLES HAVE
FINISHED COUNTING BY LSA. THE RESULTS FOLLOW.

RESULTS OF THE THIRD SAMPLING OF W1-50 and W1-5 AND THE INITIAL SAMPLING OF K3*A SOLUTIONS

TD 6/14/94

cc verification

sh pgs TO 6/5/94
GC-11. 143-144
NWRA controlled
copy 081

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W1-50-FEP-1G	480.686	0.5001	961.1798	1.11148602E+14	1.845403E-10	43.0052	3.7478
W1-50-FEP-1H	478.069	0.5014	953.4683	1.10256865E+14	1.830597E-10	42.6602	4.5200
W1-50-FEP-1I	473.939	0.5015	945.0429	1.09282569E+14	1.814421E-10	42.2832	5.3637
W1-50-FEP-2G	474.452	0.5037	941.9337	1.0892303E+14	1.808451E-10	42.1441	5.6751
W1-50-FEP-2H	503.963	0.5164	975.9160	1.12852661E+14	1.879695E-10	43.6645	2.2721
W1-50-FEP-2I	483.755	0.5046	958.6901	1.10860697E+14	1.840623E-10	42.8938	3.9971
W1-50-FEP-3G	485.469	0.5017	967.6480	1.11896573E+14	1.857821E-10	43.2946	3.1001
W1-50-FEP-3H	497.008	0.5029	988.2840	1.14282867E+14	1.897441E-10	44.2179	1.0336
W1-50-FEP-3I	492.990	0.5031	979.9046	1.13313898E+14	1.881353E-10	43.8430	1.8727
W1-50-PP-1G	443.641	0.4984	890.1304	1.0293262E+14	1.708993E-10	39.8263	10.8627
W1-50-PP-1E	442.845	0.5014	883.2170	1.02133167E+14	1.695719E-10	39.5170	11.5550
W1-50-PP-1I	457.153	0.5023	910.1195	1.05244105E+14	1.74737E-10	40.7206	8.8610
W1-50-PP-2G	358.096	0.4982	718.7796	83118009017855	1.38001E-10	32.1597	28.0217
W1-50-PP-2H	372.113	0.5000	744.2260	86060571018884	1.428866E-10	33.2982	25.4735
W1-50-PP-2I	364.972	0.5013	728.0511	84190139253815	1.397811E-10	32.5745	27.0932
W1-50-PP-3G	255.982	0.4544	563.3407	65143410272568	1.081577E-10	25.2050	43.5873
W1-50-PP-3H	296.873	0.5027	590.5570	68290642857857	1.133631E-10	26.4228	40.8618
W1-50-PP-3I	292.360	0.5013	583.2037	67440321758682	1.119719E-10	26.0938	41.5982
W1-5-FEP-1G	37.271	0.4917	75.8003	8765369372850	1.455316E-11	3.3915	20.0560
W1-5-FEP-1H	36.729	0.4926	74.5615	8622120373420	1.431533E-11	3.3360	21.3625
W1-5-FEP-1I	37.715	0.4927	76.5476	8851786588957	1.469664E-11	3.4249	19.2679
W1-5-FEP-2G	43.630	0.4937	88.3735	10219307582113	1.696714E-11	3.9540	6.7955
W1-5-FEP-2H	43.493	0.4943	87.9891	10174852904269	1.689333E-11	3.9368	7.2009
W1-5-FEP-2I	43.532	0.4951	87.9257	10167521026573	1.688116E-11	3.9340	7.2678
W1-5-FEP-3G	42.757	0.4929	86.7458	10031082278657	1.665463E-11	3.8812	8.5122
W1-5-FEP-3H	43.329	0.4945	87.6218	10132386674222	1.682282E-11	3.9204	7.5882
W1-5-FEP-3I	44.295	0.4949	89.5029	10349911523853	1.718398E-11	4.0045	5.6043
W1-5-PP-1G	32.889	0.4964	66.2550	7661578947128	1.272054E-11	2.9644	30.1231
W1-5-PP-1H	34.169	0.5025	67.9980	7863132387831	1.305518E-11	3.0424	28.2848
W1-5-PP-1I	34.161	0.5017	68.0905	7873826835585	1.307293E-11	3.0465	28.1873
W1-5-PP-2G	34.261	0.4801	71.3622	8252161407653	1.370108E-11	3.1929	24.7367
W1-5-PP-2H	34.702	0.4943	70.2043	8118266053938	1.347877E-11	3.1411	25.9579
W1-5-PP-2I	35.705	0.4933	72.3799	8369842910039	1.389647E-11	3.2384	23.6634
W1-5-PP-3G	33.979	0.4980	68.2309	7890065993823	1.309989E-11	3.0528	28.0392
W1-5-PP-3H	32.757	0.4965	65.9758	7629292276063	1.266693E-11	2.9519	30.4175
W1-5-PP-3I	32.777	0.4965	65.7513	7603322705608	1.262381E-11	2.9418	30.6544

K3*A*A*IU1	557.411	0.4976	1120.1990	1.28537213E+14	2.150709E-10	50.1200
K3*A*A*IU2	551.372	0.4973	1108.7311	1.28211102E+14	2.128692E-10	49.6070
K3*A*B*IU1	540.529	0.4975	1086.4905	1.2563924E+14	2.085991E-10	48.8119
K3*A*B*IU2	550.433	0.4987	1103.7357	1.27633442E+14	2.119101E-10	49.3834
K3*A*C*IU1	539.273	0.4960	1087.2440	1.25726373E+14	2.087438E-10	48.6456
K3*A*C*IU2	563.505	0.4955	1137.2452	1.31508402E+14	2.183437E-10	50.8827

The samples taken from K3*A solutions was done before the addition of NaHCO₃ to adjust the pH.

18 MAY 1993 TO 0930

The pH of the K3*A solutions were remeasured.

K3*A*A
*B
*C
pH (T°C)
7.00/22.4
6.96/22.4
6.97/22.4

19 MAY 1993 TO

The pH of the K3*A solutions was remeasured.

K3*A*A
*B
*C
pH (T°C)
7.04/21.0
6.96/20.9
7.02/20.9

The W2-50 samples, taken on 13 May 1993 (see p 271-272) have finished counting by LSA. The results follow.

Protocol #: 5 Name: U-233 3% 2 sigma 19-May-93 03:07
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS	FLAG
1	999.98	19.06 1.45	3.257 3.50	28.61 1.18	142.60	B
2	5.06	0.00 0.00	877.178 3.01	879.69 3.05	672.08	
3	3.74	4.47 112.5	1185.25 3.01	1190.37 3.03	655.77	
4	8.14	0.59 525.6	543.057 3.02	544.24 3.08	691.09	
5	8.12	4.21 80.63	544.526 3.02	553.53 3.06	684.62	
6	9.85	0.54 526.6	443.577 3.02	445.36 3.10	690.27	
7	9.61	0.00 0.00	459.490 3.02	461.71 3.09	695.12	
8	5.52	0.32 1168.	802.802 3.01	804.00 3.06	680.72	
9	4.50	1.38 309.1	987.187 3.01	980.94 3.04	667.99	
10	8.17	2.72 120.3	540.807 3.02	545.20 3.07	683.66	
11	8.25	3.48 95.25	536.016 3.02	539.51 3.08	686.60	
12	8.83	0.08 3845.	500.140 3.02	501.29 3.09	691.60	
13	10.75	4.57 65.23	410.138 3.02	415.67 3.09	690.08	
14	4.63	1.67 253.7	958.514 3.01	961.02 3.04	662.95	
15	7.01	0.34 886.4	631.836 3.01	634.44 3.07	685.53	
16	8.50	5.41 62.95	519.684 3.02	524.57 3.08	683.47	
17	8.52	2.42 131.9	518.339 3.02	522.80 3.08	686.99	
18	9.55	1.88 158.2	462.083 3.02	465.84 3.09	687.55	
19	11.53	1.75 154.1	382.693 3.02	387.35 3.10	691.57	
20	6.42	4.15 91.95	689.578 3.01	695.53 3.05	678.53	
21	6.43	2.87 129.2	687.878 3.01	691.14 3.06	679.08	
22	8.58	1.92 163.8	514.808 3.02	516.96 3.09	685.95	
23	8.25	0.45 682.1	535.773 3.02	538.78 3.08	688.74	
24	10.35	1.42 199.0	426.501 3.02	429.07 3.10	688.93	
25	9.29	2.14 141.6	475.645 3.02	478.17 3.09	687.95	
26	6.16	0.09 3785.	718.983 3.01	720.09 3.06	674.45	
27	7.58	4.68 75.80	583.023 3.02	589.46 3.06	677.24	
28	10.71	0.27 1018.	411.776 3.02	414.34 3.11	687.32	
29	10.65	3.10 93.57	414.020 3.02	418.43 3.10	684.82	
30	14.21	0.43 547.3	309.692 3.03	310.38 3.15	692.70	
31	14.65	3.05 80.98	300.292 3.03	304.22 3.14	683.00	
32	2.92	2.86 192.1	1521.74 3.00	1525.50 3.02	635.27	
33	3.97	7.39 69.99	1117.65 3.01	1125.04 3.03	656.58	
34	7.91	0.03 11392	559.575 3.01	560.52 3.08	683.60	
35	8.05	2.43 135.1	549.165 3.02	553.75 3.07	681.18	
36	13.10	3.08 85.02	336.209 3.03	340.32 3.12	686.45	
37	9.61	4.56 69.05	459.386 3.02	463.90 3.09	685.52	
38	4.66	2.40 179.4	951.249 3.01	954.22 3.04	670.99	
39	3.88	3.36 143.3	1143.65 3.01	1148.97 3.03	664.13	
40	8.16	2.14 151.3	541.473 3.02	545.29 3.08	684.70	
41	8.25	0.82 381.8	536.016 3.02	537.09 3.08	683.73	
42	8.50	3.88 85.01	519.684 3.02	526.10 3.07	684.49	
43	8.97	1.45 209.4	492.284 3.02	493.24 3.09	688.13	
44	4.19	7.43 67.79	1059.03 3.01	1067.81 3.03	682.21	
45	4.18	6.66 74.80	1065.49 3.01	1077.88 3.03	683.17	
46	4.10	5.57 88.13	1084.06 3.00	1089.93 3.03	685.16	
47	4.12	8.81 80.30	1078.54 3.00	1089.11 3.02	681.30	

