

Geochemistry
Research
Laboratory
Notebook
Vol. GC-07

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Geochemistry
Research Lab Notebook
Vol. GC-07

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PM Paula Muller Student Scientist
TD - Todd Dietrich Student Scientist

Pages 1 through 304 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Hek
SWR5-QA
10/14/94

3/21/92

1525 PM

H⁺-VO₂²⁺ ION EXCHANGE EXPERIMENT H/U-I
continued (see GC-05)

pH measurement of H/U*1a... 1l solutions

| solution | pH | temp °C | solution | pH | temp °C |
|----------|------|---------|---------------|------|---------|
| U/H-I*1a | 1.78 | 21.5 | U/H-I*1g | 2.96 | 21.6 |
| b | 1.98 | 21.4 | h | 3.20 | 21.4 |
| c | 2.17 | 21.4 | i | 3.36 | 21.4 |
| d | 2.37 | 21.4 | j | 3.56 | 21.5 |
| e | 2.58 | 21.6 | k | 3.77 | 21.4 |
| f | 2.77 | 21.6 | l | 3.96 | 21.6 |
| | | | pH buffer 4.0 | 3.99 | 23.5 |

The solutions' pH differ by > 0.01 units so the solutions were re covered with Kimwipes + placed back on the gyratory shaker.

3/22/92

0815 PM

pH measurement of H/U*1a... 1l solutions

| solution | pH | temp °C | solution | pH | temp °C |
|----------|------|---------|---------------|------|---------|
| U/H-I*1a | 1.80 | 21.3 | U/H-I*1g | 2.97 | 21.5 |
| b | 2.00 | 21.1 | h | 3.18 | 21.2 |
| c | 2.16 | 21.6 | i | 3.38 | 21.4 |
| d | 2.36 | 21.5 | j | 3.59 | 21.2 |
| e | 2.57 | 21.6 | k | 3.77 | 21.5 |
| f | 2.76 | 21.6 | l | 3.97 | 21.4 |
| | | | pH buffer 4.0 | 4.00 | 25.7 |

The Δ pH readings is down to 0.00 to one 0.03, so clinoptilolite addition can be done.

0920 PM H/U-I-1-D:

0.010 \pm 0.001 gm of H⁺ clinoptilolite was added into each bottle after being quickly tared on weighing paper.

Weights of H^+ di-nephthylate used:

| | |
|----------|----------|
| a 0.0102 | g 0.0109 |
| b 0.0101 | h 0.0108 |
| c 0.0101 | i 0.0109 |
| d 0.0107 | j 0.0107 |
| e 0.0108 | k 0.0110 |
| f 0.0106 | l 0.0110 |

1010 PM The bottles were covered with kumwipes + placed on the gyratory shaker, set at 125 rpm.

1015 PM Three 1-ml aliquots from $H/U-I*100ppm*101$ or 2 were taken with an Eppendorf pipet + transferred into 10-ml volumetric flasks. The aliquots were diluted to the mark with a solution of 0.0556 M tartaric acid + 0.0556 M triethanolamine (T) so that the final background electrolyte concentration = 0.05 M T. The solutions were transferred into 15-ml PP bottles labeled $H/U-I*100ppm*101$ or $2, 3$ for later U analysis with the polarograph.

$H/U-I-4$: Control Experiments to Evaluate Uranium Losses from Solution Due to Adsorption or Precipitation

1045 PM Two 100 mL aliquots of solution $H/U-I*100ppm*101$ or 2 were taken by volumetric pipet + put in 125-ml PP bottles labeled $H/U-I-4 a$ (or b).

Two 1-ml aliquots were taken from both solutions with an Eppendorf pipet, transferred into 10-ml volumetric flasks, and diluted as above with T (see GC-05-169). The solutions were transferred into 15-ml polypropylene bottles labeled

$H/U-I-4*100a*101$ or 2 or $H/U-I-4*100b*101$ or 2
For later U analysis with the polarograph.

pH of solutions

| | |
|----------------|-------------|
| $H/U-I-4*100a$ | 3.97 @ 21.2 |
| b | 4.06 @ 21.2 |

3/23/92

1354 PM

Experiment A-II-3 continued

10-ml samples of solutions A-II-3* pH were taken by max pipet + put in 15-ml PP bottles labeled A-II-3* $pH*U$. 500 ml of 1.05 M tartaric acid/triethanolamine (see GC-05-252) was added to each sample by micro pipet.

The Orion 920A pH/ISE meter was calibrated over 5 pH buffers (Fisher 2.0, 4.0, 7.0, 9.0, + 10.0) + the A-II-3* pH solutions read.

| solution | pH | temp °C |
|------------------|-------|---------|
| A-II-3* pH 2.5 | 2.93 | 23.7 |
| 3.0 | 3.71 | 23.8 |
| 3.5 | 4.65 | 23.8 |
| 4.0 | 6.05 | 24.1 |
| 4.5 | 6.50 | 24.0 |
| 5.0 | 6.72 | 23.9 |
| 5.5 | 6.76 | 24.0 |
| 6.0 | 6.90 | 24.0 |
| 6.5 | 6.89 | 24.1 |
| 7.0 | 7.07 | 24.2 |
| 7.5 | 7.31 | 24.4 |
| 8.0 | 7.85 | 23.9 |
| 8.5 | 8.55 | 24.3 |
| 9.0 | 8.94 | 24.2 |
| pH buffer 10.0 | 10.02 | 25.1 |

1553 PM The solutions were placed back on the gyratory shaker

TD 0900

Test were done on the Polarograph with the Tartaric Acid/Triethanolamine at concentrations from ~1ppb - ~10ppb. The results were non-linear. There was also a test to investigate the effect of a constant ionic strength of .1M Na⁺ on PK Height of samples from 10ppb - 100ppb. plots for all runs can be found in Polarographic Analyses of Uranium Vol 9.

3/24/92 PM Expt. A-1-3 continued
0840

10-ml aliquots were taken from solutions A-1-3 & pH with an Eppendorf max pipet and transferred into 15 mL PP bottles labeled A-1-3 & pH & U. 500 μ l of 1.05M IT (see GC-05-252) was added to each sample by Eppendorf micropipet.

0927PM 1 mL of 4.1M NaNO₃ prepared previously (see GC-05-94) was diluted to 100 mL in a 100 mL volumetric flask with ultrapure water (> 17 MS resistance). The solution was transferred into an appropriately labeled PP bottle & put in the glove box.

0954PM The 290A Orion pH/ISE meter was calibrated with five Fisher buffers (pH = 2.0, 4.0, 7.0, 9.0 & 10.0). 10 mL of the degassed ultrapure water was put in a 15 mL PP bottle via max pipet. 25 μ l of the 0.041M NaNO₃ solution prepared above was added to the water. Final Na concentration: 1×10^{-3} M. The pH of the water/NaNO₃ and the pH of the A-1-3 & pH solutions were read.

| solution | pH | temp °C |
|--|------|---------|
| 1012 PM 1×10^{-3} NaNO ₃ | 5.70 | 23.0 |
| A-1-3 & pH 2.5 | 2.99 | 23.4 |
| 3.0 | 3.73 | 22.9 |
| 3.5 | 4.53 | 23.3 |
| 4.0 | 6.03 | 23.4 |
| 4.5 | 6.90 | 23.2 |
| 5.0 | 7.13 | 23.1 |
| 5.5 | 7.01 | 23.1 |
| 6.0 | 7.08 | 23.3 |
| 6.5 | 6.95 | 23.6 |
| 7.0 | 7.43 | 23.5 |
| 7.5 | 7.42 | 23.5 |
| 8.0 | 7.20 | 23.8 |
| 8.5 | 7.25 | 23.5 |
| 9.0 | 7.40 | 23.6 |
| 110 PM pH buffer 10.0 | 9.95 | 22.8 |

3/25/92 TD 0930

Prepared new standards with PIPES and oxine. U was added (in the amounts ^{TD 3/25/92} amounts given below) to 1 mL PIPES (1.0M) and 200 μ l .01M oxine in a 100 mL volumetric flask. The solution was diluted to the mark with Ultrapure H₂O. The standard was transferred to an appropriately labeled 125 mL PP bottle.

| SOLN CONCENTRATION | AMT STD NEEDED |
|--------------------|----------------|
| 50 ppm | 5 mL 100 ppm |
| 40 ppm | 4 mL 1000 ppm |
| 30 ppm | 3 mL 1000 ppm |
| 20 ppm | 2 mL 1000 ppm |
| 10 ppm | 1 mL 1000 ppm |
| 500 ppb | 1 mL 50 ppm |
| 400 ppb | 1 mL 40 ppm |
| 300 ppb | 1 mL 30 ppm |
| 200 ppb | 1 mL 20 ppm |

| SOLN CONC | AMT STD USED |
|-----------|---------------|
| 100 ppb | 1 ml 10 ppm |
| 50 ppb | 0.1 ml 50 ppm |
| 30 ppb | 0.1 ml 30 ppm |
| 10 ppb | 0.1 ml 10 ppm |
| 5 ppb | 1 ml 500 ppm |
| 1 ppb | 1 ml 100 ppb |

The 50-1 ppb standards were analyzed on the Polarograph using DPS at 120s and 60s deposition time.