RESULTS FROM THE THIRD SAMPLING OF W2-50

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-pH3.00-PE	877.178	0.8053	1089.2562	1.2595906E+14	2.0913E-10	48.7356	-1.1020
W2-50-pH3.00-PF	1185.250	1.0767	1100.8173	1.2729596E+14	2.1135E-10	49.2529	-2.1751
W2-50-pH3.00-EE	543.057	0.4979	1090.6949	1.2612543E+14	2.09406E-10	48.8000	-1.2355
W2-50-pH3.00-EF	544.526	0.4961	1097.6134	1.2692547E+14	2.10735E-10	49.1095	-1.8777
W2-50-pH3.00-SF	443.577	0.4340	1022.0668	1.1818944E+14	1.9623E-10	45.7294	5.1344
W2-50-pH3.00-SE	459.490	0.4323	1062.8961	1.2291085E+14	2.04069E-10	47.5562	1.3447
W2-50-pH4.00-PE	802.902	0.7454	1077.1425	1.2455826E+14	2.06804E-10	48.1936	0.0224
W2-50-pH4.00-PF	987.187	0.9041	1091.9002	1.2626481E+14	2.09638E-10	48.8539	-1.3474
W2-50-pH4.00-EE	540.807	0.4931	1096.7491	1.2682553E+14	2.10569E-10	49.0709	-1.7975
W2-50-pH4.00-EF	536.016	0.4941	1084.8330	1.2544758E+14	2.08281E-10	48.5377	-0.6914
W2-50-pH4.00-SE	500.140	0.4653	1074.8764	1.2429622E+14	2.06369E-10	48.0922	0.2327
W2-50-pH4.00-SF	410.138	0.3827	1071.6958	1.2392843E+14	2.05759E-10	47.9499	0.5279
W2-50-pH5.00-PE	958.514	0.9326	1027.7868	1.1885089E+14	1.97328E-10	45.9853	4.6034
W2-50-pH5.00-PF	631.836	0.5855	1079.1392	1.2478916E+14	2.07188E-10	48.2829	-0.1629
W2-50-pH5.00-EE	519.684	0.4946	1050.7157	1.2150233E+14	2.01731E-10	47.0112	2.4752
W2-50-pH5.00-EF	518.339	0.4943	1048.6324	1.2126142E+14	2.01331E-10	46.9180	2.6686
W2-50-pH5.00-SE	462.083	0.4742	974.4475	1.1268285E+14	1.87088E-10	43.5988	9.5543
W2-50-pH5.00-SF	382.693	0.5420	706.0756	8.1648952E+13	1.35562E-10	31.5913	34.4639
W2-50-pH6.00-PE	689.578	0.6605	1044.0242	1.2072854E+14	2.00446E-10	46.7118	3.0963
W2-50-pH6.00-PF	687.878	0.6622	1038.7768	1.2012174E+14	1.99438E-10	46.4770	3.5834
W2-50-pH6.00-EE	514.808	0.4955	1038.9667	1.201437E+14	1.99475E-10	46.4855	3.5658
W2-50-pH6.00-EF	535.773	0.4958	1080.6232	1.2496077E+14	2.07473E-10	48.3493	-0.3007
W2-50-pH6.00-SE	426.501	0.4355	979.3364	1.1324819E+14	1.88026E-10	43.8176	9.1005
W2-50-pH6.00-SF	475.645	0.4840	982.7376	1.136415E+14	1.88679E-10	43.9697	8.7848
W2-50-pH7.00-PE	718.983	0.7800	921.7731	1.065917E+14	1.76974E-10	41.2421	14.4434
W2-50-pH7.00-PF	583.023	0.6762	862.2050	9.9703386E+13	1.65538E-10	38.5769	19.9723
W2-50-pH7.00-EE	411.776	0.4950	831.8707	9.6195602E+13	1.59714E-10	37.2196	22.7879
W2-50-pH7.00-EF	414.020	0.4939	838.2669	9.6935238E+13	1.60942E-10	37.5058	22.1942
W2-50-pH7.00-SE	309.692	0.4323	716.3821	8.2840772E+13	1.37541E-10	32.0524	33.5072
W2-50-pH7.00-SF	300.292	0.4633	648.1589	7.4951589E+13	1.24442E-10	29.0000	39.8395
W2-50-pH8.00-PE	1521.740	1.3943	1091.4007	1.2620705E+14	2.09542E-10	48.8316	-1.3010
W2-50-pH8.00-PF	1117.650	1.0530	1061.3960	1.2273738E+14	2.03781E-10	47.4891	1.4839
W2-50-pH8.00-EE	559.575	0.4954	1129.5418	1.306176E+14	2.16865E-10	50.5381	-4.8412
W2-50-pH8.00-EF	549.165	0.4953	1108.7523	1.2821354E+14	2.12873E-10	49.6079	-2.9116
W2-50-pH8.00-SE	336.209	0.3422	982.4927	1.1361318E+14	1.88632E-10	43.9588	8.8075
W2-50-pH8.00-SF	459.386	0.4353	1055.3320	1.2203614E+14	2.02617E-10	47.2178	2.0468
W2-50-pH9.00-PE	951.249	0.8360	1137.8577	1.3157922E+14	2.18461E-10	50.9101	-5.6130
W2-50-pH9.00-PF	1143.65	1.0186	1122.7665	1.2983412E+14	2.15564E-10	50.2349	-4.2123
W2-50-pH9.00-EE	541.473	0.4942	1095.6556	1.2669908E+14	2.10359E-10	49.0219	-1.6960
W2-50-pH9.00-EF	536.016	0.4938	1085.4921	1.2552379E+14	2.08407E-10	48.5672	-0.7526
W2-50-pH9.00-SE	519.684	0.4645	1118.8030	1.2937579E+14	2.14803E-10	50.0576	-3.8444
W2-50-pH9.00-SF	492.284	0.4391	1121.1205	1.2964378E+14	2.15248E-10	50.1613	-4.0595

14/94 TD
verification
pH 14.1-14.5
6C-11.
JWRA controlled
ap7 081

20 MA-1 1993 TD

The pH of the K3*A solutions was remeasured.

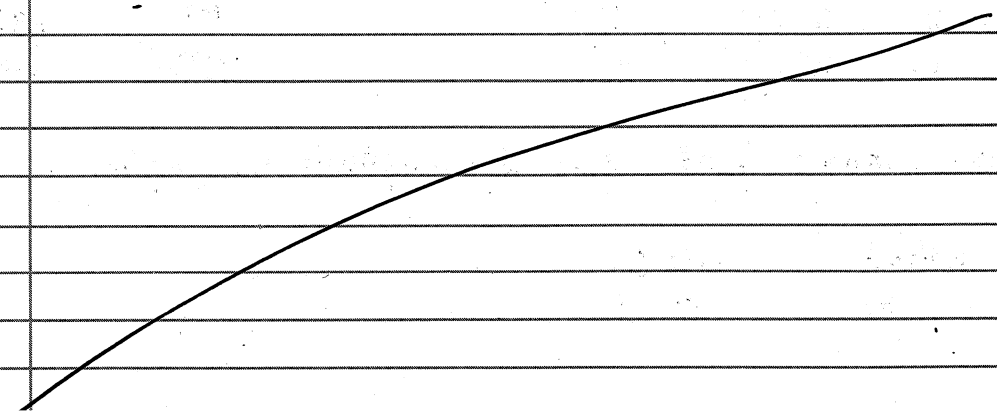
	pH/T(°C)
K3*A*A	7.04/21.5
*B	6.98/21.5
*C	7.02/21.5

Since the pH has stabilized, the experiment will proceed to the next step.

2 500 µL samples of each solution were taken and prepared for LSA.

VIAL #	SAMPLE	WT. VIAL (g)	WT. VIAL & SAMPLE	WT. SAMPLE
2	K3*A*A*IU3	7.7418	8.2416	0.4998
3	*IU4	7.8444	8.3487	0.4993
4	*B *IU3	7.8298	8.3301	0.5003
5	*IU4	7.8361	8.3349	0.4988
6	*C *IU3	7.7801	8.2763	0.4962
7	*IU4	7.8089	8.3059	0.4970
8	K3*A*A*1	7.7925	8.2896	0.4971
9	*B 1	7.7715	8.2667	0.4952
10	*C 1	7.7996	8.2966	0.4970
11	*A 2	7.8521	8.3490	0.4969
12	*B 2	7.7866	8.2831	0.4965
13	*C 2	7.8359	8.3327	0.4968

SAMPLE	DATE/TIME	Δ TIME (hrs)	pH / T(°C)
1	TD 5/20, 13:00	2	7.00/22.5
2	TD 5/20, 1500	4	7.06/23.6
3	TD 5/21, 9:00	22	7.10/20.9
4	TD 5/22, 900	46	7.11/21.5
5	TD 5/23, 900	70	7.10/21.1
6	TD 5/24, 900	94	7.07/20.8
7	TD 5/26, 900	142	7.04/21.0
8	TD 5/28, 930	190.5	7.11/22.2
9	TD 5/30, 915	238.25	7.09/22.1
10	TD 6/2, 900	310	7.12/21.4
11	TD 6/5, 930	382.5	7.05/20.6
12	TD 6/8, 930	454.5	7.13/21.6



K3*A*B INITIAL pH = 6.98 5/20/93 11:01

SAMPLE	DATE/TIME	ΔTIME (hrs)	pH/T(°C)
1	TD 5/20, 1301	2	6.90/22.6
2	TD 5/20, 1501	4	6.96/23.6
3	TD 5/21, 900	22	6.97/22.5
4	TD 5/22, 901	46	6.98/21.5
5	TD 5/23, 901	70	6.96/21.1
6	TD 5/24, 901	94	6.93/20.9
7	TD 5/26, 901	142	6.96/20.0
8	TD 5/26, 931	190.5	6.96/22.2
9	TD 5/30, 916	238.25	6.94/22.1
10	TD 6/2, 901	310	6.96/21.4
11	TD 6/5, 936	382.5	6.96/20.6
12	TD 6/6, 931	454.5	6.92/21.6

K3*A*C INITIAL pH = 7.02 5/20/93 11:02

SAMPLE	DATE/TIME	ΔTIME (hrs)	pH/T(°C)
1	TD 5/20, 1302	2	6.99/22.6
2	TD 5/20, 1502	4	6.99/22.6
3	TD 5/21, 902	22	6.97/21.0
4	TD 5/22, 902	46	6.98/21.5
5	TD 5/23, 902	70	6.97/21.4
6	TD 5/24, 902	94	6.96/20.9
7	TD 5/26, 902	142	6.95/21.0
8	TD 5/28, 932	190.5	6.97/22.2
9	TD 5/30, 917	238.25	6.96/22.1
10	TD 6/2, 902	310	6.98/21.4
11	TD 6/5, 932	382.5	6.95/20.6
12	TD 6/8, 932	454.5	6.94/21.6

To K3*A*A & *B, 0.5g Na-clinoptilolite was added

K3*A*A 0.5000g
 *B 0.5005g

The bottles were swirled and returned to the gyratory shaker.

21 MAY 1993 TD

Two samples from each K3*A solutions^{stills} were taken, weighed, acidified, and prepared for counting by liquid scintillation analysis. The sample weights are in the table below.

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*3a	7.8135	8.3117	0.4982
3	K3*A*A*3b	7.8336	8.3348	0.5012
4	K3*A*B*3a	7.8212	8.3218	0.5006
5	K3*A*B*3b	7.8198	8.3215	0.5017
6	K3*A*C*3a	7.7803	8.2885	0.5082
7	K3*A*C*3b	7.7974	8.3009	0.5035
52	K3*A*A*4	7.7491	8.2505	0.5014
53	K3*A*B*4	7.7545	8.2558	0.5013
54	K3*A*C*4	7.7527	8.2528	0.5001

The pH of each solution was also measured, and can be found in the tables on page 279-280. The time of sampling was also noted.

The W2-50 solutions were sampled in the same manner as before. This is the first sampling for the W2 reverse experiments. The samples with pH between 4 & 8 had the pH adjusted (see page 272-273).

22 May 1993 TD

The K3*A experimental solutions (A,B,C) were sampled.
The weights are included on the ^{5th} previous page
and the pH, T, and time are on pages 279-280.

23 May 1993 TD

The 5th K3*A*A(BorC) sample was taken today
The pH, T, and time can be found on p. 279-280.

24 May 1993 TD

The 6th sample, 2 each from K3*A*A(BorC), was
taken today. The pH and time can be found on
page 279-280.

WT FOR 5th & 6th samples

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*5	7.7551	8.2553	0.5002
3	K3*A*B*5	7.7729	8.2757	0.5028
4	K3*A*C*5	7.7511	8.2512	0.5001

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
5	K3*A*A*6a	7.8000	8.2997	0.4997
6	K3*A*A*6b	7.7509	8.2422	0.4913
7	K3*A*B*6a	7.7521	8.2520	0.4999
8	K3*A*B*6b	7.7504	8.2519	0.5015
9	K3*A*C*6a	7.6815	8.1829	0.5014
10	K3*A*C*6b	7.7106	8.2125	0.5019

The LSA of the ^{5th} K3*A initial solutions - sample 4, and
the WZ-S-R 1st sampling has been completed.
The results follow.

K3*A*...IU - K3*A... C2 Liquid Scintillation Data

Protocol #: 5 Name: U-233 3% 2 sigma 22-May-93 03:25
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS	FLAG
1	999.98	18.84 1.46	3.228 3.52	28.67 1.18	143.90	B
2	8.35	3.43 95.49	528.987 3.02	533.13 3.08	699.01	
3	8.29	3.11 105.0	533.081 3.02	534.90 3.08	699.76	
4	8.71	1.94 160.0	507.220 3.02	510.60 3.08	702.35	
5	8.68	2.70 117.1	508.753 3.02	511.89 3.08	699.93	
6	8.45	0.80 381.5	522.926 3.02	525.65 3.08	697.54	
7	8.74	2.21 141.0	505.239 3.02	509.55 3.08	701.40	
8	8.91	1.58 192.0	495.537 3.02	500.51 3.08	706.78	
9	9.41	0.07 3867.	469.142 3.02	470.69 3.10	704.27	
10	8.73	0.52 579.5	506.738 3.02	509.59 3.08	702.27	
11	9.16	0.00 0.00	482.143 3.02	482.80 3.10	702.32	
12	9.60	0.00 0.00	460.001 3.02	458.94 3.11	705.25	
13	8.68	3.74 86.60	508.984 3.02	513.15 3.08	696.27	

SYSTEM NORMALIZED
C14 IPA DATA PROCESSED
C14 CHI SQUARE IPA DATA PROCESSED
H3 IPA DATA PROCESSED
H3 CHI SQUARE IPA DATA PROCESSED
BKG IPA DATA PROCESSED

Results of calculations

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	AVG [U]
K3*A*A*IU3	528.987	0.4998	1058.3974	1.223906E+14	2.0321E-10	47.3549	
K3*A*A*IU4	533.081	0.4993	1067.6567	1.234614E+14	2.0498E-10	47.7692	47.5621
K3*A*B*IU3	507.220	0.5003	1013.8317	1.172371E+14	1.9465E-10	45.3610	
K3*A*B*IU4	508.753	0.4988	1019.9539	1.179451E+14	1.9582E-10	45.6349	45.4979
K3*A*C*IU3	522.926	0.4962	1053.8613	1.218661E+14	2.0233E-10	47.1520	
K3*A*C*IU4	505.239	0.4970	1016.5775	1.175547E+14	1.9518E-10	45.4838	46.3179
K3*A*A1	495.537	0.4971	996.8558	1.152741E+14	1.9139E-10	44.6014	
K3*A*B1	469.142	0.4952	947.3788	1.095527E+14	1.8189E-10	42.3877	
K3*A*C1	506.738	0.4970	1019.5936	1.179034E+14	1.9576E-10	45.6188	
K3*A*A2	482.143	0.4969	970.3019	1.122035E+14	1.8629E-10	43.4133	
K3*A*B2	460.001	0.4965	926.4874	1.071369E+14	1.7788E-10	41.4530	
K3*A*C2	508.984	0.4968	1024.5250	1.184737E+14	1.967E-10	45.8394	

The S# corresponds to vial # on page 279.

6/14/94 TD
See p 143-144
of GC-11 for
verification
of calculation
COWBA controlled
copy of

LSA data for solutions found on page 281.
5# corresponds to Vial #

Protocol #: 5 Name: U-233 3% 2 sigma 24-May-93 03:03
Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
Region C: LL-UL= 0.0-2000. Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
Time =999.99 QIP = SIS
U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	19.15	1.45	4.128	3.11 29.82 1.16 163.87 B
2	10.68	2.48	115.4	412.164	3.03 416.15 3.11 676.91
3	10.69	0.59	465.6	411.681	3.03 412.09 3.12 679.25
4	11.98	0.80	325.1	366.907	3.03 367.84 3.13 675.37
5	12.48	0.00	0.00	352.042	3.04 352.79 3.14 681.02
6	8.38	1.14	275.3	526.540	3.02 526.74 3.10 678.77
7	8.68	1.82	171.8	508.429	3.02 511.19 3.09 679.50

(1 missing vial)

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
9	5.10	3.99	107.1	867.833	3.01 872.34 3.05 668.89
10	7.61	2.66	127.6	580.497	3.02 582.92 3.08 676.15
11	7.82	0.54	589.1	565.053	3.02 567.24 3.08 678.76
12	8.23	0.00	0.00	535.969	3.02 535.67 3.10 682.17
13	8.85	0.00	0.00	498.697	3.02 501.03 3.09 684.26
14	8.90	3.77	85.45	495.760	3.02 500.85 3.08 678.84
15	6.62	1.39	253.8	668.077	3.02 672.14 3.06 678.00
16	4.04	5.85	85.20	1097.61	3.01 1105.57 3.03 655.96
17	7.77	1.57	208.9	567.815	3.02 572.11 3.08 677.77
18	7.71	3.16	108.1	572.526	3.02 574.98 3.08 677.23
19	9.04	0.00	0.00	487.465	3.03 485.33 3.11 687.22
20	8.36	0.82	376.4	528.049	3.02 529.99 3.09 683.42
21	7.32	0.38	853.6	603.659	3.02 605.42 3.08 680.56
22	4.41	1.71	254.9	1006.08	3.01 1011.90 3.04 661.65
23	7.89	0.00	0.00	559.370	3.02 559.28 3.09 683.76
24	8.38	2.09	153.0	526.779	3.02 529.72 3.09 680.21
25	8.92	0.80	373.8	494.078	3.03 495.74 3.10 683.38
26	9.02	3.13	100.7	488.555	3.03 491.69 3.09 679.21
27	20.09	0.21	937.6	217.076	3.06 218.31 3.22 694.08
28	9.53	1.52	194.6	462.294	3.03 464.30 3.10 683.83
29	8.24	0.02	13105	535.192	3.02 536.20 3.09 683.92
30	8.47	2.22	143.8	520.665	3.02 524.96 3.08 678.71
31	8.92	1.36	223.2	494.527	3.02 497.87 3.09 678.17
32	8.26	1.43	221.7	534.371	3.02 537.13 3.09 678.04
33	6.45	5.03	77.12	685.174	3.02 692.66 3.06 665.65
34	5.02	2.16	190.9	882.326	3.01 884.32 3.05 663.47
35	8.62	2.54	125.3	512.113	3.02 515.54 3.09 674.63
36	8.75	1.08	283.5	504.900	3.02 506.52 3.09 675.28
37	12.29	1.11	232.9	357.467	3.04 358.38 3.14 681.21
38	9.20	1.72	176.1	479.133	3.03 482.46 3.09 672.02
39	8.19	2.70	121.2	539.095	3.02 543.32 3.08 670.19
40	3.63	4.82	106.9	1222.32	3.01 1227.48 3.03 642.76
41	7.69	2.44	138.1	574.285	3.02 578.11 3.08 665.99
42	7.87	0.00	0.00	560.675	3.02 560.90 3.09 669.51
43	9.63	0.00	0.00	457.346	3.03 456.68 3.11 667.23
44	7.82	4.38	79.49	564.158	3.02 569.28 3.08 663.91
45	14.83	0.00	0.00	295.602	3.04 294.45 3.18 689.46
46	4.45	2.65	167.6	995.647	3.01 997.82 3.05 656.18
47	7.75	2.14	155.5	569.420	3.02 573.15 3.08 674.62
48	7.84	1.51	215.5	563.091	3.02 567.37 3.08 672.75
49	9.06	2.92	107.2	486.711	3.02 488.94 3.10 672.16

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
50	8.14	5.05	68.51	541.818	3.02 547.08 3.08 671.31

(1 missing vial)

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
52	12.39	0.00	0.00	354.548	3.04 353.63 3.15 673.73
53	14.76	0.77	304.9	296.956	3.04 298.50 3.16 670.60
54	8.63	3.68	88.81	510.820	3.02 515.02 3.09 667.76

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED

C14 CHI SQUARE IPA DATA PROCESSED

H3 IPA DATA PROCESSED

H3 CHI SQUARE IPA DATA PROCESSED

BKG IPA DATA PROCESSED

Calculations done with data on page 284

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A3a	412.164	0.4982	827.3063	95667784808641	1.588374E-10	37.0154
K3*A*A3b	411.681	0.5012	821.3907	94983713867296	1.577017E-10	36.7507
K3*A*B3a	366.907	0.5006	732.9345	84754845638222	1.407187E-10	32.7930
K3*A*B3b	352.042	0.5017	701.6982	81142757731130	1.347215E-10	31.3954
K3*A*C3a	526.540	0.5082	1036.0882	1.19810835E+14	1.989222E-10	46.3568
K3*A*C3b	508.429	0.5035	1009.7895	1.16769716E+14	1.93873E-10	45.1801
K3*A*A4	354.548	0.5014	707.1161	81769265237003	1.357617E-10	31.6379
K3*A*B4	296.956	0.5013	592.3718	68500506866981	1.137315E-10	26.5040
K3*A*C4	510.820	0.5001	1021.4357	1.1811646E+14	1.96109E-10	45.7012

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-R*PH3.00*P1	867.833	0.7649	1134.5705	1.3119911E+14	2.1783E-10	50.7631	-5.3079
W2-50-R*PH3.00*P2	580.497	0.5127	1132.2352	1.3092906E+14	2.17382E-10	50.6586	-5.0912
W2-50-R*PH3.00*E1	565.053	0.5004	1129.2026	1.3057838E+14	2.168E-10	50.5229	-4.8097
W2-50-R*PH3.00*E2	535.969	0.5012	1069.3715	1.2365964E+14	2.05312E-10	47.8459	0.7437
W2-50-R*PH3.00*S1	498.697	0.4552	1095.5558	1.2668754E+14	2.1034E-10	49.0175	-1.6867
W2-50-R*PH3.00*S2	495.760	0.4629	1070.9873	1.2384649E+14	2.05623E-10	47.9182	0.5937
W2-50-R*PH4.00*P1	668.077	0.5905	1131.3751	1.308296E+14	2.17217E-10	50.6201	-5.0113
W2-50-R*PH4.00*P2	1097.610	0.9613	1141.7976	1.3203483E+14	2.19218E-10	51.0864	-5.9787
W2-50-R*PH4.00*E1	567.815	0.4999	1135.8572	1.3134789E+14	2.18077E-10	50.8206	-5.4274
W2-50-R*PH4.00*E2	572.526	0.4988	1147.8067	1.3272971E+14	2.20371E-10	51.3553	-6.5365
W2-50-R*PH4.00*S1	487.465	0.4407	1106.1153	1.2790861E+14	2.12367E-10	49.4899	-2.6668
W2-50-R*PH4.00*S2	528.049	0.4824	1094.6289	1.2658036E+14	2.10162E-10	48.9760	-1.6007
W2-50-R*PH5.00*P1	603.659	0.5504	1096.7642	1.2682727E+14	2.10572E-10	49.0715	-1.7989
W2-50-R*PH5.00*P2	1006.080	0.9381	1072.4656	1.2401744E+14	2.05906E-10	47.9844	0.4565
W2-50-R*PH5.00*E1	559.370	0.5009	1116.7299	1.2913606E+14	2.14405E-10	49.9648	-3.6520
W2-50-R*PH5.00*E2	526.779	0.4986	1056.5162	1.2217309E+14	2.02844E-10	47.2707	1.9369
W2-50-R*PH5.00*S1	494.078	0.4645	1063.6771	1.2300115E+14	2.04219E-10	47.5911	1.2722
W2-50-R*PH5.00*S2	488.555	0.4608	1060.2322	1.226028E+14	2.03558E-10	47.4370	1.5920
W2-50-R*PH6.00*P1	217.076	0.2011	1079.4431	1.248243E+14	2.07246E-10	48.2965	-0.1911
W2-50-R*PH6.00*P2	462.294	0.4343	1064.4577	1.2309143E+14	2.04369E-10	47.6261	1.1997
W2-50-R*PH6.00*E1	535.192	0.5008	1068.6741	1.23579E+14	2.05178E-10	47.8147	0.8084
W2-50-R*PH6.00*E2	520.665	0.5004	1040.4976	1.2032073E+14	1.99769E-10	46.5540	3.4237
W2-50-R*PH6.00*S1	494.527	0.4693	1053.7545	1.2185373E+14	2.02314E-10	47.1472	2.1932
W2-50-R*PH6.00*S2	534.371	0.5215	1024.6807	1.1849171E+14	1.96732E-10	45.8464	4.8918
W2-50-R*PH7.00*P1	685.174	0.6500	1054.1138	1.2189528E+14	2.02383E-10	47.1633	2.1598
W2-50-R*PH7.00*P2	682.326	0.8360	1055.4139	1.2204562E+14	2.02633E-10	47.2214	2.0392
W2-50-R*PH7.00*E1	512.113	0.5011	1021.9776	1.1817913E+14	1.96213E-10	45.7254	5.1426
W2-50-R*PH7.00*E2	504.900	0.4986	1012.6354	1.1709881E+14	1.94419E-10	45.3074	6.0098
W2-50-R*PH7.00*S1	357.467	0.4167	857.8522	9.9200038E+13	1.64702E-10	38.3821	20.3764
W2-50-R*PH7.00*S2	479.133	0.4547	1053.7343	1.218514E+14	2.0231E-10	47.1463	2.1951
W2-50-R*PH8.00*P1	539.095	0.4867	1107.6536	1.280665E+14	2.12662E-10	49.5587	-2.8096
W2-50-R*PH8.00*P2	1222.320	1.0880	1123.4559	1.2991384E+14	2.15696E-10	50.2658	-4.2763
W2-50-R*PH8.00*E1	574.285	0.5014	1145.3630	1.3244712E+14	2.19902E-10	51.2459	-6.3097
W2-50-R*PH8.00*E2	560.675	0.5007	1119.7823	1.2948903E+14	2.14991E-10	50.1014	-3.9353
W2-50-R*PH8.00*S1	457.346	0.4011	1140.2294	1.3185348E+14	2.18917E-10	51.0162	-5.8332
W2-50-R*PH8.00*S2	564.158	0.4960	1137.4153	1.3152807E+14	2.18376E-10	50.8903	-5.7220
W2-50-R*PH9.00*P1	295.602	0.2588	1142.2025	1.3208165E+14	2.19295E-10	51.1045	-6.0163
W2-50-R*PH9.00*P2	995.647	0.8927	1115.3209	1.2897313E+14	2.14134E-10	49.9018	-3.5212
W2-50-R*PH9.00*E1	569.420	0.5007	1137.2479	1.3150871E+14	2.18344E-10	50.8828	-5.5564
W2-50-R*PH9.00*E2	563.091	0.5003	1125.5067	1.3015099E+14	2.1609E-10	50.3575	-4.6667
W2-50-R*PH9.00*S1	486.711	0.4298	1132.4128	1.3094959E+14	2.17416E-10	50.6665	-5.1077
W2-50-R*PH9.00*S2	541.818	0.4711	1150.1125	1.3299635E+14	2.20814E-10	51.4584	-6.7505