3/27/92 PK

~~2/27/92 PK~~

Expt H/U-I-1 continued

1230

The 100 μ L micropipet tip will be calibrated by adding 2 M HNO_3 solution (GC-05-301) dropwise to a 5 mL volumetric flask. This calculation will determine the approximate volume in mL of HNO_3 added to solutions U/H-I*1a...1d.

$$\frac{392 \text{ drops}}{5 \text{ mL}} = \frac{1 \text{ drop}}{x \text{ mL}} \quad x = 0.0128 \text{ mL/drop}$$

3/30/92 TD 0830

Prepared U standards from 10 ppb - 50 ppm according to the table below. All had 5 mL 1.0 M π added and all were diluted to 100 mL

| | |
|------------------------------------|-----------------------------------|
| 50 ppm \rightarrow 5 mL 1000 ppm | 200 ppb \rightarrow 1 mL 20 ppm |
| 40 ppm \rightarrow 4 mL 1000 ppm | 100 ppb \rightarrow 1 mL 10 ppm |
| 30 ppm \rightarrow 3 mL 1000 ppm | 50 ppb \rightarrow .1 mL 50 ppm |
| 20 ppm \rightarrow 2 mL 1000 ppm | 30 ppb \rightarrow .1 mL 30 ppm |
| 10 ppm \rightarrow 1 mL 1000 ppm | 10 ppb \rightarrow .1 mL 10 ppm |
| 50 ppb \rightarrow 1 mL 50 ppm | |
| 40 ppb \rightarrow 1 mL 40 ppm | |
| 30 ppb \rightarrow 1 mL 30 ppm | |

1220 PK Expt H/U-I-1 continued

H/U-I-1-E - samples taken from solutions with H^+ form clinoptilolite

After at least five days have passed since the clinoptilolite was added to solutions U/H-I*1a...1d, 2 1-mL aliquots were taken from each solution by Eppendorf micropipet. The aliquots were put into 10-mL volumetric flasks and brought to volume with 0.0556 M tartaric acid / 0.0556 M triethanolamine (0.0556 M π , see GC-05-169).

The solutions were shaken to mix & transferred into 15-mL PP bottles labeled H/U-I*1a*1 (or 2) etc. The samples will be analyzed for uranium content with the polarograph. Note: the GC-05-169 solution was used up halfway through the dilution process so the remaining dilutions were done with that made March 19 (see GC-05-301).

1440 PK H/U-1-4 continued -

Two additional 1-mL aliquots from each solution H/U-1-4*100 a and b were taken by Eppendorf pipet, transferred into 10-mL volumetric flasks, and diluted to volume with the 0.0556 M π used above. The solutions were transferred into 15-mL PP bottles labeled H/U-1-4*100 a * FU1 (or 2) and H/U-1-4*100 b * FU1 (or 2). For later U analysis with the polarograph.

1458 PK The pH and temperature of solutions H/U-1*1a...1d and H/U-1-4*100 a & b were measured and recorded. See tables on the following pages.

| Solution Label | HNO ₃ added | | Initial pH/°C (after CO ₂ equil.) | Measured Final 4/2/92 TD | |
|----------------|------------------------|----------------------|--|-----------------------------|----------------|
| | # of drops | Approx. vol. (ml) | | pH/°C | U ppm *1 *2 |
| H/U-I*1a | 71 | 0.91 | 1.80/21.3 | 1.76 / 22.6 | 93.6 94.2 |
| H/U-I*1b | 42 | 0.54 | 2.00/21.1 | 1.94 / 22.4 | 94.2 93.6 |
| H/U-I*1c | 54 | 0.69 | 2.16/21.6 | 2.16 / 22.5 | 94.2 93.8 |
| H/U-I*1d | 33 | 0.42 | 2.36/21.5 | 2.36 / 22.5 | 94.4 95.0 |
| H/U-I*1e | 46 | 0.59 | 2.57/21.6 | 2.54 / 22.4 | 94.0 95.3 |
| H/U-I*1f | 26 | 0.33 | 2.76/21.6 | 2.76 / 22.4 | 94.0 95.0 |
| H/U-I*1g | 80 | 1.02 | 2.97/21.5 | 2.94 / 22.5 | 93.1 95.0 |
| H/U-I*1h | 50 | 0.60 | 3.18/21.2 | 3.14 / 22.5 | 95.0 98.4 |
| H/U-I*1i | 35 | 0.45 | 3.38/21.4 | 3.30 / 22.5 | 96.3 97.7 |
| H/U-I*1j | 41 | 0.52 | 3.59/21.2 | 3.48 / 22.5 | 95.3 96.4 |
| H/U-I*1k | 19 | 0.24 | 3.77/21.5 | 3.67 / 22.5 | 95.3 97.7 |
| H/U-I*1l | 7 | 0.09 | 3.97/21.4 | 3.81 / 22.7 | 95.3 98.2 |

Measured
4/3/92
TD

APM Data table for Experiment #10-I-4
U(ppm) for (WITAC Socin) 4/2/92
(1) 95.34
(2) 96.3
(3) 96.7

24 Data table for Experiment H10-I-4

| Solution Label | Initial | | Final | |
|--------------------|-----------|-------------|-----------|-------------------|
| | pH/°C | *1 U ppm *2 | pH/°C | *1 U ppm *2 |
| H/U-I-4*100a*1 & 2 | 3.97/21.2 | 95.9 97.3 | 3.91/22.6 | 94.8 98.9 |
| H/U-I-4*100b*1 & 2 | 4.06/21.2 | 96.1 99.8 | 4.02/22.7 | 92.7 99.1 3/38/92 |
| H/U-I-4*10a*1 & 2 | - | - | - | - |
| H/U-I-4*10b*1 & 2 | - | - | - | - |
| H/U-I-4*1a*1 & 2 | - | - | - | - |
| H/U-I-4*1b*1 & 2 | - | - | - | - |

1030 TD

The standards made earlier were used to calibrate the Polarograph from 10ppb - 500ppb. 2 curves, one from 10ppb - 100ppb and one from 100ppb - 400ppb, were generated using DPS. The lower concentrations were run on Cell 1 and the higher on cell 2. The solutions A-II-3 *pH*U were analysed for U concentration.

Cell 1

| SAMPLE | PEAK HEIGHT (nAmps) | | CONC (ppb) |
|--------------|---------------------|------|------------|
| | RUN1 | RUN2 | |
| 10ppb | 2.2 | 2 | |
| 30ppb | 5.11 | 4.04 | |
| 50ppb | 9.2 | 9.6 | |
| 100ppb | 18.3 | 18.9 | |
| A-II-3 *pH*U | 3.65 | 3.5 | 20.7 |
| *pH 4.5*U | 1.6 | 2.7 | 13.1 |
| *pH 5.0*U | 2.5 | 2.2 | 14.2 |
| *pH 5.5*U | 3.1 | 2.8 | 17.3 |
| *pH 6.0*U | 5.3 | 3.7 | 25.4 |
| *pH 6.5*U | 5.2 | 4.2 | 26.4 |
| *pH 7.0*U | 7.8 | 6.6 | 39.8 |
| 30 ppb U | 5.2 | 5.0 | (28.7) |

CELL 2

| SAMPLE | PEAK HEIGHT (nAmps) | | CONC (ppb) |
|---------------------------------------|---------------------|-------|------------|
| | RUN 1 | RUN 2 | |
| 100 ppb U | 17.7 | 17.8 | |
| 200 ppb U | 29.1 | 30.0 | |
| 300 ppb U | 39.2 | 39.4 | |
| 400 ppb U | 47.4 | 49.1 | |
| A-I-3* $\text{pH}2.5 \times \text{U}$ | 41.2 | 41.6 | 324 |
| * $\text{pH}3.0 \times \text{U}$ | 44.1 | 44.9 | 360 |
| * $\text{pH}3.5 \times \text{U}$ | 20.6 | 17.5 | 110 |
| * $\text{pH}7.5 \times \text{U}$ | 17.6 | 22.7 | 119 |
| * $\text{pH}8.0 \times \text{U}$ | 35.3 | 22.6 | 194 |
| * $\text{pH}8.5 \times \text{U}$ | 42.7 | 43.4 | 343 |
| * $\text{pH}9.0 \times \text{U}$ | 47.1 | 47.9 | 396 |
| 300 ppb U | 37.4 | 36.8 | (276) |

Plots, Parameters and calibration curves can be found in Polarographic Analyses of Uranium, vol. 10.

3/31/92 TD

Analyzed solution A-I-3* $\text{pH}i \times \text{U}$ on the polarograph using DPS. The method was the same as yesterday except higher concentrations ($>100 \text{ ppb}$) were run at 0.5 deposition.

CELL 1 (10ppb - 100ppb)

| SAMPLE | PEAK HEIGHT (nAmps) | | CONC (ppb) |
|---------------------------------------|---------------------|-------|------------|
| | RUN 1 | RUN 2 | |
| 10 ppb | 2.1 | 2.2 | |
| 30 ppb | 5.9 | 4.9 | |
| 50 ppb | 8.5 | 8.1 | |
| 100 ppb | 15.2 | 14.8 | |
| A-I-3* $\text{pH}3.5 \times \text{U}$ | 12.5 | 12.4 | 79.9 |
| * $\text{pH}4.0 \times$ | .62 | .96 | (3.95) |
| * $\text{pH}4.5 \times$ | .67 | 1.01 | (2.55) |
| * $\text{pH}5.0 \times$ | 1.1 | 1.4 | (4.92) |

| SAMPLE | PEAK HEIGHT (nAmps) | | CONC (ppb) |
|--|-----------------------|-------|------------|
| | RUN 1 | RUN 2 | |
| A-I-3* $\text{pH}5.5 \times \text{U}$ | 1.8 | | (9.87) |
| * $\text{pH}6.0 \times$ | 1.8 | 1.8 | (7.23) |
| * $\text{pH}6.5 \times$ | 2.3 | 2.3 | 11.6 |
| * $\text{pH}7.0 \times$ | TD 2.5 3.5 3/31/92 | 3.7 | 18.9 |
| * $\text{pH}7.5 \times$ | 3.4 | 3.6 | 18.3 |
| * $\text{pH}8.0 \times$ | 3.5 | 4.2 | 20.4 |
| * $\text{pH}8.5 \times$ | 9.2 | 11.0 | 62.4 |
| * $\text{pH}9.0 \times$ | 12 | 10.7 | 71.6 |
| 500 ppb | 6.7 | 6.6 | [38.4] |
| ALL CONC. BELOW 10 (IN PARENTHESES) ARE OUT OF THE CALIBRATION RANGE | | | |

CELL 2 (100 - 500 ppb, 0.5 dep)

| SAMPLE | PEAK HEIGHT | | CONC (ppb) |
|--|-------------|-------|------------|
| | RUN 1 | RUN 2 | |
| 100 ppb | 4.6 | 4.4 | |
| 200 ppb | 10.9 | 11.4 | |
| 300 ppb | 20 | 21.4 | |
| 400 ppb | 26 | 24.4 | |
| 500 ppb | 29.5 | 29.6 | |
| A-I-3* $\text{pH}2.5 \times \text{U}$ | 16.1 | 16.6 | 245 |
| * $\text{pH}3.0 \times \text{U}$ | 11.3 | 12.9 | 188 |
| A-II-3* $\text{pH}7.5 \times \text{U}$ | 5.6 | 6.3 | 123 |
| * $\text{pH}8.0 \times$ | 13.1 | 14.2 | 207 |
| * $\text{pH}8.5 \times$ | TD 20.6 | 22.7 | 329 |
| 200 ppb U | 12.6 | 13.0 | (196) |

Plots, parameters, and calibration curves can be found in Polarographic Analyses of Uranium Vol 10.