6/14/94 TD

See GC-11-143

for verification

of calculations

CNRA CONTROL

WPT 081

26 MAY 1993 TD

The 7th sample from the K3*A experiment was taken today. The pH and time of sampling can be found on pg 279-280. The sample weights can be found on the following page.

SAMPLE 7 WEIGHTS

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*7	7.7009	8.2011	0.5002
3	K3*A*B*7	7.7555	8.2546	0.4991
4	K3*A*B*7	7.7302	8.2283	0.4981

TD 5/26/93

The Liquid Scintillation Analysis of K3*A*...*5 and 6a (orb) has finished. The raw data, as well as the calculated U concentration are given below. The S# corresponds to the vial # on pg 282. Vial 1 is a blank.

Protocol #: 5 Name: U-233 3% 2 sigma 26-May-93 03:15
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.92 1.45	3.425 3.42	29.15 1.17	147.91 B
2	13.64	0.58 417.1	322.455 3.03	323.57 3.14	693.34
3	16.88	1.22 180.8	259.904 3.04	262.74 3.17	689.98
4	8.69	1.21 251.9	508.082 3.02	508.02 3.10	695.46
5	15.02	0.00 0.00	292.447 3.04	292.16 3.17	699.16
6	15.22	1.12 207.3	288.625 3.04	291.03 3.15	695.28
7	18.01	0.34 609.0	243.327 3.04	244.48 3.19	695.60
8	18.03	1.32 161.9	243.053 3.04	243.95 3.19	692.45
9	8.74	1.90 163.2	505.042 3.02	507.24 3.08	686.40
10	8.47	0.44 693.1	521.250 3.02	521.86 3.09	696.27

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A5	322.455	0.5002	644.6521	7.454608E+13	1.2377E-10	28.8431
K3*A*B5	259.904	0.5028	516.9133	5.977466E+13	9.9244E-11	23.1278
K3*A*C5	508.082	0.5001	1015.9608	1.174834E+14	1.9506E-10	45.4562
K3*A*A6a	292.447	0.4997	585.2451	6.767639E+13	1.1236E-10	26.1851
K3*A*A6b	288.625	0.4913	587.4720	6.79339E+13	1.1279E-10	26.2847
K3*A*B6a	243.327	0.4999	486.7514	5.62868E+13	9.3453E-11	21.7783
K3*A*B6b	243.053	0.5015	484.6520	5.604404E+13	9.305E-11	21.6843
K3*A*C6a	505.042	0.5014	1007.2637	1.164776E+14	1.9339E-10	45.0671
K3*A*C6b	521.250	0.5019	1038.5535	1.200959E+14	1.994E-10	46.4671

5/28/93 TD all the 8th sample of K3*A solutions was taken today and the pH measured (PH recorded on page 299-280)

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*8	7.7712	8.2704	0.4992
3	K3*A*B*8	7.7531	8.2548	0.5017
4	K3*A*C*8	7.7870	8.2868	0.4998

The Liquid Scintillation Analysis of K3*A*...*7 has completed. The raw data and U concentration calculations follow.

Protocol #: 5 Name: U-233 3% 2 sigma 28-May-93 04:32
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:2S%	CPMB B:2S%	CPMC C:2S%	SIS FLAG
1	999.98	18.84 1.46	3.308 3.48	28.81 1.18	145.09 B
2	16.65	0.98 223.4	263.599 3.04	265.48 3.17	680.51
3	19.94	0.47 420.5	219.561 3.05	220.53 3.21	686.04
4	8.62	1.35 227.6	512.933 3.02	514.69 3.09	681.18

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A7	263.599	0.5002	526.9872	6.093958E+13	1.0118E-10	23.5785
K3*A*B7	219.561	0.4991	439.9138	5.087062E+13	8.4461E-11	19.6827
K3*A*C7	512.933	0.4981	1029.7792	1.190813E+14	1.9771E-10	46.0745

6/14/94 TD Verification on page

GC-11-143.4.

(NWEA controlled copy 06)

29 May 1993 TD

The second set of samples from the reverse experiments was taken. The pH of each solution was also measured. The weights are in the table on the following page. The samples were counted using liquid scintillation. Several of the samples taken by pouring were possibly too large (too much volume) and this could effect the counting of these samples.

6/14/94 TD

Verification of calculations is on page 143-144 of GC-11.

(NWEA controlled copy 08)

SAMPLE WEIGHTS FOR REVERSE W2, 2nd sampling time (5th overall)

P=pouring, E=using Eppendorf, S=syringe. T for pH measurements=22.3 C

VIAL #	SAMPLE NAME	pH	WT VIAL (g)	WT VIAL+SAMPLE (g)	WT SAMPLE (g)
19	W2-50-R*PH3.00*P3	2.95	7.8130	9.2113	1.3983
20	W2-50-R*PH3.00*P4		7.7734	8.8614	1.0880
21	W2-50-R*PH3.00*E3		7.7455	8.2449	0.4994
22	W2-50-R*PH3.00*E4		7.7706	8.2729	0.5023
23	W2-50-R*PH3.00*S3		7.7763	8.0783	0.3020
24	W2-50-R*PH3.00*S4		7.7215	8.2636	0.5421
25	W2-50-R*PH4.00*P3	3.21	7.7583	8.6413	0.8830
26	W2-50-R*PH4.00*P4		7.7229	8.6562	0.9333
27	W2-50-R*PH4.00*E3		7.7367	8.2386	0.5019
28	W2-50-R*PH4.00*E4		7.7202	8.2222	0.5020
29	W2-50-R*PH4.00*S3		7.7949	8.2501	0.4552
30	W2-50-R*PH4.00*S4		7.7482	8.2014	0.4532
31	W2-50-R*PH5.00*P3	3.75	7.7801	8.6397	0.8596
32	W2-50-R*PH5.00*P4		7.7914	8.8354	1.0440
33	W2-50-R*PH5.00*E3		7.7852	8.2885	0.5033
34	W2-50-R*PH5.00*E4		7.7048	8.2070	0.5022
35	W2-50-R*PH5.00*S3		7.7394	8.1717	0.4323
36	W2-50-R*PH5.00*S4		7.6662	8.1143	0.4481
37	W2-50-R*PH6.00*P3	4.27	7.7770	8.7360	0.9590
38	W2-50-R*PH6.00*P4		7.7778	8.7627	0.9849
39	W2-50-R*PH6.00*E3		7.7729	8.2714	0.4985
40	W2-50-R*PH6.00*E4		7.8151	8.3167	0.5016
41	W2-50-R*PH6.00*S3		7.7070	8.0189	0.3119
42	W2-50-R*PH6.00*S4		7.7451	8.1837	0.4386
43	W2-50-R*PH7.00*P3	7.79	7.7297	8.7263	0.9966
44	W2-50-R*PH7.00*P4		7.7310	8.7679	1.0369
45	W2-50-R*PH7.00*E3		7.7282	8.2274	0.4992
46	W2-50-R*PH7.00*E4		7.7423	8.2423	0.5000
47	W2-50-R*PH7.00*S3		7.8005	8.2742	0.4737
48	W2-50-R*PH7.00*S4		7.7801	8.0666	0.2865
49	W2-50-R*PH8.00*P3	9.29	7.7739	8.2488	0.4749
50	W2-50-R*PH8.00*P4		7.7509	9.1045	1.3536
51	W2-50-R*PH8.00*E3		7.7047	8.2053	0.5006
52	W2-50-R*PH8.00*E4		7.7679	8.2686	0.5007
53	W2-50-R*PH8.00*S3		7.7054	8.2102	0.5048
54	W2-50-R*PH8.00*S4		7.7795	8.2356	0.4561
55	W2-50-R*PH9.00*P3	8.94	7.7763	9.5030	1.7267
56	W2-50-R*PH9.00*P4		7.7499	8.9770	1.2271
57	W2-50-R*PH9.00*E3		7.7356	8.2358	0.5002
58	W2-50-R*PH9.00*E4		7.7852	8.2845	0.4993
59	W2-50-R*PH9.00*S3		7.7556	8.1805	0.4249
60	W2-50-R*PH9.00*S4		7.7678	8.1637	0.3959

30 May 1993 TD

The 9th sample was taken from each kinetics experiment solution
2 samples of each were taken and prepared for LSA.

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*9a	7.7334	8.2461	0.5127
3	K3*A*A*9b	7.7055	8.2187	0.5132
4	K3*A*B*9a	7.7116	8.2127	0.5011
5	K3*A*B*9b	7.7735	8.2821	0.5086
6	K3*A*C*9a	7.7867	8.2857	0.4990
7	K3*A*C*9b	7.7849	8.2885	0.5036

The weights are shown
in the Table.

31 May 1993 TD

The LSA of the samples has finished. Below are the raw data
& concentration calculations. S# corresponds to vial # on pg 288 or 286.

Protocol #: 5 Name: U-233 3% 2 sigma 31-May-93 03:42
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:25%	CPMB B:25%	CPMC C:25%	SIS	FLAG
1	999.98	19.11	1.45	3.239	3.51	29.03
2	17.80	0.00	0.00	246.424	3.04	247.15
3	21.44	1.45	136.0	204.130	3.05	205.44
4	8.64	3.34	96.91	511.113	3.02	517.27
(14 missing vials)						
19	3.48	3.30	154.1	1273.77	3.01	1279.88
20	2.79	5.98	100.5	1590.31	3.01	1595.70
21	7.78	1.19	271.6	568.483	3.02	569.69
22	7.81	2.91	115.9	567.312	3.01	569.18
23	13.25	3.75	70.39	332.459	3.03	337.46
24	7.29	0.00	0.00	606.912	3.01	604.99
25	4.63	2.92	149.9	957.668	3.01	963.20
26	4.05	0.00	0.00	1096.76	3.01	1096.90
27	7.42	3.80	92.85	595.818	3.02	599.27
28	7.56	0.07	4881.	585.253	3.02	586.18
29	8.95	2.90	108.7	493.856	3.02	496.33
30	8.63	1.97	158.9	512.404	3.02	514.54
31	4.73	3.72	118.4	936.719	3.01	941.80
32	3.96	1.34	339.8	1118.98	3.01	1120.72
33	7.93	0.00	0.00	557.164	3.02	556.85
34	7.80	3.32	102.5	567.658	3.01	570.07
35	9.60	2.34	128.1	459.782	3.02	462.01
36	9.19	0.00	0.00	480.874	3.02	482.29
37	4.20	2.79	164.0	1055.09	3.01	1059.30
38	4.18	0.02	17594	1063.03	3.00	1064.75
39	8.24	2.73	119.7	536.931	3.02	540.15
40	7.91	1.24	259.8	558.961	3.02	559.97
41	14.05	1.24	195.2	313.416	3.03	315.60
42	9.89	1.41	205.1	446.104	3.02	447.92
43	4.30	2.75	164.5	1031.18	3.01	1035.16
44	3.96	1.34	339.8	1119.49	3.01	1119.71
45	8.24	2.00	160.5	536.203	3.02	538.45
46	8.15	0.03	11516	542.651	3.02	542.38
47	9.86	3.00	100.4	448.079	3.02	451.40
48	15.40	0.50	458.4	285.397	3.03	286.43
49	8.08	0.07	4498.	546.761	3.02	547.33
50	2.73	5.79	104.4	1627.16	3.00	1633.98
51	7.41	0.32	1020.	597.031	3.02	599.04
52	7.45	1.56	214.7	593.405	3.02	596.61
53	7.59	0.00	0.00	582.268	3.02	584.28
54	8.39	4.25	78.85	527.154	3.02	531.64
55	2.33	7.07	94.96	1907.49	3.00	1916.47
56	3.18	7.62	76.23	1396.45	3.00	1407.76
57	7.34	1.32	253.4	602.211	3.02	607.07
58	7.50	2.09	161.8	589.561	3.02	593.10
59	8.87	0.00	0.00	498.001	3.02	497.01
60	9.63	2.28	131.5	458.651	3.02	462.66

SYSTEM NORMALIZED
 C14 IPA DATA PROCESSED
 C14 CHI SQUARE IPA DATA PROCESSED
 H3 IPA DATA PROCESSED
 H3 CHI SQUARE IPA DATA PROCESSED

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A8	246.424	0.4992	493.6378	5.708313E+13	9.4775E-11	22.0864
K3*A*B8	204.130	0.5107	399.7063	4.622111E+13	7.6741E-11	17.8837
K3*A*C8	511.113	0.4998	1022.6351	1.182551E+14	1.9634E-10	45.7548

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-R*PH3.00*P3	1273.770	1.0880	1170.7445	1.35382181E+14	2.247753E-10	52.3816	-8.6655
W2-50-R*PH3.00*P4	1590.310	1.3983	1137.3167	1.31516674E+14	2.183574E-10	50.8859	-5.5628
W2-50-R*PH3.00*E3	568.483	0.4994	1138.3320	1.31634076E+14	2.185523E-10	50.8314	-5.6571
W2-50-R*PH3.00*E4	567.312	0.5023	1129.4286	1.30604511E+14	2.16843E-10	50.5330	-4.8307
W2-50-R*PH3.00*S3	332.459	0.3020	1100.8576	1.27300625E+14	2.113575E-10	49.2547	-2.1788
W2-50-R*PH3.00*S4	606.912	0.5421	1119.5573	1.29463011E+14	2.149477E-10	50.0913	-3.9144
W2-50-R*PH4.00*P3	957.668	0.8830	1084.5617	1.25416206E+14	2.082288E-10	48.5256	-0.6663
W2-50-R*PH4.00*P4	1096.760	0.9333	1175.1420	1.35890696E+14	2.256106E-10	52.5783	-9.0737
W2-50-R*PH4.00*E3	595.818	0.5019	1187.1249	1.37276377E+14	2.279203E-10	53.1145	-10.1859
W2-50-R*PH4.00*E4	585.253	0.5020	1165.8426	1.34815342E+14	2.238342E-10	52.1622	-8.2105
W2-50-R*PH4.00*S3	493.856	0.4552	1084.9209	1.25457742E+14	2.082978E-10	48.5416	-0.6996
W2-50-R*PH4.00*S4	512.404	0.4532	1130.6355	1.30744068E+14	2.170747E-10	50.5870	-4.9427
W2-50-R*PH5.00*P3	936.719	0.8596	1089.7150	1.26012117E+14	2.092182E-10	48.7561	-1.1446
W2-50-R*PH5.00*P4	1116.980	1.0440	1071.8199	1.23942774E+14	2.057825E-10	47.9555	0.5164
W2-50-R*PH5.00*E3	557.164	0.5033	1107.0217	1.28013421E+14	2.12541E-10	49.5305	-2.7509
W2-50-R*PH5.00*E4	567.658	0.5022	1130.3425	1.30710188E+14	2.170184E-10	50.5739	-4.9155
W2-50-R*PH5.00*S3	459.782	0.4323	1063.5716	1.22888956E+14	2.041988E-10	47.5864	1.2820
W2-50-R*PH5.00*S4	480.874	0.4481	1073.1399	1.24095415E+14	2.060359E-10	48.0145	0.3939
W2-50-R*PH6.00*P3	1055.090	0.9590	1100.1981	1.27224363E+14	2.112309E-10	49.2252	-2.1176
W2-50-R*PH6.00*P4	1063.030	0.9849	1079.3279	1.24810973E+14	2.072239E-10	48.2914	-0.1805
W2-50-R*PH6.00*E3	536.931	0.4985	1077.0933	1.24552572E+14	2.067949E-10	48.1914	0.0270
W2-50-R*PH6.00*E4	558.961	0.5016	1114.3561	1.28861554E+14	2.139491E-10	49.8586	-3.4317
W2-50-R*PH6.00*S3	313.416	0.3119	1004.8605	1.16199745E+14	1.929267E-10	44.9596	6.7314
W2-50-R*PH6.00*S4	446.104	0.4386	1017.1090	1.17616127E+14	1.952783E-10	45.5076	5.5945
W2-50-R*PH7.00*P3	1031.180	0.9966	1034.6980	1.19650077E+14	1.986553E-10	46.2946	3.9620
W2-50-R*PH7.00*P4	1119.490	1.0369	1079.6509	1.24848328E+14	2.072899E-10	48.3058	-0.2104
W2-50-R*PH7.00*E3	536.203	0.4992	1074.1246	1.24209281E+14	2.062248E-10	48.0586	0.3025
W2-50-R*PH7.00*E4	542.651	0.5000	1085.3020	1.2550181E+14	2.083709E-10	48.5587	-0.7350
W2-50-R*PH7.00*S3	448.079	0.4737	945.9130	1.09383191E+14	1.816092E-10	42.3221	12.2028
W2-50-R*PH7.00*S4	285.397	0.2865	996.1501	1.15192489E+14	1.912543E-10	44.5698	7.5399
W2-50-R*PH8.00*P3	546.761	0.4749	1151.3182	1.33135767E+14	2.210456E-10	51.5124	-6.8624
W2-50-R*PH8.00*P4	1627.160	1.3536	1202.0981	1.39007841E+14	2.30795E-10	53.7644	-11.5757
W2-50-R*PH8.00*E3	597.031	0.5006	1192.6308	1.37913069E+14	2.269774E-10	53.3608	-10.6970
W2-50-R*PH8.00*E4	593.405	0.5007	1185.1508	1.37048092E+14	2.275412E-10	53.0261	-10.0027
W2-50-R*PH8.00*S3	582.268	0.5048	1153.4628	1.33383762E+14	2.214573E-10	51.6083	-7.0615
W2-50-R*PH8.00*S4	527.154	0.4561	1155.7860	1.33652418E+14	2.219034E-10	51.7123	-7.2771
W2-50-R*PH9.00*P3	1907.49	1.7267	1104.7026	1.27745252E+14	2.120957E-10	49.4267	-2.5357
W2-50-R*PH9.00*P4	1396.450	1.2271	1138.0083	1.31596646E+14	2.184902E-10	50.9169	-5.6270
W2-50-R*PH9.00*E3	602.211	0.5002	1203.9404	1.39220862E+14	2.311487E-10	53.8668	-11.7467
W2-50-R*PH9.00*E4	589.561	0.4993	1180.7751	1.36542096E+14	2.267011E-10	52.8303	-9.5965
W2-50-R*PH9.00*S3	498.001	0.4249	1172.0428	1.35532319E+14	2.250246E-10	52.4397	-8.7860
W2-50-R*PH9.00*S4	458.651	0.3959	1158.5021	1.33966505E+14	2.224249E-10	51.8338	-7.5292

6/14/94 TD

Verification can

be found on

pg GC-11-142.5

NWA controlled copy

copy 081

2 June 1993 TD

The 10th sample of the kinetics experiment was taken today. The pH, T, and time were recorded on pages 279-280. The weights of the samples are given below.

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*10	7.8246	8.3255	0.5009
3	K3*A*B*10	7.8406	8.3418	0.5012
4	K3*A*C*10	7.7976	8.2980	0.5004

"cocktail" was added and the samples were allowed to sit until counting time.

The liquid scintillation analysis of the 9th samples has finished. The raw data and calculation of concentrations are shown below.

Protocol #: 5 Name: U-233 3% 2 sigma 02-Jun-93 04:12
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:25%	CPMB B:25%	CPMC C:25%	SIS FLAG
1	999.98	19.11 1.45	3.294 3.48	29.22 1.17 143.49	B
2	18.12	2.19 99.77	242.015 3.04	244.89 3.18 674.64	
3	17.78	2.49 89.36	246.706 3.04	249.23 3.18 668.35	
4	22.80	1.90 102.2	191.618 3.05	194.50 3.23 670.64	
5	22.29	0.00 *****	196.078 3.05	196.75 3.24 682.02	
6	8.70	4.11 79.84	507.970 3.02	515.26 3.07 675.23	
7	8.39	4.73 71.56	527.099 3.02	532.87 3.07 671.33	

SYSTEM NORMALIZED
 C14 IPA DATA PROCESSED
 C14 CHI SQUARE IPA DATA PROCESSED
 H3 IPA DATA PROCESSED
 H3 CHI SQUARE IPA DATA PROCESSED

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A*9a	242.015	0.5127	472.0402	5.458563E+13	9.0629E-11	21.1201
K3*A*A*9b	246.706	0.5132	480.7210	5.558946E+13	9.2295E-11	21.5085
K3*A*B*9a	191.618	0.5011	382.3947	4.421924E+13	7.3417E-11	17.1091
K3*A*B*9b	196.078	0.5086	385.5250	4.458121E+13	7.4018E-11	17.2492
K3*A*C*9a	507.970	0.4990	1017.9760	1.177164E+14	1.9544E-10	45.5464
K3*A*C*9b	527.099	0.5036	1046.6620	1.210336E+14	2.0095E-10	46.8299

6/14/94 TD

Verification on

GC-11-193.4

NWA controlled copy

copy 081

1 June 1993 TD

6.0 Kg 0.1M NaNO₃ was prepared in 1000 g portions.
 The amount of NaNO₃ needed is 8.499 g and this is diluted to 1000 g with nanopure H₂O.