4/1/92 TD

Reran solutions A-II-3* pH_i *U on the polarograph using DPS using the same method as before (3-31-92). CELL 2 was used for low concentrations, CELL 1 for high.

CALIBRATION

| U ppb 1 | Pk Height 2 | Regress-Ord2 3 | U ppb 5 | Pk Height 6 | Regress-Ord2 7 |
|------------|----------------|-------------------|------------|----------------|-------------------|
| 10 | 2 | -4.1417 | 100 | 5.6 | 29.8 |
| 10 | 2.3 | 6.4715 | 100 | 5.4 | 10.429 |
| 30 | 6 | -0.050998 | 300 | 15.5 | 0.41415 |
| 30 | 5.6 | 0 | 300 | 16.8 | 0 |
| 50 | 9.3 | 0 | 400 | 19.9 | 0 |
| 50 | 8.3 | 0 | 400 | 19.4 | 0 |
| 100 | 19.3 | 0 | 500 | 23.9 | 0 |
| 100 | 18.5 | 0 | 500 | 22.8 | 0 |
| | | 0 | | | 0 |
| | | 0 | | | 0 |
| | | 0.99802 | | | 0.99642 |

CELL 2

CELL 1

ANALYSIS

| A-II-3* pH_i *U 1 | Pk Ht(nAmps) 2 | Pk Ht2(nAmp) 3 | U CONC(ppb) 4 |
|------------------------|-------------------|-------------------|------------------|
| 2.5 | 16.9 | 17.5 | 331.7 |
| 3 | 11.7 | 13.7 | 229 |
| 3.5 | 12 | 12.7 | 68 |
| 4 | 0.66 | 1.1 | 1.51 |
| 4.5 | 1.1 | 1 | 2.6 |
| 5 | 1.5 | 1.2 | 4.5 |
| 5.5 | 1.4 | 1.4 | 4.81 |
| 6 | 1.6 | 2.2 | 7.97 |
| 6.5 | 2.5 | 3.2 | 13.9 |
| 7 | 3.6 | 3.7 | 18.8 |
| 7.5 | 3.1 | 3.1 | 15.4 |
| 8 | 3.5 | 3.7 | 18.5 |
| 8.5 | 10.1 | 10.9 | 58.2 |
| 9 | 10.9 | 10.7 | 59.8 |
| 300 PPB | 14.9 | 16 | 290 |
| 30 PPB U | 5.2 | 5.1 | 27.8 |

NOTE - ALL CONCENTRATIONS BELOW 10 PPB WERE OUT OF THE CALIBRATION RANGE

SAMPLES RUN on cell 1 had 0s deposition times while cell 2 had 60s deposition. A-II-3^{TD} Plots, parameters and calibration curves can be found in Polarographic Analysis of Uranium, Vol 10.

4/2/92 PM

0902

Expt A-II-3 continued

310-ml aliquots of stock solution A-II-3 were taken by max pipet & put in 15-ml PP bottles labeled A-II-3-51 (or 52 or 53). 500 μ l of 1.05 M TT (see GC-05 2/19/92) was added to each sample taken. The solutions were shaken to mix & will be analyzed for U by polarograph.

0929 PM The pH of solutions A-II-3* pH_i will be read and the pH & temp ($^{\circ}$ C) recorded. The pH of each solution will then be adjusted down ~ 1 pH unit & the final pH recorded. 1.0, 0.5, 0.1, & 0.05 M HNO_3 (see GC-05 3/20/92) will be used for adjustments.

| solution | pH | $^{\circ}$ C | adj pH | added |
|------------------|------|--------------|--------|----------------------------|
| A-II-3* pH_2 5 | 2.84 | 21.1 | 2.40 | 6d 1.0M, 2d 0.5M HNO_3 |
| * pH_3 0 | 3.55 | 21.1 | 3.00 | 2d 1.0M, 2d 0.5M |
| * pH_3 5 | 4.50 | 21.1 | 3.50 | 1d 0.5M, 5d 0.1M |
| * pH_4 0 | 5.84 | 21.1 | 3.99 | 5d 0.1M |
| * pH_4 5 | 6.55 | 21.5 | 4.55 | 2d 0.1M |
| * pH_5 0 | 6.81 | 21.2 | 4.74 | 3d 0.1M |
| * pH_5 5 | 6.83 | 21.6 | 5.28 | 2d 0.1M, 1d 0.05M HNO_3 |
| * pH_6 0 | 6.96 | 21.6 | 5.91 | 4d 0.05M |
| * pH_6 5 | 6.98 | 21.6 | 6.48 | 2d 0.05M |
| * pH_7 0 | 7.15 | 21.7 | 6.83 | 1d 0.05M |
| * pH_7 5 | 7.38 | 21.7 | 7.12 | 1d 0.05M |
| * pH_8 0 | 7.83 | 21.7 | 7.53 | 2d 0.05M |
| * pH_8 5 | 8.37 | 21.7 | 7.60 | 8d 0.05M |
| * pH_9 0 | 8.98 | 21.8 | 8.01 | 2d 0.5M, 3d 0.1M, 3d 0.05M |

pH buffer 10.0 10.00 25.5

1106 PM Solutions were set back on gyratory shaker.

1238 PM Expt A-II-2 continued

Solutions A-II-2 *pH will be remeasured for pH and the pH and temperature recorded. The pH/ISE meter will be standardized using 5 Fisher pH buffers (pH = 2.0, 4.0, 7.0, 9.0, +10.0).

Next, the solutions will be adjusted down ~1 pH unit using HNO_3 (see previous page) as before.

| solution | pH | °C | adj. pH | added |
|----------------|------|------|---------|---|
| A-II-2 *pH 2.5 | 2.46 | 22.4 | 2.05 | 14 d 1.0M HNO_3 |
| *pH 3.0 | 3.11 | 22.3 | 2.53 | 6 d 1.0M |
| *pH 3.5 | 3.92 | 22.4 | 3.05 | 2 d 1.0M, 1 d 0.5M |
| *pH 4.0 | 5.10 | 22.4 | 3.54 | 1 d 0.5M, 6 d 0.1M |
| *pH 4.5 | 6.00 | 22.2 | 4.16 | 1 d 0.1M, 6 d 0.05M |
| *pH 5.0 | 6.28 | 22.6 | 4.51 | 1 d 0.1M, 2 d 0.05M |
| *pH 5.5 | 6.50 | 22.2 | 4.63 | 1 d 0.1M, 4 d 0.05M |
| *pH 6.0 | 6.66 | 22.5 | 5.29 | 1 d 0.1M, 1 d 0.05M |
| *pH 6.5 | 6.79 | 22.5 | 4.81 | 5 d 0.05M HNO_3 |
| *pH 7.0 | 6.93 | 22.5 | 6.20 | 2 d 0.05M HNO_3 |
| *pH 7.5 | 7.15 | 22.5 | 6.76 | 1 d 0.05M |
| *pH 8.0 | 7.70 | 22.6 | 7.32 | 2 d 0.05M |
| *pH 8.5 | 8.26 | 22.5 | 7.83 | 3 d 0.05M |
| *pH 9.0 | 8.86 | 22.5 | 8.11 | 1 d 0.5M, 2 d 0.1M, 2 d 0.05M HNO_3 |
| pH buffer 10.0 | 9.99 | 25.5 | | |

1425 PM The A-II-2 solutions were all put back on the gyratory shaker

0900 TD

Analyzed solutions $4(u-I + 1(a-l)) \times 1.02$ on the Polarograph using DPP. The machine was calibrated between 5 ppm and 15 ppm using standards made today. A 10 ppm check standard was made and the 10 ppm std made 3/30/92 was also used as a check.

15 ppm = 1.5 ml 1000 ppm Ustd, 5 ml 1.0M TT diluted to 100 ml
 10 ppm = 1.0 ml 1000 ppm Ustd, 5 ml 1.0M TT diluted to 100 ml
 5 ppm = 0.5 ml 1000 ppm Ustd, 5 ml 1.0M TT diluted to 100 ml

Ustd lot # D145

1.0M TT (Tartaric Acid/Triethanolamine) 2/19/92

Results were entered into the tables on pages 8 and 9. Plots, Parameters, and calibration curves can be found in Polarographic Analyses of Uranium vol 10.

4/3/92 PM Expt A-I-3 continued

1204

3 10-ml aliquots were taken from stock solution A-I-3 by Eppendorf maxipipet + put in 15-ml PP bottles labeled A-1-3-51 (or 2, 3). 500 μ l of 1.05M TT (see GC-05 2/19/92) was added to each 10 ml sample + shaken to mix. The samples will be left in the glove box until they are analyzed for U content by the polarograph.

1222 PM The pH/ISE meter was standardized using 5 fresh Fisher pH buffers (pH = 2.0, 4.0, 7.0, 9.0, +10.0). Solutions A-I-3 *pH were measured for pH and the pH/temp °C for each was recorded. ^{Solutions} ~~Samples~~ A-I-3 *pH 2.5... *pH 7.5 were adjusted down ^{4/3/92} ~1 pH unit each, using the HNO_3 already in the box + the glass droppers there as well.

Solutions A-I-3* pH 8.0... *pH 9.0 were adjusted upward using the NaOH (0.005M) prepared 2/20/92 (see GC-05-254), also already in the box.

| solution | pH | temp | adj. pH | added |
|----------------|------|------|---------|--|
| A-1-3* pH 2.5 | 3.14 | 22.2 | 2.51 | 10 d 0.5M HNO ₃ |
| *pH 3.0 | 3.90 | 22.2 | 2.96 | 5 d 0.5M HNO ₃ |
| *pH 3.5 | 4.73 | 22.4 | 3.71 | 1 d 0.5M, 1 d 0.05M HNO ₃ |
| *pH 4.0 | 6.33 | 22.4 | 4.01 | 9 d 0.05M HNO ₃ |
| *pH 4.5 | 7.16 | 22.5 | 4.42 | 4 d 0.05M HNO ₃ |
| *pH 5.0 | 7.37 | 22.6 | 4.89 | 2 d 0.05M HNO ₃ |
| *pH 5.5 | 7.35 | 22.6 | 5.48 | 1 d 0.05, 12 d 0.005M HNO ₃ |
| *pH 6.0 | 7.37 | 22.8 | 6.06 | 15 d 0.005M HNO ₃ |
| *pH 6.5 | 7.18 | 22.9 | 6.54 | 5 d 0.005M HNO ₃ |
| *pH 7.0 | 7.67 | 23.2 | 6.98 | 3 d 0.005M HNO ₃ |
| *pH 7.5 | 7.59 | 23.1 | 7.34 | 1 d 0.005M HNO ₃ |
| *pH 8.0 | 7.48 | 23.0 | 7.87 | 5 d 0.005M NaOH |
| *pH 8.5 | 7.46 | 23.2 | 8.45 | 8 d 0.005M NaOH |
| *pH 9.0 | 7.64 | 23.3 | 8.78 | 18 d 0.005M NaOH |
| pH buffer 10.0 | 9.97 | 21.3 | | |

1438 PM All A-1-3 *pH solutions were put back on the gyratory shaker set at ~125 rpm.

4/4/92 PM Expt A-I-2 continued

0848 The pH/ISE meter was calibrated using the same buffers as yesterday. The A-1-2 *pH solutions were measured for pH, the pH/temp °C recorded, and the pH adjusted downward as before.

| solution | pH | temp | adj. pH | added |
|---------------|------|------|---------|---------------------------|
| A-1-2* pH 2.5 | 2.12 | 21.5 | 1.99 | 6 d 0.5M HNO ₃ |
| 3.0 | 3.05 | 21.6 | 2.51 | 6 d 0.5M |
| 3.5 | 4.05 | 22.3 | 3.03 | 3 d 0.5M |
| 4.0 | 6.24 | 22.2 | 3.64 | 1 d 0.5M |

| solution | pH | temp | adj. pH | added |
|-----------------------|------|------|---------|--|
| XN 0946 A-1-2* pH 4.5 | 7.01 | 22.1 | 4.00 | 6 d 0.05M HNO ₃ |
| 5.0 | 7.31 | 22.2 | 4.52 | 3 d 0.05M |
| 5.5 | 7.56 | 22.1 | 4.75 | 3 d 0.05M |
| 6.0 | 7.52 | 22.2 | 5.37 | 2 d 0.05M |
| 6.5 | 7.66 | 22.2 | 5.96 | 1 d 0.05M, 6 d 0.005M HNO ₃ |
| 7.0 | 7.45 | 22.8 | 6.44 | 11 d 0.005M |
| 7.5 | 7.67 | 22.4 | 7.06 | 5 d 0.005M |
| 8.0 | 7.76 | 22.4 | 7.32 | 2 d 0.005M |
| 8.5 | 7.77 | 22.4 | 7.57 | 1 d 0.005M |
| 9.0 | 7.91 | 22.6 | 7.64 | 1 d 0.005M |

pH buffer 10.0 9.97 20.8

1045 PM All A-1-2 *pH solutions were set back on the gyratory shaker.

4/6/92 0830 TD

Prepared 100 ml of 4.0M NaNO₃ solution by dissolving 33.9960 gm of NaNO₃ in a 100 ml volumetric flask using Ultrapure water and diluting to the mark.

WT USED 33.9957 gm

LOT NUMBER 7808 KDJE

0900 Prepared 11 std in .1 M NaNO₃ with .05M Tartaric Acid / Triethanol amine by mixing Ultrapure H₂O, 2.5 ml 4M NaNO₃ (prepared above), 5 ml 1.0 M TT (2/19/92), and the amount of standard given below in a 100 ml volumetric flask and diluting to the mark with H₂O.

| | | | | | | |
|---------|------|-----------------------|---------|-------------|-------|--------------|
| 50 ppm | 5 ml | 1000 ppm (lot # D145) | 300 ppb | 1 ml 30 ppm | 5 ppb | 1 ml 500 ppb |
| 40 ppm | 4 ml | 1000 ppm | 200 ppb | 1 ml 20 ppm | | |
| 36 ppm | 3 ml | 1000 ppm | 100 ppb | 1 ml 10 ppm | | |
| 20 ppm | 2 ml | 1000 ppm | 50 ppb | 1 ml 50 ppm | | |
| 10 ppm | 1 ml | 1000 ppm | 30 ppb | 1 ml 30 ppm | | |
| 500 ppb | 1 ml | 50 ppm | 20 ppb | 1 ml 20 ppm | | |
| 400 ppb | 1 ml | 40 ppm | 10 ppb | 1 ml 10 ppm | | |

4/6/92 1100 TD
 Analyzed standards prepared earlier today to TD 4/6/92
 on the Polarograph using DPB to examine
 the effect of Na^+ concentration on detectability
 and peak height

4/8/92 TD

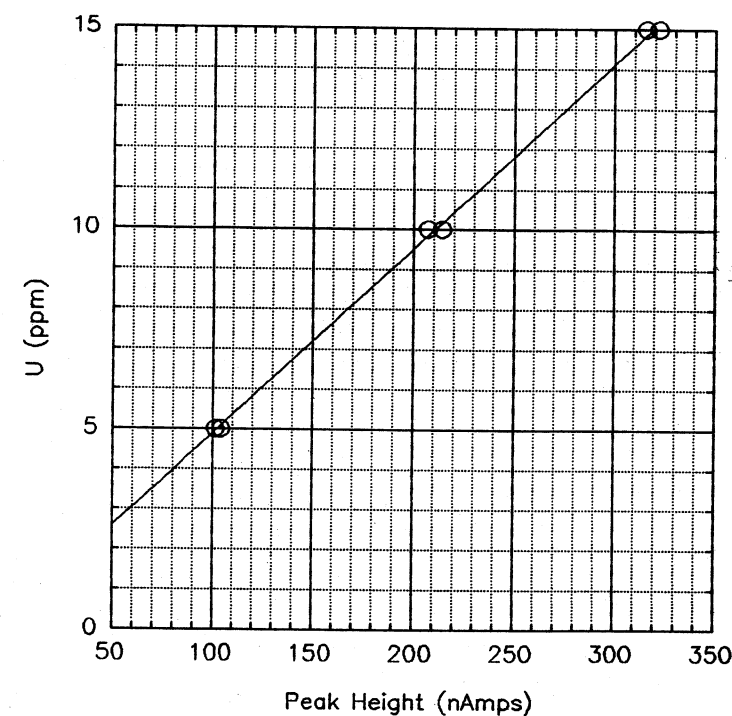
Remeasured the pH and U concentration of
 H/U-I*1 solutions

| SAMPLE | pH | U CONC (ppm) | |
|--------------------|-------------|--------------|-------------|
| | | *1 (4/9/92) | *2 (4/8/92) |
| H/U-I-4*100a | 3.86/20.6°C | 94.1 | 92.5 |
| H/U-I-4*100b | 3.98/20.3°C | 92.1 | 95 |
| H/U-I*1a | 1.83/21.5°C | 93.0 | 99.5 |
| *1b | 2.00/21.3°C | 93.6 | 102 |
| *1c | 2.18/21.4°C | 95.6 | 99.2 |
| *1d | 2.38/21.3°C | 96.1 | 99.9 |
| *1e | 2.57/21.4°C | 96.7 | 104 |
| *1f | 2.76/21.3°C | 96.7 | 102 |
| *1g | 2.98/21.5°C | 99.2 | 101 |
| *1h | 3.15/21.5°C | 97.2 | 102 |
| *1i | 3.31/21.4°C | 95.4 | 101 |
| *1j | 3.49/21.5°C | 96.3 | 99.3 |
| *1k | 3.84/21.1°C | 97.6 | 99.8 |
| H/U-I*100 ppm *1M1 | - | - | 95.1 |
| *1U2 | - | - | 96.8 |
| *1U3 | - | - | 97.5 |
| H/U-I*1k* | 3.67/21.1°C | 97.8 | 97.7 |

Plots for these runs as well as calibration curves
 can be found in Polarographic analysis of
 Uranium Vol 10.

CALIBRATION VALUES AND CURVE FOR 4/8/92 CALIBRATIONS
 Standards from lot # D145 prepared 9/2/92 (GC-07-15)

| 1 | U, ppm 2 | Pk Ht (nAmps) 3 | Regress-Ord1 4 |
|----|-------------|--------------------|-------------------|
| 1 | ANALYZED | 5 | 104 |
| 2 | ON | 5 | 101 |
| 3 | CELL #1 | 10 | 214 |
| 4 | | 10 | 207 |
| 5 | | 15 | 322 |
| 6 | | 15 | 316 |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |
| | | | 0.2792 |
| | | | 0.046143 |
| | | | 0 |
| | | | 0 |
| | | | 0 |
| | | | 0 |
| | | | 0 |
| | | | 0 |
| | | | 0.9995 |



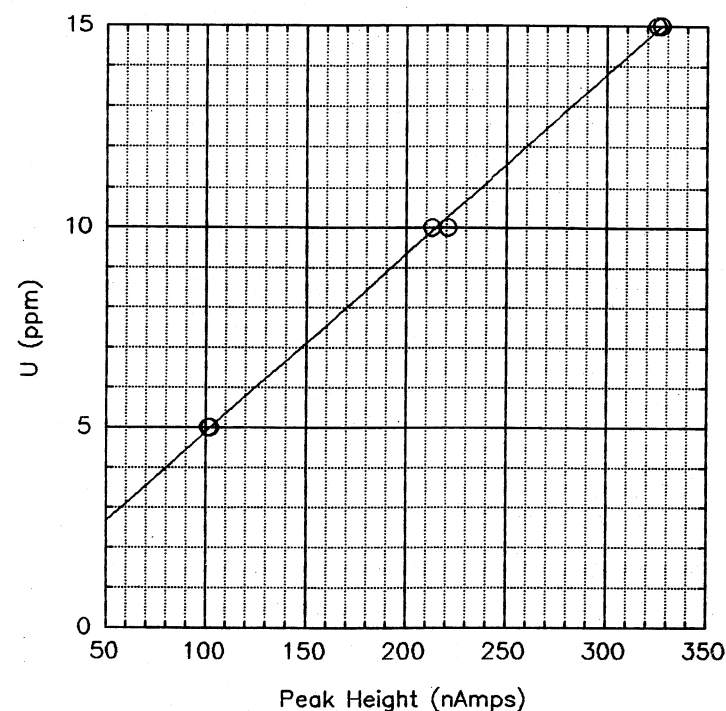
4/9/92 0830 TD

Reanalyzed the solutions 4/4-I/1(a-l)*1 using DPP on the Polarograph. The results were entered in the table on page 18. The machine was calibrated using the 5, 10, and 15 ppm U stds prepared 4/2/92 (see page 15). For an explanation of the DPP procedure see the Model 384B Polarographic Analyzer Instruction Manual pgs V31 and V40-43.