Bottle #	Am. Na NO ₃ used
1	8.5000
2	8.4998
3	8.4994
4	8.5000
5	8.5004
6	8.5000

1.0 6/2/93
 1000 mL 0.1 M HNO₃ was prepared by diluting 63 mL concentrated (16M) HNO₃ to 1000 mL in a volumetric flask with distilled H₂O.
 lot # (HNO₃): FL-04-390

6/2/93 TD

1000 mL 0.1 M HNO_3 was prepared by diluting 6.3 mL concentrated HNO_3 to 1000 mL with deionized H_2O in a volumetric flask

1000 mL 0.001 M HNO_3 was prepared by diluting 10 mL 0.1 M HNO_3 prepared earlier to 1000 mL with deionized H_2O

1000 mL 0.02 M HNO_3 was prepared by diluting 20 mL of the 1 M HNO_3 prepared earlier to 1000 mL with deionized H_2O .

6/3/93 TD

~2 L Nano pure H_2O was degassed. This was done by boiling it for at least 30 minutes and then cooling it with a rubber stopper tightly placed in the opening of the flask. This water was used to prepare 500 mL each of 1.0 M, 0.5 M, and 0.1 M NaHCO_3 solutions. (See Table 1 for the weights of NaHCO_3 needed and used) in volumetric flasks. The finished solutions were stored in tightly capped, glass reagent bottles.

TABLE 1 NaHCO_3 wts needed and used

Molarity	wt. needed (g)	wt. used (g)
1.0	42.005	42.0082
0.5	21.003	21.0049
0.1	4.2010	4.2044

The LSA of K3A*...*10 samples has finished.

HOWEVER The results for both this set and set 9 might not be correct. The lid of the scintillator was opened, allowing light inside and this could have caused more H^+ counts than should be there. The samples will be rerun.

S#	TIME	CPMA A:25%	CPMB B:25%	CPMC C:25%	SIS FLAG
61	5.03	0.00 0.00 882.153	3.01 880.81 3.05 706.73		
62	4.75	2.04 207.3 932.865	3.01 936.10 3.04 708.19		
63	4.68	1.92 220.5 948.361	3.01 950.74 3.04 709.68		
64	4.86	1.56 264.6 911.680	3.01 913.45 3.05 710.66		
65	4.80	1.61 258.5 925.407	3.01 929.18 3.04 704.36		
66	4.16	7.42 68.01 1067.78	3.01 1077.72 3.03 706.79		
67	4.19	5.33 90.66 1058.44	3.01 1065.03 3.03 707.08		
68	4.15	3.87 121.5 1068.43	3.01 1071.23 3.04 709.28		
69	4.06	2.16 211.4 1092.19	3.01 1095.36 3.04 709.94		
70	6.05	3.63 106.9 731.906	3.01 737.75 3.05 707.02		
71	6.23	0.00 0.00 710.348	3.01 709.99 3.07 711.12		
72	8.32	2.62 123.7 531.240	3.02 535.77 3.07 707.21		
73	7.81	0.00 0.00 566.391	3.02 566.99 3.08 715.58		
74	5.10	5.30 82.61 868.630	3.01 875.59 3.04 706.90		
75	4.87	0.70 580.2 909.391	3.01 911.72 3.05 709.91		
76	5.63	7.09 60.85 788.385	3.01 794.92 3.04 705.05		
77	5.32	1.85 214.9 832.955	3.01 837.64 3.05 708.18		
78	5.86	0.95 390.3 755.909	3.01 759.18 3.05 710.58		
79	5.93	1.72 217.5 747.961	3.01 749.89 3.06 707.36		
80	6.15	1.31 278.8 720.442	3.01 720.41 3.06 707.70		
81	6.00	4.48 88.51 738.365	3.01 744.64 3.05 708.19		
82	4.98	5.08 86.79 889.435	3.01 893.56 3.05 708.48		
83	5.21	4.40 96.59 850.224	3.01 854.96 3.05 707.55		
84	5.33	4.81 88.10 830.824	3.01 837.51 3.04 711.21		
85	5.12	3.44 121.9 866.006	3.01 872.84 3.04 709.09		

(5 missing vials)

91	18.98	1.21 171.5 231.059	3.04 231.90 3.20 706.33
92	23.64	0.00 0.00 184.851	3.05 184.29 3.26 709.84
93	8.60	2.73 117.1 513.609	3.02 517.05 3.08 709.40

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED

C14 CHI SQUARE IPA DATA PROCESSED

H3 IPA DATA PROCESSED

K3*A*A10

K3*A*B10

K3*A*C10

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A10	231.059	0.5009	461.2877	5.334224E+13	8.8564E-11	20.6390
K3*A*B10	184.851	0.5012	368.8168	4.264913E+13	7.081E-11	16.5016
K3*A*C10	513.609	0.5004	1026.3969	1.186902E+14	1.9706E-10	45.9231

6/14/94 Verification

on pages 143-144

6/15/94

6/15/94

CWWRA controlled copy 08/

4 JUNE 1993 TD

~2 L Deionized water was degassed by boiling it in Erlenmeyer flasks, and cooling while the mouth was stoppered shut. This was used to make the NaHCO_3 solutions with molarities given below. The weights needed and used are also given.

Molarity	wt. Needed (g)	wt. Used (g)
1.0	42.0050	42.0082
0.5	21.0030	21.0049
0.1	4.2010	4.2044
0.05	2.1000	2.1005
0.01	0.4201	0.4213
0.005	0.2100	0.2116
0.001	0.0420	0.0436

6/4/93 TD

The 3rd set of Reverse samples was taken from W2-50.

The weights are as follows.

P=pouring, E=using Eppendorf, S=syringe. T for pH measurements=20.6 C

VIAL #	SAMPLE NAME	pH	WT VIAL (g)	WT VIAL+SAMPLE (g)	WT SAMPLE (g)
19	W2-50-R*PH5.00*P5	2.90	7.8529	8.5328	0.6799
20	W2-50-R*PH5.00*P6		7.9050	8.3600	0.4550
21	W2-50-R*PH5.00*E5		7.8696	8.3736	0.5040
22	W2-50-R*PH5.00*E6		7.9590	8.4620	0.5030
23	W2-50-R*PH5.00*S5		7.7796	8.1674	0.3878
24	W2-50-R*PH5.00*S6		7.8979	8.3838	0.4859
25	W2-50-R*PH6.00*P5	3.16	7.8147	8.6965	0.8818
26	W2-50-R*PH6.00*P6		7.8587	8.6624	0.8037
27	W2-50-R*PH6.00*E5		7.8361	8.3373	0.5012
28	W2-50-R*PH6.00*E6		8.3311	8.8310	0.4999
29	W2-50-R*PH6.00*S5		7.9552	8.4438	0.4886
30	W2-50-R*PH6.00*S6		7.8938	8.3704	0.4766
31	W2-50-R*PH5.00*P5	3.69	7.7797	8.3383	0.5586
32	W2-50-R*PH5.00*P6		7.8032	8.5011	0.6979
33	W2-50-R*PH5.00*E5		7.8485	8.3526	0.5041
34	W2-50-R*PH5.00*E6		7.8420	8.3449	0.5029
35	W2-50-R*PH5.00*S5		7.8117	8.2729	0.4612
36	W2-50-R*PH5.00*S6		7.8555	8.2934	0.4379
37	W2-50-R*PH6.00*P5	4.29	7.8409	8.2083	0.3674
38	W2-50-R*PH6.00*P6		7.8138	8.6345	0.8207
39	W2-50-R*PH6.00*E5		7.8563	8.3595	0.5032
40	W2-50-R*PH6.00*E6		7.8889	8.3918	0.5029
41	W2-50-R*PH6.00*S5		7.7993	8.1858	0.3865
42	W2-50-R*PH6.00*S6		7.8242	8.2491	0.4249
43	W2-50-R*PH7.00*P5	7.72	7.7975	8.7619	0.9644
44	W2-50-R*PH7.00*P6		7.8237	8.9746	1.1509
45	W2-50-R*PH7.00*E5		7.8490	8.3516	0.5026
46	W2-50-R*PH7.00*E6		7.8825	8.3858	0.5033
47	W2-50-R*PH7.00*S5		7.9043	8.3383	0.4340
48	W2-50-R*PH7.00*S6		7.7679	8.1984	0.4305
49	W2-50-R*PH8.00*P5	9.3	7.8307	8.5044	0.6737
50	W2-50-R*PH8.00*P6		7.8527	9.1235	1.2708
51	W2-50-R*PH8.00*E5		7.8587	8.3603	0.5016
52	W2-50-R*PH8.00*E6		7.8559	8.3576	0.5017
53	W2-50-R*PH8.00*S5		7.8225	8.2278	0.4053
54	W2-50-R*PH8.00*S6		7.8331	8.2478	0.4147
55	W2-50-R*PH9.00*P5	8.90	7.8711	8.9039	1.0328
56	W2-50-R*PH9.00*P6		7.7895	8.8406	1.0511
57	W2-50-R*PH9.00*E5		7.7740	8.2769	0.5029
58	W2-50-R*PH9.00*E6		7.8216	8.3247	0.5031
59	W2-50-R*PH9.00*S5		7.8042	8.2511	0.4469
60	W2-50-R*PH9.00*S6		7.8309	8.2424	0.4115

5 June 1993 TD

The 11th sample of each of the Kinetics solutions was taken

TD 6/5/93
Sample Weights for 5 June 1993

VIAL #	SAMPLE NAME	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
2	K3*A*A*11	7.8230	8.3263	0.5033
3	K3*A*B*11	7.8579	8.3604	0.5025
4	K3*A*C*11	7.8339	8.3355	0.5016

7 June 1993 TD

The recounting of the 9th and 10th K3*A samples has been completed. The raw data and results of calculations follow.

S#	TIME	CPMA A:25%	CPMB B:25%	CPMC C:25%	SIS FLAG
61	5.07	0.19 2028.874.597	3.01 877.15	3.05 707.67	
62	4.68	1.59 265.3 947.312	3.01 950.92	3.04 711.68	
63	4.71	3.79 116.6 940.409	3.01 945.11	3.04 710.57	
64	4.79	8.63 55.89 925.277	3.01 936.15	3.03 703.61	
65	4.75	5.07 89.15 932.674	3.01 937.54	3.04 705.67	
66	4.18	6.22 79.29 1061.96	3.01 1071.43	3.03 711.06	
67	4.05	5.55 89.04 1095.65	3.01 1102.30	3.03 709.70	
68	4.10	5.99 82.84 1082.98	3.01 1088.51	3.03 708.48	
69	4.08	2.92 159.4 1090.51	3.00 1095.94	3.03 709.71	
70	6.04	4.37 90.44 732.812	3.01 737.49	3.05 711.13	
71	6.27	3.67 104.2 705.976	3.01 711.79	3.05 710.12	
72	8.35	0.00 0.00 529.100	3.02 528.92	3.09 728.35	
73	7.73	2.47 136.0 571.788	3.02 575.44	3.07 722.91	
74	5.02	5.76 77.43 882.542	3.01 889.36	3.04 709.86	
75	4.93	4.19 104.0 898.305	3.01 903.48	3.04 711.16	
76	5.66	1.71 224.8 783.281	3.01 785.22	3.05 712.69	
77	5.52	0.00 0.00 803.769	3.01 803.14	3.06 712.25	
78	5.79	3.14 125.1 764.761	3.01 766.25	3.06 709.79	
79	5.96	7.21 58.48 743.865	3.01 752.64	3.04 707.48	
80	5.95	1.03 357.9 743.944	3.01 745.55	3.06 714.87	
81	5.86	0.15 2477.756.271	3.01 755.39	3.06 714.26	
82	4.98	5.96 75.43 889.254	3.01 896.94	3.04 713.94	
83	5.16	2.96 140.3 858.900	3.01 861.74	3.05 717.42	
84	5.30	5.39 79.96 835.375	3.01 842.00	3.04 712.73	
85	5.12	1.37 292.6 864.854	3.01 868.50	3.05 715.45	
86	3.88	2.51 188.3 1143.79	3.01 1150.04	3.03 721.55	
87	3.78	2.29 208.1 1174.13	3.01 1176.20	3.04 718.47	