CALIBRATION VALUES AND CURVE FOR 4/9/92

| 1 | U, ppm 2 | Pk Ht (nAmps) 3 | Regress-Ord1 4 |
|----|-------------|--------------------|-------------------|
| 1 | ANALYZED | 5 | 102 |
| 2 | ON | 5 | 101 |
| 3 | CELL #1 | 10 | 221 |
| 4 | | 10 | 213 |
| 5 | | 15 | 327 |
| 6 | | 15 | 325 |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |

0.4398
0.044501
0
0
0
0
0
0
0
0
0.99952



4/16/92 PPL Kinetics Experiment K1 4/24/92
1155 (Initial pH ~ 4.5, 5.5; ~~0.1 M NaNO₃~~, equil with atm. CO₂)

K1-1: Solution K1-1 (2 bottles)
- Initial $\Sigma U = 500$ ppb
Initial $\Sigma Na = 0$
Initial pH = 4.5
Initial solution mass = 950 g
Wt zeolite used = 0.95 g
Equilibrium with atmospheric CO₂ (g)

K-1-1-A: 2000 mL of 500 ppb U solution was made by diluting 1 mL of commercial 1000 ppm U standard (UO₂(NO₃)₂ · 6H₂O in water, Lot # standard = L0-10, expiration date = Dec '92. The 1 mL was diluted to 2000 mL volume in a volumetric flask with ultrapure water.

1211 PM K1-1-B: Two 950 ± 1 g aliquots of the 500 ppb solution were transferred into 1000 mL PP bottles labeled K1-1*A and K1-1*B

| | | |
|-------------|----------------------------|-------|
| wt solution | K1-1*A (bottle wt = 90.76) | 950.2 |
| | K1-1*B | 90.99 |
| | | 951.2 |

1225 PM The Initial pH of solutions K1-1*A and K1-1*B (~5.45 calculated using EQ3) were measured using an Orion combination glass electrode / ATC probe. The pH/ISE meter was calibrated using pH buffers 4.0 & 7.0 (Fisher, lot #s 5B98-500 & 5B108-500).

| | |
|-----------|-------------|
| pH K1-1*A | 5.27 @ 20.5 |
| K1-1*B | 5.29 @ 20.6 |

1250 PM 17 drops of 0.05 M HNO₃ (see GC-05-301) were added to each solution using glass dropper previously calibrated (see GC-05-214). As the dropper dispenses 0.0427 mL/drop,

~16.7 drops of 0.05 M HNO_3 is needed to add $\sim 3.57 \times 10^{-5}$ moles HNO_3 resulting in a pH ≈ 4.5 according to EQ3 calculations.

1258 PM The bottles were covered with Kimwipes + agitated in a gyratory shaker set at ~ 100 rpm for at least an hour.

K1-1-D: Three 10-mL aliquots of solutions K1-1*10 were taken by Eppendorf maxipipet (pre-rinsed with solution K1-1*10) and put in 15-mL PP bottles labeled K1-1*10a (or b, c). 500 μL of 1.05 M tartaric acid/triethanolamine solution (see 66-05-252) was added to each sample.

1415 PM Two 0.95 g of Na-clinoptilolite were tared into weighing paper.

wt. a 0.9508 g
b 0.9497 g

1425 PM The pH meter was recalibrated as before and solutions K1-1*A + B were measured for pH.

PM 4/4/94

K1-1*A 4.53 @ 21.6 °C

*B 4.55 @ 21.4 °C

1430 PM The 0.95 g of Na clinoptilolite were added to their respective bottles. Room temp = 70.4 °F. The solutions were replaced on the gyratory shaker set at ~ 100 rpm.

K1-1-E: At Δ time = 2, 4, 22, 27, 46, 70, 94, 166, 214, 262, 286, 334 hours, two 10-mL samples will be taken with a maxipipet from solutions K1-1*A and *B + put into

on 4/1/94

15-mL PP bottles labeled K1-1*A*i a (or b) and K1-1*B*i a (or b) where i is the sampling time number (1, 2, 3...). 500 μL of 1.05 M tartaric acid/triethanolamine will be added to each sample.

Also, the pH of the solutions K1-1*A + K1-1*B will be read, + the pH + solution temperature recorded.

solution K1-1*A

| sampling number | time | hours | pH | sol'n temp °C | U concentration ppb 1 2 |
|--------------------|-----------|-------|----------------------|------------------|----------------------------|
| 1 | 4/10 1645 | 2.1 | 4.75 | 22.3 | |
| 2 | 1848 | 4.2 | 4.67 ^{7 PM} | 22.4 | |
| 3 | 4/11 1243 | 22.1 | 4.94 | 21.6 | |
| 4 | 1748 | 27.1 | 5.19 | 22.5 | |
| 5 | 4/12 1240 | 46.0 | 5.39 | 21.4 | |
| 6 | 4/13 1252 | 70.1 | 5.89 | 22.5 | |
| 7 | 4/14 1241 | 94.0 | 6.06 | 22.0 | |
| 8 | 4/17 1240 | 166.0 | 6.21 | 21.3 | |
| 9 | 4/19 1235 | 213.9 | 6.39 | 21.3 | |
| 10 | 4/21 1243 | 262.1 | 6.42 | 27.2 | |
| 11 | 4/22 1311 | 286.5 | 6.41 | 25.4 | |
| 12 | 4/24 1245 | 334.1 | 6.42 | 24.0 | |
| 13 | 4/27 1645 | 410.1 | 6.44 | 24.8 | |

After the pH was measured + the samples taken, solutions K1-1*A + K1-1*B (next page) were recovered with Kimwipes + put back on the gyratory shaker.

solution K1-1*B

| sampling time number | sampling times | Δ hours | pH | soln temp °C | 0 concentration ppb 1 2 |
|----------------------|----------------|---------|------|--------------|----------------------------|
| 1 | 4/10 1647 | 2.1 | 4.74 | 22.3 | |
| 2 | 1849 | 4.2 | 4.76 | 22.3 | |
| 3 | 4/11 1244 | 22.1 | 4.99 | 21.6 | |
| 4 | 1750 | 27.2 | 5.16 | 22.4 | |
| 5 | 4/12 1245 | 46.1 | 5.23 | 21.4 | |
| 6 | 4/13 1301 | 70.3 | 5.68 | 22.4 | |
| 7 | 4/14 1245 | 94.1 | 5.91 | 21.9 | |
| 8 | 4/17 1249 | 166.2 | 6.26 | 21.3 | |
| 9 | 4/19 1240 | 214.0 | 6.37 | 21.4 | |
| 10 | 4/21 1247 | 262.1 | 6.45 | 27.3 | |
| 11 | 4/22 1314 | 286.6 | 6.46 | 25.4 | |
| 12 | 4/24 1250 | 334.2 | 6.48 | 24.0 | |
| 13 | 4/27 1649 | 410.2 | 6.49 | 24.8 | |
| 14 | 5/5 0850 | 6.52 | 22.2 | | |

A fresh portion of pH buffer (Fisher) was measured for the K1-1*A,B solutions were measured for pH to help monitor the condition of the calibration buffers.

| | | |
|-----------|--------------------------------|--------------|
| 4/10 1701 | pH buffer 4.0 (lot # SB98-500) | 4.01 at 22.1 |
| 1858 | " | 4.02 at 21.3 |
| 4/11 1257 | " | 4.02 at 21.0 |
| 1758 | " | 4.04 at 21.5 |
| 4/12 1249 | " | 4.04 at 21.1 |
| 4/13 1305 | " | 4.02 at 21.4 |
| 4/14 1249 | " | 4.02 at 21.2 |
| 4/17 1253 | " | 4.02 at 21.0 |
| 4/19 1245 | " | 4.01 at 20.6 |
| 4/21 1250 | " | 4.00 at 24.0 |

A-II-3 PM 4/11/92

4/11/92 PM

Expt A-II-3 continued

1008 PM 10 mL aliquots of solutions A-II-3* μ Hi were taken by Eppendorf micropipet and put in 15-mL PP bottles. The bottles were labeled A-II-3* μ Hi*VR2, where $i = 2.5 \dots 9.0$. 500 μ L of 1.05 M tartaric acid/triethanolamine was added to each sample.

Also, 3 10-mL aliquots were taken from solution A-II-3*10 and placed in 3 15-mL PP bottles labeled A-II-3*10*7 (or 8, 9). 500 μ L of the 1.05 M TI was added to each sample.

1050 PM The pH and temperature of solutions A-II-3* μ Hi were measured after the pH/5E meter was calibrated using 5 Fisher buffers (pH = 2.00, 4.00, 7.00, 9.00, + 10.00 lot # SB96-500, SB98-500, SB108-500, SB114-500, SB116-500).

| solution | pH | temp. °C |
|----------------------|-------|----------|
| A-II-3* μ Hi 2.5 | 2.55 | 21.4 |
| 3.0 | 3.21 | 21.4 |
| 3.5 | 3.92 | 21.4 |
| 4.0 | 4.77 | 21.3 |
| 4.5 | 6.01 | 21.5 |
| 5.0 | 6.27 | 21.6 |
| 5.5 | 6.42 | 21.5 |
| 6.0 | 6.67 | 21.6 |
| 6.5 | 6.89 | 21.7 |
| 7.0 | 7.10 | 21.8 |
| 7.5 | 7.35 | 21.7 |
| 8.0 | 7.80 | 21.6 |
| 8.5 | 8.30 | 21.7 |
| 9.0 | 8.91 | 21.9 |
| pH buffer 10.0 | 10.00 | 25.3 |

1135 PM All A-II-3* μ Hi solutions were set back in the shaker.

A-II-3 PM 4/11/92

1530 PM

Expt. A-II-2 *pH cont

A-II-3 PM 4/11/92

10 ml aliquots of solutions A-II-2 *pH were sampled taken by Eppendorf max pipet + put in 15-ml bottles (PP) labeled A-II-2 *pH *UR2. 3 10-ml aliquots were taken from solution A-II-2 *U1 and put in 15-ml PP bottles labeled A-II-2 *U1 *7 (or 8, 9).

1612 PM

Expt A-II-2 continued

10-ml aliquots of solutions A-II-2 *pH were taken by max pipet + put in 15-ml PP bottles labeled A-II-2 *pH *UR2. Three ten-ml aliquots were also taken from solution A-II-2 *U1 and put in 3 15-ml PP bottles labeled A-II-2 *U1 *7 (or 8, 9).

500 μ l of 1.05 M tartaric acid/triethanolamine solution (6C-05-252) was added to each sample taken.

1652 PM The pH meter was recalibrated with 5 buffers (see previous page for buffer ID) + the A-II-2 *pH solutions were measured for pH + temperature.

| solution | pH | temp °C | solution | pH | temp °C |
|----------------|------|---------|----------------|-------|---------|
| A-II-2 *pH 2.5 | 2.13 | 22.4 | A-II-2 *pH 6.0 | 6.34 | 23.0 |
| 3.0 | 2.66 | 22.8 | 6.5 | 6.18 | 22.9 |
| 3.5 | 3.31 | 22.8 | 7.0 | 6.79 | 22.9 |
| 4.0 | 4.02 | 22.8 | 7.5 | 7.12 | 23.0 |
| 4.5 | 4.82 | 22.7 | 8.0 | 7.71 | 22.9 |
| 5.0 | 5.57 | 22.9 | 8.5 | 8.30 | 23.0 |
| 5.5 | 5.90 | 23.6 | 9.0 | 8.85 | 23.0 |
| | | | pH buffer 10.0 | 10.00 | 25.4 |

4/13/92 0830 TD

Began calibrating the Polarograph using the standards prepared on 3/30/92 in Teflon cups. Could not get a reproducible peak height on either high or low standards.

1000 ID

Switched to glass cells. Peaks were more reproducible but calibration values did not fit along a first or second degree calibration regression. New standards will be made.

230 TD

Prepared new U standards from 10 ppb - 50 ppm in .05 M Tartaric Acid / Triethanolamine according to the chart below.

| | |
|--|----------|
| 50 ppm = 5 ml 1000 ppm U std (Ricca lot # D145), 5 ml .05 M TT (2/19/92) | → 100 ml |
| 40 ppm = 4 ml 1000 ppm U std | → 100 ml |
| 30 ppm = 3 ml 1000 ppm U std | " |
| 20 ppm = 2 ml 1000 ppm U std | " |
| 10 ppm = 1 ml 1000 ppm U std | " |
| 500 ppb = 1 ml 50 ppm U std prepared above | " |
| 400 ppb = 1 ml 40 ppm U std | " |
| 300 ppb = 1 ml 30 ppm U std | " |
| 200 ppb = 1 ml 20 ppm U std | " |
| 100 ppb = 1 ml 10 ppm U std | " |
| 50 ppb = .1 ml 50 ppm U std | " |
| 30 ppb = .1 ml 30 ppm U std | " |
| 10 ppb = .1 ml 10 ppm U std | " |

All were placed in clean acid washed and dried PP bottles and labeled.

1305 PM Expt A-1-2 continued

The pH meter in the glove box was standardized with 5 Fisher buffers (pH = 2.0, 4.0, 7.0, 9.0, + 10.0) Fastused 4/3/92, lot #s SB96-500 SB98-500, SB108-500 SB114-500 + SB116-500). The solutions A-1-2 *pHi were measured for pH.

| solution | pH | temp °C |
|-----------------------|------|---------|
| 1320 PM A-1-2 *pH 2.5 | 2.11 | 23.2 |
| 3.0 | 2.78 | 23.3 |
| 3.5 | 3.50 | 23.6 |
| 4.0 | 4.42 | 23.1 |
| 4.5 | 5.03 | 23.2 |
| 5.0 | 6.12 | 23.2 |
| 5.5 | 6.61 | 23.2 |
| 6.0 | 6.99 | 23.2 |
| 6.5 | 7.17 | 23.2 |
| 7.0 | 7.31 | 23.5 |
| 7.5 | 7.58 | 23.5 |
| 8.0 | 7.72 | 23.2 |
| 8.5 | 7.84 | 23.3 |
| 9.0 | 7.86 | 23.6 |

1410 PM pH buffer 10.0 9.97 22.4

One 10-ml aliquot was taken from each solution A-1-2 *pHi + three ten ml aliquots were taken from A-1-2 K10 and put in 15-ml PP bottles. The bottles were labeled A-1-2 *pHi *UR2 or A-1-2 *10 *4 (or 5, 6). 500 μ l of 1.05 M tartaric acid/triethanolamine (GC05-252) was added to each sample. All samples were left in the glove box until later U analysis by polarography.

4/14/92 PM pH Buffer Readings with + without CO₂

1350 PM The pH readings made on the pH meter calibrated in the glove box may be shifted due to the N₂ atmosphere. To check, the meter will be calibrated with 5 fresh Fisher buffers put in the box ~ 3 1/2 hours ago. The buffers are pH 2.0, 4.0, 7.0, 9.0, + 10.0, from the same lots as before (see previous page). The buffers will then be read inside the box, and the pH and temperature noted.

Next, the pH meter will be taken out of the box, + 5 fresh Fisher buffers (same pHs + lot #s used above) will be measured, + the pH + temperature noted.

| buffer | pH inside box | temp °C | pH outside box | temp °C |
|----------|---------------|---------|----------------|---------|
| pH = 2.0 | 1.96 | 22.6 | 1.95 | 20.3 |
| 4.0 | 3.97 | 22.5 | 3.94 | 20.0 |
| 7.0 | 6.97 | 22.7 | 6.99 | 20.1 |
| 9.0 | 8.95 | 22.9 | 8.97 | 20.0 |
| 10.0 | 9.99 | 22.7 | 10.01 | 20.3 |

0830 TD

Calibrated the Polarograph using the U standards prepared 4/13/92 (p27). Cell 1 was used for low concentrations and cell 2 for high (low = 10E XE 100, high \geq 100). Measured U concentrations of solutions A-II-3 *pHi *U using DPS and computed the concentrations using the average peak height and the 2nd order regression equation.

14-APR-92
 30-MAR-92 CELL NUMBER 1
 DPS
 INITIAL E 0.100 V
 FINAL E -0.700 V
 PURGE 480 SECONDS
 STEP TIME 0.5 SECONDS
 SCAN INCREMENT 6 MV
 CONDITION 0 SECONDS
 CONDITION 0.000 V
 EQUILIBRATE 10 SECONDS
 DEPOSITION 60 SECONDS
 PULSE HEIGHT 0.020 V
 REPLICATIONS 1

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

DPS PARAMETERS

| U ppb | Pk Height | Regress-Ord2 | |
|-------|-----------|--------------|----------|
| 1 | 2 | 3 | 4 |
| 10 | 1.5 | 1.9824 | <----- |
| 10 | 1.5 | 5.4176 | ANALYZED |
| 30 | 5.1 | 0.0065683 | ON |
| 30 | 5 | 0 | CELL #1 |
| 50 | 9.5 | 0 | WITH 60S |
| 50 | 8.4 | 0 | DEPOSIT |
| 100 | 18.3 | 0 | |
| 100 | 16.9 | 0 | |
| | | 0 | |
| | | 0 | |
| | | 0.99702 | |

CELL 1

CALIBRATION

VALUES AND
REGRESSION

| U ppb | Pk Height | Regress-Ord2 | |
|-------|-----------|--------------|----------|
| 5 | 6 | 7 | 8 |
| 100 | 4.5 | 9.3256 | <----- |
| 100 | 4.1 | 21.078 | ANALYZED |
| 200 | 8.7 | 0.028309 | ON |
| 200 | 9.2 | 0 | CELL #2 |
| 300 | 13.3 | 0 | WITH 0S |
| 300 | 13.6 | 0 | DEPOSIT |
| 400 | 18.3 | 0 | |
| 400 | 18.1 | 0 | |
| 500 | 22.4 | 0 | |
| 500 | 22.7 | 0 | |
| | | 0.99957 | |