(3 missing vials)

91	17.79	0.00 0.00 246.857	3.04 248.33	3.18 680.23	K3*A*A*9a
92	17.50	2.86 78.90 250.885	3.04 254.86	3.16 668.31	9b
93	22.56	0.00 0.00 193.871	3.05 192.00	3.26 682.85	B 9a
94	22.23	2.37 83.95 196.795	3.05 199.46	3.22 671.02	9b
95	8.26	4.71 72.34 535.263	3.02 542.02	3.07 668.21	C 9a
96	8.38	0.91 340.8 528.627	3.01 531.94	3.08 673.87	9b
97	18.89	1.46 144.6 232.248	3.04 233.21	3.20 705.23	K3*A*A*10a
98	24.91	0.17 1030.175.367	3.05 175.65	3.27 711.25	B * 10a
99	8.60	2.14 147.4 513.978	3.02 516.20	3.08 710.27	C * 10

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED

C14 CHI SQUARE IPA DATA PROCESSED

H3 IPA DATA PROCESSED

H3 CHI SQUARE IPA DATA PROCESSED

BKG IPA DATA PROCESSED

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A*9a	246.857	0.5127	481.4843	5.567773E+13	9.2442E-11	21.5426
K3*A*A*9b	250.885	0.5132	488.8640	5.65311E+13	9.3859E-11	21.8728
K3*A*B*9a	193.871	0.5011	386.8908	4.473916E+13	7.4281E-11	17.3103
K3*A*B*9b	196.795	0.5086	386.9347	4.474424E+13	7.4289E-11	17.3123
K3*A*C*9a	535.263	0.4990	1072.6713	1.240412E+14	2.0595E-10	47.9936
K3*A*C*9b	528.627	0.5036	1049.6962	1.213844E+14	2.0153E-10	46.9656

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A*10	233.210	0.5009	465.5820	5.383882E+13	8.9389E-11	20.8311
K3*A*B*10	175.650	0.5012	350.4589	4.052626E+13	6.7286E-11	15.6803
K3*A*C*10	513.978	0.5004	1027.1343	1.187754E+14	1.972E-10	45.9561

6/14/94 TD

Verification

can be found

on p 142-143 of

GC-11. P143-144

CNRRA controlled

copy 081

6 June 1993 TD

The 12th Kinetics samples were taken today (2 from each bottle). The pH was also measured and can be found on page 279-280.

SAMPLE NAM	WT VIAL	WT VIAL+SAMPLE	WT SAMPLE
K3*A*A*12a	7.8303	8.3344	0.5041
K3*A*A*12b	7.9246	8.4285	0.5039
K3*A*B*12a	7.8786	8.3828	0.5042
K3*A*B*12b	7.9081	8.4126	0.5045
K3*A*C*12a	7.7627	8.2660	0.5033
K3*A*C*12b	7.8125	8.3166	0.5041

The 11th samples as well as the final sampling of W2-50 have finished counting. The raw data and calculations follow.

Protocol #: 5 Name: U-233 3% 2 sigma 07-Jun-93 16:55
 Region A: LL-UL= 0.0-100. Lcr= 0 Bkg= 0.00 %2 Sigma=0.25
 Region B: LL-UL=100.-350. Lcr= 0 Bkg= 0.00 %2 Sigma=3.00
 Region C: LL-UL= 0.0-2000 Lcr= 0 Bkg= 0.00 %2 Sigma=0.10
 Time =999.99 QIP = SIS
 U-233 3% 2 sigma error for 50 ppb experiments

S#	TIME	CPMA A:25%	CPMB B:25%	CPMC C:25%	SIS FLAG
1	999.98	18.91 1.45	2.963 3.67	27.77 1.20	137.56 B
2	19.44	0.22 904.7	225.638 3.04	226.09 3.20	710.13 K3*A*A*11
3	23.92	1.53 122.3	182.823 3.05	185.44 3.22	711.57 B11
4	8.61	0.00 0.00	513.297 3.02	509.86 3.10	718.52 C11
(2 missing vials)					
7	5.66	2.99 131.8	782.726 3.01	786.90 3.05	698.71 W2-50+ph300#P5
8	8.47	1.39 223.3	521.830 3.02	523.35 3.08	714.34
9	7.61	0.00 0.00	581.005 3.02	582.48 3.08	716.12
10	7.83	3.44 98.68	564.853 3.02	569.80 3.07	699.66
11	9.94	1.01 283.0	444.321 3.02	445.67 3.10	717.69
12	8.10	0.00 0.00	546.296 3.01	547.54 3.08	711.56
13	4.52	0.33 1241.	980.223 3.01	980.86 3.05	683.97
14	4.96	2.25 183.6	893.206 3.01	896.42 3.05	692.59
15	7.74	3.70 92.80	571.197 3.02	576.11 3.07	699.32
16	8.05	3.07 107.9	549.335 3.02	553.85 3.07	681.50
17	8.26	4.57 74.00	535.294 3.02	541.84 3.07	707.94
18	8.69	1.11 275.0	508.775 3.02	512.85 3.08	712.51
19	7.05	3.64 98.60	627.959 3.01	633.08 3.06	703.66
20	5.67	4.19 96.58	780.811 3.01	787.75 3.05	690.13
21	7.78	1.39 232.6	568.502 3.02	572.75 3.07	709.43
22	8.07	0.66 470.8	548.214 3.02	549.55 3.08	707.60
23	9.21	0.00 0.00	479.773 3.02	481.35 3.09	713.29
24	9.16	2.26 134.8	482.626 3.02	484.90 3.09	709.73
25	10.90	2.83 100.3	405.386 3.02	408.01 3.10	714.83
26	4.82	2.25 186.9	920.066 3.01	926.80 3.04	690.80
27	8.00	2.84 116.7	553.037 3.01	557.11 3.07	705.89
28	7.92	0.66 480.8	558.527 3.02	559.86 3.08	711.34
29	10.88	0.75 358.2	405.769 3.02	407.25 3.11	715.06
30	9.88	1.33 216.5	446.835 3.02	448.34 3.10	714.07
31	4.11	4.44 107.5	1079.28 3.01	1083.67 3.04	677.19
32	3.46	0.74 646.3	1281.43 3.01	1285.82 3.03	666.49
33	7.90	0.33 960.8	559.822 3.02	561.22 3.08	710.57
34	7.79	4.06 84.80	567.769 3.02	575.44 3.06	706.84
35	10.70	0.34 798.0	412.644 3.02	414.85 3.10	726.23
36	9.70	3.15 96.23	455.594 3.02	457.00 3.08	708.84

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)
K3*A*A11	225.638	0.5033	448.3171	5.1842352E+13	8.6074E-11	20.0586
K3*A*B11	182.823	0.5025	363.8269	4.2072096E+13	6.98524E-11	16.2784
K3*A*C11	513.297	0.5016	1023.3194	1.1833428E+14	1.96471E-10	45.7855

SAMPLE NAME	CPM B	WEIGHT (g)	MASS	ATOM CONV	MOLE CONV	ppb U(233)	% U(233) LOST
W2-50-R*H3.00*P3	782.726	0.5799	1151.2369	1.3312637E+14	2.2103E-10	51.5088	-6.8549
W2-50-R*H3.00*P3	521.830	0.4550	1146.8791	1.3262245E+14	2.2018E-10	51.3138	-6.4504
W2-50-R*H3.00*P3	581.005	0.5040	1152.7877	1.333057E+14	2.21328E-10	51.5781	-6.9988
W2-50-R*H3.00*P3	564.853	0.5030	1122.9682	1.2985744E+14	2.15803E-10	50.2439	-4.2310
W2-50-R*H3.00*P3	444.321	0.3878	1145.7478	1.3249162E+14	2.19976E-10	51.2632	-6.3454
W2-50-R*H3.00*P3	546.296	0.4858	1124.2972	1.3001112E+14	2.15858E-10	50.3034	-4.3544
W2-50-R*H4.00*P3	893.206	0.8818	1009.5521	1.1674226E+14	1.93827E-10	45.1695	6.2960
W2-50-R*H4.00*P3	571.197	0.8037	1111.3674	1.2851596E+14	2.13375E-10	49.7249	-3.1543
W2-50-R*H4.00*P3	549.335	0.5012	1139.6588	1.3178751E+14	2.18807E-10	50.9907	-5.7802
W2-50-R*H4.00*P3	535.294	0.4999	1098.8888	1.2707307E+14	2.1098E-10	49.1666	-1.9961
W2-50-R*H4.00*P3	508.775	0.4886	1095.5869	1.2668882E+14	2.10342E-10	49.0180	-1.6877
W2-50-R*H5.00*P3	627.959	0.4766	1067.5094	1.2344432E+14	2.04955E-10	47.7626	0.9165
W2-50-R*H5.00*P3	780.811	0.5586	1124.1658	1.2998593E+14	2.15833E-10	50.2975	-4.3422
W2-50-R*H5.00*P3	568.502	0.6979	1118.8007	1.2937552E+14	2.14802E-10	50.0575	-3.8442
W2-50-R*H5.00*P3	548.214	0.5041	1127.7564	1.3041114E+14	2.16522E-10	50.4582	-4.6755
W2-50-R*H5.00*P3	479.773	0.5029	1090.1054	1.2805726E+14	2.09293E-10	48.7736	-1.1808
W2-50-R*H5.00*P3	482.626	0.4612	1040.2710	1.2029453E+14	1.99725E-10	46.5439	3.4447
W2-50-R*H6.00*P3	405.386	0.4379	1102.1375	1.2744862E+14	2.11603E-10	49.3119	-2.2976
W2-50-R*H6.00*P3	920.066	0.3674	1103.3914	1.2753963E+14	2.11844E-10	49.3680	-2.4140
W2-50-R*H6.00*P3	553.037	0.8207	1121.0747	1.2963848E+14	2.15238E-10	50.1592	-4.0553
W2-50-R*H6.00*P3	558.527	0.5032	1099.0401	1.2709046E+14	2.11009E-10	49.1734	-2.0101
W2-50-R*H6.00*P3	405.769	0.5029	1110.6124	1.2842865E+14	2.1323E-10	49.6911	-3.0842
W2-50-R*H6.00*P3	446.835	0.3865	1049.8551	1.2140281E+14	2.01565E-10	46.9727	2.5551
W2-50-R*H7.00*P3	1079.280	0.4249	1051.6239	1.2160735E+14	2.01805E-10	47.0519	2.3910
W2-50-R*H7.00*P3	1281.430	0.9644	1119.1207	1.2941253E+14	2.14864E-10	50.0718	-3.8739
W2-50-R*H7.00*P3	559.822	1.1509	1113.4156	1.287528E+14	2.13769E-10	49.8165	-3.3444
W2-50-R*H7.00*P3	567.769	0.5026	1113.8520	1.2880326E+14	2.13852E-10	49.8361	-3.3849
W2-50-R*H7.00*P3	412.644	0.5033	1128.0926	1.3045001E+14	2.1658E-10	50.4732	-4.7067
W2-50-R*H7.00*P3	455.594	0.4340	950.7926	1.0994748E+14	1.82546E-10	42.5405	11.7499
W2-50-R*H8.00*P3	823.803	0.4305	1058.2904	1.2237825E+14	2.03185E-10	47.3501	1.7722
W2-50-R*H8.00*P3	1586.680	0.6737	1222.8039	1.4140221E+14	2.3477E-10	54.7108	-13.4975
W2-50-R*H8.00*P3	602.887	1.2708	1248.5678	1.4438149E+14	2.39717E-10	55.8635	-15.8889
W2-50-R*H8.00*P3	587.577	0.5016	1201.9278	1.3898815E+14	2.30762E-10	53.7768	-11.5599
W2-50-R*H8.00*P3	491.037	0.5017	1191.1042	1.3773654E+14	2.28684E-10	53.2925	-10.5553
W2-50-R*H8.00*P3	487.545	0.4053	1211.5396	1.4009963E+14	2.32608E-10	54.2068	-12.4520
W2-50-R*H9.00*P3	1252.970	0.4147	1175.6571	1.3595026E+14	2.25719E-10	52.6014	-9.1215
W2-50-R*H9.00*P3	593.681	1.0328	1218.2707	1.40878E+14	2.339E-10	54.5080	-13.0768
W2-50-R*H9.00*P3	588.914	1.0511	1192.0559	1.3784659E+14	2.28867E-10	53.3351	-10.6436
W2-50-R*H9.00*P3	535.247	0.5029	1180.5150	1.3651202E+14	2.26651E-10	52.8187	-9.5724
W2-50-R*H9.00*P3	501.918	0.5031	1170.5705	1.3536206E+14	2.24742E-10	52.3738	-8.6494
W2-50-R*H9.00*P3		0.4469	1197.6885	1.3849793E+14	2.29948E-10	53.5871	-11.1664
W2-50-R*H9.00*P3		0.4115	1219.7278	1.410465E+14	2.3418E-10	54.5732	-13.2120

6/15/94 TD
 Calculations

verified on pages
 143-144 of
 G1-011.