CELL 2

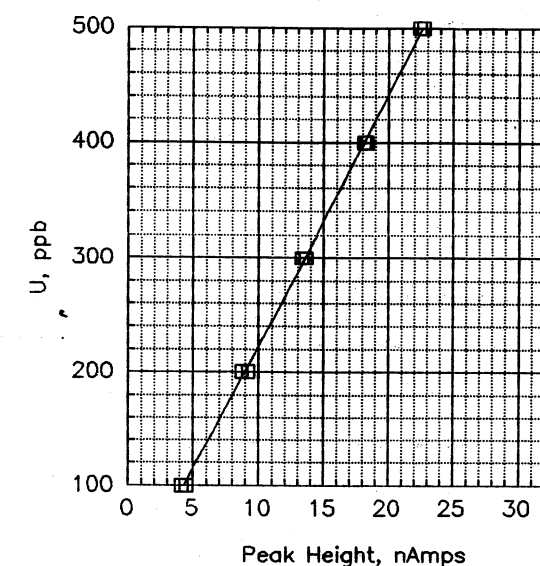
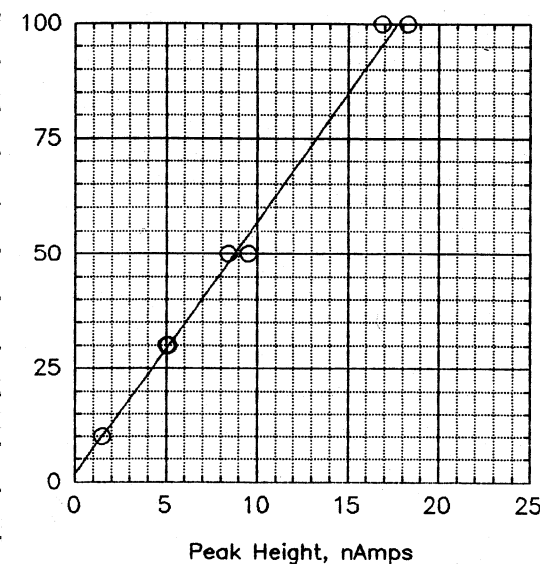
CALIBRATION

VALUES AND
REGRESSION

CALIBRATION CURVES (4/14/92)

CELL 1

CELL 2



U concentration Analysis

| A-II-3*PHI*U | Pk Ht1(nAmps) | Pk Ht2(nAmps) | U conc(ppb) |
|--------------|---------------|---------------|-------------|
| 1 | 2 | 3 | 4 |
| 1 | 2.5 | 16.5 | 18 |
| 2 | 3 | 14.3 | 14.8 |
| 3 | 3.5 | 4.9 | 5 |
| 4 | 4 | 4.5 | 4.5 |
| 5 | 4.5 | 2.2 | 2.1 |
| 6 | 5 | 2.6 | 2.6 |
| 7 | 5.5 | 2.3 | 2.6 |
| 8 | 6 | 3.6 | 3.8 |
| 9 | 6.5 | 4.9 | 4.3 |
| 10 | 7 | 7 | 6.3 |
| 11 | 7.5 | 6.2 | 6.2 |
| 12 | 8 | 14.6 | 14.4 |
| 13 | 8.5 | 20.5 | 21.6 |
| 14 | 9 | 21.9 | 22.6 |
| 15 | 300 PPB | 11.9 | 12.4 |
| 16 | 30 PPB U | 4.9 | 4.9 |

(270)
(28.7)

Plots for these runs can be found in Polarographic
 Analyses of Uranium, vol 11.

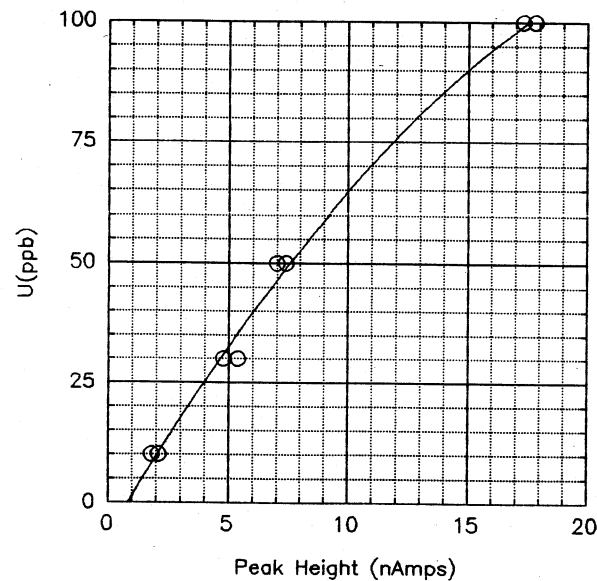
4/16/92 0800 TD

Analyzed the U concentration of A-II-3*PHI*UR2 on the Polarograph using DPS as before (p 30). The standards prepared on 4/13/92 were used for ~~standards~~ calibration. Plots can be found in Polarographic Analyses of Uranium, Vol 11.

UDPP0416.SPG: Thu, 16-Apr-92

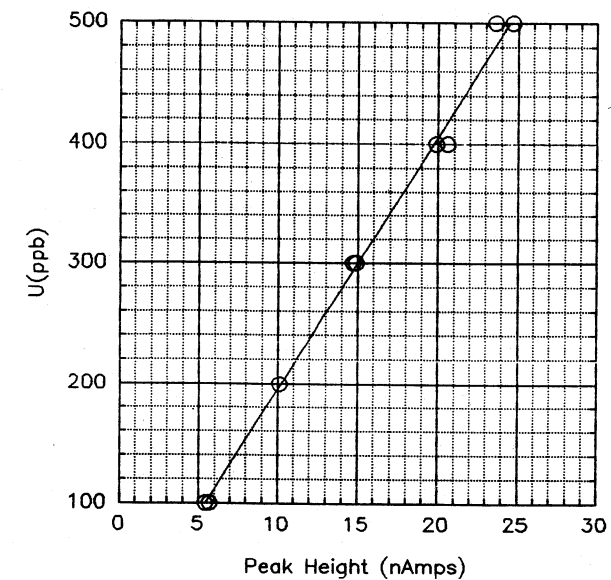
| U(ppb) | Pk Ht(nAmps) | Regress-2ord | |
|--------|--------------|--------------|---|
| 1 | 2 | 3 | 4 |
| 100 | 17.8 | -7.1489 | <----- ANALYZED ON CELL #2 WITH 60 S DEP |
| 100 | 17.3 | 8.6432 | |
| 50 | 7.4 | -0.1443 | |
| 50 | 7 | 0 | |
| 30 | 4.8 | 0 | |
| 30 | 5.4 | 0 | |
| 10 | 1.8 | 0 | |
| 10 | 2.1 | 0 | |
| | | 0 | |
| | | 0 | |
| | | 0.99719 | |

CALIBRATION VALUES AND CURVE FOR CELL #2



| U(ppb) | Pk Ht(nAmps) | Regress-2ord | |
|--------|--------------|--------------|---|
| 5 | 6 | 7 | 8 |
| 500 | 23.6 | -14.005 | <----- ANALYZED ON CELL #1 WITH 05 DEP |
| 500 | 24.7 | 20.884 | |
| 400 | 19.9 | 0.0050883 | |
| 400 | 20.6 | 0 | |
| 300 | 14.9 | 0 | |
| 300 | 14.7 | 0 | |
| 200 | 10.1 | 0 | |
| 200 | 10.1 | 0 | |
| 100 | 5.7 | 0 | |
| 100 | 5.4 | 0 | |
| | | 0.99803 | |

CALIBRATION VALUES AND CURVE FOR CELL 1



RESULTS OF U ANALYSIS

| A-II-3*PHI*R2 | Pk Ht1(nAmps) | Pk Ht2(nAmps) | U conc(ppb) |
|---------------|---------------|---------------|-------------|
| 1 | 2 | 3 | 4 |
| 2.5 | 17.1 | 17.7 | 351 |
| 3 | 19.7 | 17.8 | 379 |
| 3.5 | 12 | 11.6 | 233 |
| 4 | 12 | 12.2 | 76.3 |
| 4.5 | 3.7 | 3.8 | 23.2 |
| 5 | 4.6 | 3.7 | 26.2 |
| 5.5 | 3.7 | 3.4 | 21.7 |
| 6 | 5.3 | 5.3 | 34.6 |
| 6.5 | 6.7 | 6.7 | 44.3 |
| 7 | 13.1 | 13.5 | 82.3 |
| 7.5 | 7.1 | 6.2 | 125 |
| 8 | 16 | 17 | 332 |
| 8.5 | 24.1 | 24.9 | 501 |
| 9 | 25.9 | 26.4 | 536 |
| IU7 | 20 | 20.1 | 407 |
| IU8 | 22 | 21.3 | 441 |
| IU9 | 22.6 | 21.6 | 450 |
| 300 PPB | 15.8 | 16.1 | (320) |
| 30 PPB | 6.6 | 6.3 | (42.6) |

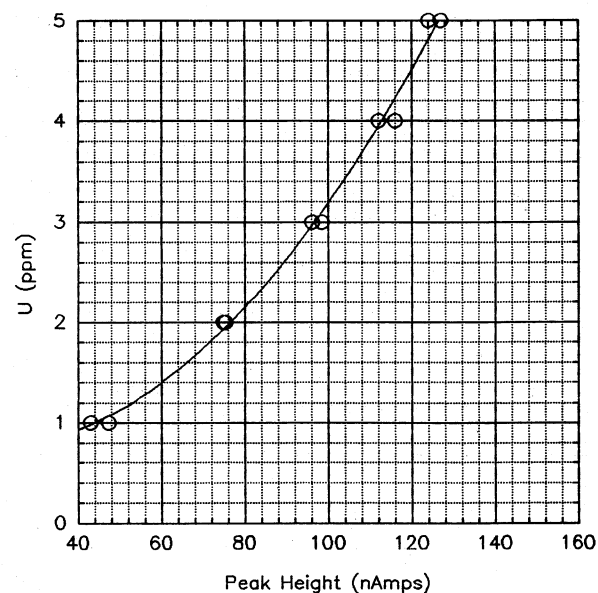
4/17/92 0800 TD

Analyzed the U concentration of solution A-II-2*PHI*URZ using DPS on the Polarograph. The 1-5 ppm U standards were made by diluting 1ml of the appropriate 10-50 ppm standard (made 4/13/92) + 500 μ L 1.0M TT to 10ml with H_2O in a volumetric flask. Plots can be found in Polarographic Analysis of Uranium Vol 11.