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9 JUNE 1993 JB

URANIUM SORPTION EXPERIMENT B-IB:

Kd vs pH: Equilibrium with atmospheric pCO₂; Initial $\Sigma U = 50$ ppbWRITTEN BY: R.T. PABALAN
REVISION NO.: 0DATE WRITTEN: May 27, 1993
DATE REVISED:

OBJECTIVE:

- To investigate the importance of uranium sorption on the zeolite mineral clinoptilolite as a function of solution pH and total uranium concentration. Experimental data will be correlated with uranium aqueous speciation.
- To investigate reversibility and reproducibility of uranium sorption reactions.

Note: This procedure is similar to that for Expt. B-I. Teflon (FEP) bottles are used here instead of polypropylene (PP) to minimize uranium losses to container walls. Smaller solution volumes and zeolite weights are also used compared to B-I. In addition, liquid scintillation counting, instead of alpha-spectrometry, will be used to measure uranium concentrations. Instead of NaHCO₃ solid, aqueous solutions of NaHCO₃ are used here to raise the starting pH of the uranium solutions.

EQUIPMENT:

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

SUPPLIES:

- pH buffer (pH = 2,4,7,9,10)
40 60-ml FEP bottles (to contain experimental mixtures, control solutions, and B-IB*IU)
1 2000-ml teflon 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 HNO₃ solution into scintillation vial)
various Eppendorf micropipets (fixed- or variable-volume; for adding HNO₃ or NaHCO₃ solutions to uranium solutions)
various scintillation vials
various weighing paper
various Na⁺-clinoptilolite (CDV*100/200*UC*WA*HL*CPT*Naf)
various reagent grade NaHCO₃
various 500 ppb U stock solution prepared from 50 ppm ²³³U commercial spike
4 L 0.1 M NaNO₃ stock solution

- 1000 ml stock solution of 1.0 M HNO₃
1000 ml stock solution of 0.1 M HNO₃
1000 ml stock solution of 0.02 M HNO₃
500 ml stock solution of 1.0 M NaHCO₃
500 ml stock solution of 0.5 M NaHCO₃
500 ml stock solution of 0.1 M NaHCO₃
500 ml stock solution of 0.05 M NaHCO₃
500 ml stock solution of 0.01 M NaHCO₃
500 ml stock solution of 0.005 M NaHCO₃
500 ml ultrapure water

PROCEDURE:

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

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

- Initial $\Sigma U = 50$ ppb
- Initial pH = 2.0 to 9.0, every 0.25 pH unit; adjustments made with HNO₃ or NaHCO₃
- Initial volume = 50 ml
- Ionic strength = 0.1 M NaNO₃
- Wt. zeolite to use = 0.100 ± 0.001
- Initial [Na⁺] = 0.1 M NaNO₃ + [NaHCO₃] added
- pCO₂ = atmospheric = $10^{-3.48}$ bar

a) Prepare 2000-g of 50 ppb U solution in a pre-cleaned 2-liter teflon bottle by diluting 200 g of a 500 ppb stock solution (in 0.1 M NaNO₃ matrix; prepared previously from commercial 50 ppm ²³³U spike) to a total of 2000 g by carefully taring 0.1 M NaNO₃ solution into the teflon bottle on a Mettler 4600 balance.

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

Into each of 10 60-ml FEP bottle labeled B-IB-C*pHi [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. These are control solutions to determine uranium loss to the container walls as a function of pH.

Transfer the remaining solution into a 60-ml FEP bottle labeled B-IB*IU. Take two 0.5-ml samples from B-IB*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-IB-IU*a (or b)] and pre-weighed scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO₃. Reweigh each vial. Homogenize the mixture and save for later analysis of uranium concentration by liquid scintillation counting.

c) For each solution B-IB*pHi and B-IB-C*pHi:

Adjust the pH of each solution to the approximate value *i* by adding HNO₃ solution or NaHCO₃ solution with an Eppendorf micropipet. The concentration and approximate amount to be added is given in Table B-IB-1. Swirl 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 ~120 rpm. Leave the bottles on the shaker for about ten days to allow the solutions to reach equilibrium with atmospheric CO₂(g).

d) Measure and record the pH of each solution B-IB*pHi and B-IB-C*pHi. *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-IB*pHi and B-IB-C*pHi, take 2 0.5-ml sample with an Eppendorf pipet, transfer into pre-labeled [e.g., B-IB*IU-phi*a (or b)] scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO₃. Reweigh each vial. Homogenize the mixtures 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.

e) Tare 0.100 ± 0.001 gm of Na-clinoptilolite onto weighing paper, and carefully transfer into each of the B-IB*pHi (not the B-IB-C*pHi) bottles. Swirl each bottle by hand, replace the cover, then place on the shaker.

f) After equilibrium is reached (at least 10 days), take 2 0.5-ml samples from each bottle B-IB*phi and B-IB-C*pHi with an Eppendorf pipet, transfer into pre-labeled [e.g., B-IB-phi*a (or b)] and pre-weighed scintillation vials containing 5 ml of cocktail and 0.5 ml of 0.02 M HNO₃. Reweigh each vial. Homogenize the mixtures and save for later analysis of uranium concentration by liquid scintillation counting.

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

g) Analyze the U concentration by liquid scintillation counting.

Hold Point. Check quality of experimental data.

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.

Procedure for reversibility and reproducibility experiments will be written later.

PREPARATION:

- 1. Preclean:
 - 40 60-ml FEP bottles (to contain experimental mixtures, control solutions, and B-IB-*IU)
 - 1 2000-ml teflon bottle (for preparation of 50 ppb U solution)
- 2. Prepare:
 - 500 ppb U stock solution prepared from 50 ppm ²³³U commercial spike
 - 4 L 0.1 m NaNO₃ stock solution
 - 1000 ml stock solution of 1.0 m HNO₃
 - 1000 ml stock solution of 0.1 m HNO₃
 - 1000 ml stock solution of 0.02 m HNO₃
 - 500 ml stock solution of 1.0 M NaHCO₃ (42.005 g in 500 ml solution)
 - 500 ml stock solution of 0.5 M NaHCO₃ (21.003 in 500 ml solution)
 - 500 ml stock solution of 0.1 M NaHCO₃ (4.201 g in 500 ml solution)
 - 500 ml stock solution of 0.05 M NaHCO₃ (2.100 g in 500 ml solution)
 - 500 ml stock solution of 0.01 M NaHCO₃ (0.4201 g in 500 ml solution)
 - 500 ml stock solution of 0.005 M NaHCO₃ (0.2100 g in 500 ml solution)

The NaHCO₃ solutions should be prepared with degassed deionized water and kept in tightly-capped glass reagent bottles.

Solution pH	Volume of NaHCO ₃ needed, ml	Molarity of NaHCO ₃ solution to use
8.00	0.403	0.1
8.25	0.139	0.5
8.50	0.248	0.5
8.75	0.228	1.0
9.00	0.436	1.0
[9.25]		
[9.50]	[2.00]	[1.0]

TABLE B-IB-1 ↑

Table B-IB-1. Amount of reagent grade HNO₃ or NaHCO₃ solutions to add to 50 ml 0.1 m NaNO₃ 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.

Solution pH	Volume of HNO ₃ needed, ml	Molarity of HNO ₃ to use
2.00	0.602	1.0
2.25	0.336	1.0
2.50	0.187	1.0
2.75	0.103	1.0
3.00	0.563	0.1
3.25	0.299	0.1
3.50	0.151	0.1
3.75	0.0673	0.1
4.00	0.102	0.02
Solution pH	Volume of NaHCO ₃ needed, ml	Molarity of NaHCO ₃ solution to use
4.25	0.120	0.005
4.50	0.417	0.005
4.75	0.293	0.01
5.00	0.342	0.01
5.25	0.371	0.01
5.50	0.391	0.01
5.75	0.409	0.01
6.00	0.429	0.01
6.25	0.460	0.01
6.50	0.102	0.05
6.75	0.120	0.05
7.00	0.151	0.05
7.25	0.207	0.05
7.50	0.306	0.05
7.75	0.242	0.10

9 JUNE 1993 ID

Experiment B-IB was begun. 2000g 50 ppb ^{233}U solution was prepared by diluting 200g 500 ppb ^{233}U to 2000 g using 0.1M NaNO_3 (prepared 1 June 1993 pg 290).

Actual Weight 500 ppb ^{233}U = 200.39 g
Final Weight = 2000.2 g

NaNO_3 solutions used were #1 & #2.

50g of the ^{233}U solution (50 ppb) was weighed out into labeled 60mL Teflon (FEP) bottles.

The pH was adjusted using the values on page 300-301, rounded to the nearest 0.1. The actual adjustments are given below.

pH NAME ID	Adjustment	Molarity
2.00	0.600 mL	1.0 M HNO_3
2.25	0.340	1.0 M
2.50	0.190	1.0 M
2.75	0.100	1.0 M
3.00	0.560	0.1
3.25	0.300	0.1
3.50	0.150	0.1
3.75	0.070	0.1
4.00	0.100	0.02
4.25	0.120	0.005 M NaHCO_3
4.50	0.420	0.05
4.75	0.300	0.1
5.00	0.350	0.1
5.25	0.370	0.1
5.50	0.390	0.1
5.75	0.410	0.1

continued on next page

pH	Adjustment (uL)	Molarity
6.00	0.430	0.01
6.25	0.460	0.01
6.50	0.100	0.05
6.75	0.120	0.05
7.00	0.150	0.05
7.25	0.210	0.05
7.50	0.310	0.05
7.75	0.240	0.1
8.00	0.400	0.1
8.25	0.140	0.5
8.50	0.250	0.5
8.75	0.230	1.0
9.00	0.440	1.0
9.50	2.00	1.0

Two samples of the remaining 50 ppb were taken for liquid scintillation analysis. The samples were 500 uL samples and each was weighed, acidified and 5 mL cocktail was added.

The sample weights were

	wt. vial (g)	wt. sample + vial (g)	wt. sample (g)
6/14/93 ID B-IB*IVa	7.3527	7.8526	0.4999
IVb	7.2733	7.7729	0.4996

The bottles were covered with a kimwipe and placed on a gyrating shaker set @ 120 rpm

THIS NOTEBOOK WILL BE CONTINUED IN

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