U1-50417.SPG: Fri, 17-Apr-92

| U(ppm) | Pk Ht(nAmps) | Regress-20rd |
|--------|--------------|--------------|
| 1 | 47.2 | 0.82859 |
| 1 | 42.9 | -0.011633 |
| 2 | 74.8 | 0.00035249 |
| 2 | 75.3 | 0 |
| 3 | 96.1 | 0 |
| 3 | 98.4 | 0 |
| 4 | 116 | 0 |
| 4 | 112 | 0 |
| 5 | 124 | 0 |
| 5 | 127 | 0 |
| | | 0.99716 |

CALIBRATION VALUES AND
CURVE



RESULTS OF URANIUM CONC ANALYSIS

AII2R2.SPG: Fri, 17-Apr-92

| A-II-2*PHI*R2 | Pk Ht1(nAmps) | Pk Ht2(nAmps) | U (ppm) |
|---------------|---------------|---------------|---------|
| 1 | 2 | 3 | 4 |
| 2.5 | 95.2 | 100 | 3.03 |
| 3 | 119 | 125 | 4.62 |
| 3.5 | 123 | 128 | 4.86 |
| 4 | 128 | 126 | 5 |
| 4.5 | 113 | 114 | 4.02 |
| 5 | 71.2 | 71.6 | 1.78 |
| 5.5 | 58.3 | 58.5 | 1.34 |
| 6 | 52.2 | 52.2 | 1.18 |
| 6.5 | 48.1 | 49.3 | 1.09 |
| 7 | 49.2 | 48.5 | 1.1 |
| 7.5 | 42.8 | 42.3 | 0.967 |
| 8 | 100 | 102 | 3.22 |
| 8.5 | 125 | 124 | 4.81 |
| 9 | 129 | 132 | 5.27 |
| IU7 | 116 | 117 | 4.22 |
| IU8 | 117 | 118 | 4.29 |
| IU9 | 117 | 118 | 4.29 |
| 3 PPM | 98.7 | 96.3 | (3.02) |

4/20/92 TD 0800

Prepared new Uranium standards in .05M Tartaric Acid/Triethanolamine. The standards were prepared according to the table below. The RICCA U standard (1000 ppm), lot # D145, was used to prepare the 50-10 ppm standards and all other standards were prepared by serial dilution. The 1.0M Tartaric Acid/Triethanolamine was made 2/19/92.

| | |
|---------|--|
| 50 ppm | = 5ml 1000 ppm, 5ml 1.0M TT, dilute to 100ml with Ultrapure H_2O |
| 40 ppm | = 4ml 1000 ppm, " " " " " " " " |
| 30 ppm | = 3ml 1000 ppm, " " " " " " " " |
| 20 ppm | = 2ml 1000 ppm, " " " " " " " " |
| 10 ppm | = 1ml 1000 ppm, " " " " " " " " |
| 5 ppm | = 10ml 50 ppm, " " " " " " " " |
| 4 ppm | = 10ml 40 ppm, " " " " " " " " |
| 3 ppm | = 10ml 30 ppm, " " " " " " " " |
| 2 ppm | = 10ml 20 ppm, " " " " " " " " |
| 1 ppm | = 10ml 10 ppm, " " " " " " " " |
| 500 ppb | = 1ml 50 ppm, " " " " " " " " |
| 400 ppb | = 1ml 40 ppm, " " " " " " " " |
| 300 ppb | = 1ml 30 ppm, " " " " " " " " |
| 200 ppb | = 1ml 20 ppm, " " " " " " " " |
| 100 ppb | = 1ml 10 ppm, " " " " " " " " |
| 50 ppb | = 0.1ml 50 ppm, " " " " " " " " |
| 30 ppb | = 0.1ml 30 ppm, " " " " " " " " |
| 10 ppb | = 0.1ml 10 ppm, " " " " " " " " |

All dilutions were done in 100 ml volumetric flasks. The 5ml and 10ml measurements were done with glass volumetric pipets, other measurements were done with Eppendorf fixed volume pipets.

4/20/92 1230 TD

Reanalyzed some of the A-II-2*PHI samples using DPS on the Polarograph. Results were different enough to have all samples reanalyzed.

PARAMETERS

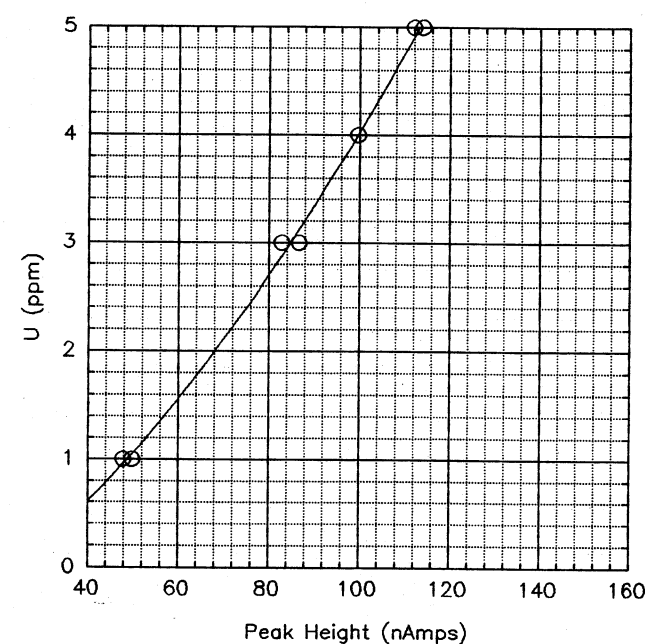
20-APR-92 CELL NUMBER 2
 DPS
 INITIAL E 0.100 V
 FINAL E -0.600 V
 PURGE 480 SECONDS
 STEP TIME 0.5 SECONDS
 SCAN INCREMENT 6 MV
 CONDITION 0 SECONDS
 CONDITION 0.000 V
 EQUILIBRATE 10 SECONDS
 DEPOSITION 0 SECONDS
 PULSE HEIGHT 0.020 V
 REPLICATIONS 1

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

U1-50420.SPG: Mon, 20-Apr-92

| U(ppm) | Pk Ht(nAmps) | Regress-20rd |
|--------|--------------|--------------|
| 1 | 2 | 3 |
| 1 | 47.8 | -0.74453 |
| 1 | 49.8 | 0.024495 |
| 3 | 86.7 | 0.00023236 |
| 3 | 82.8 | 0 |
| 4 | 99.6 | 0 |
| 4 | 99.7 | 0 |
| 5 | 114 | 0 |
| 5 | 112 | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0.99864 |

CALIBRATION CURVE



CALIBRATION VALUES

ANALYSIS

| A-II-2*PHI*R2 | Pk Ht1(nAmps) | Pk Ht2(nAmps) | U conc(ppm) |
|---------------|---------------|---------------|-------------|
| 1 | 2 | 3 | 4 |
| 2.5 | 84.1 | 85.4 | 2.98 |
| 4 | 105 | 110 | 4.55 |
| 5.5 | 51.9 | 52 | 1.15 |
| 7 | 45.6 | 49 | 0.93 |
| 8.5 | 112 | 113 | 4.92 |
| 9 | 98.8 | 93.6 | 3.74 |
| 2 PPM U | 67.6 | 71.8 | (2.08) |

All samples will be run tomorrow. The calibration standards used were those made earlier today. Plots can be found in Polarographic Analysis of Uranium vol II.

4/21/92 PM

Kinetics Experiment K-1 continued

KI-2: Solution KI-2 (2 bottles)
 - Initial $E_U = 500 \text{ ppb}$
 Initial $E_{Na} = 0$
 Initial pH = 5.5
 Initial Solution mass = 950g
 Wet zeolite used = 0.95g
 Equilibrium with atmospheric CO_2 (g)

0720PM KI-2-A: 2000 ml of 500 ppb U solution was prepared by diluting 1 ml of a 1000 ppm U stock solution (lot # 20-10, exp. date = Dec '92) to 2000 ml with ultrapure water in a volumetric flask.

KI-2-B: Two $950 \pm 1 \text{ g}$ aliquots of the 500 ppb solution was put in two 1000 mL polypropylene bottles labeled KI-2*A and KI-2*B.

KI-2*A bottle wt = 91.39g wt solu = 949.8g
 KI-2*B = 91.16g = 949.3g

The remaining solution was put into a 125-ml PP bottle labeled KI-2*U.

KI-2-C: The initial pH of solutions KI-2*A & KI-2*B were measured using a combination glass electrode/ATC probe. The meter was first calibrated using two Fisher pH buffers (pH = 5.0 & 7.0; lot # 5B102-500 & 5B108-500).

EQ3 calculations indicate the pH will be ~ 5.45

pH K1-2*A 5.11 @ 22.1
K1-2*B 5.12 @ 22.0

Fresh 5.0 buffer 5.01 @ 22.1
7.0 buffer 7.00 @ 22.1

0810 PM ~~As~~ the solutions are measuring lower than is allowed (within 0.3 of 5.5) the solutions will be remade & the experiment restarted.

0907 PM Expt K1: K1-2 re-started

K1-2-A: 2000 mL of 500 ppb U solution was prepared by diluting 1 mL of a 1000 ppm U stock solution (commercial standard prepared from $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in water, lot # L0-10) to volume in a 2000 mL volumetric flask with ultrapure water.

K1-2-B: Two 950 ± 1 g aliquots of the 500 ppb solution were poured into two 1000 mL polypropylene (PP) bottles labeled K1-2*A and K1-2*B. The remaining solution was poured into a 125-mL PP bottle labeled K1-2*U.

K1-2*A bottle wt = 91.41 g wt solu = 949.9 g
K1-2*B = 91.16 g = 949.2 g

K1-2-C: pH K1-2*A 5.21 @ 23.3
pH K1-2*B 5.30 @ 23.4

0950 PM The solution bottles K1-2*A & B were covered with Kim wipes and kept agitated on a New Brunswick gyratory shaker to equilibrate the solutions with atmospheric CO_2 (g). After at least an

hour has passed, the solutions will be remeasured.

0945 TD

A 4.1 M NaNO_3 solution was prepared by dissolving 34.8459 gms NaNO_3 in 100 mL of Ultrapure H_2O in a Volumetric flask.

wt NaNO_3 used 34.8456 gm lot # 7808 KDJE

1015 TD

10 mL aliquots of each Uranium standard from 30 ppb to 500 ppb (prepared 4/20/92 p37) were taken and placed in 15 mL PP bottles labeled with the concentrations below. Samples were taken using 10 mL glass Volumetric pipets. 250 μL of the 4.1 M NaNO_3 solution made earlier was added to each, producing a solution with a .1 M Na^+ concentration. This changed the concentration of Uranium to what is listed below. These standards will be used to calibrate the Polarograph when measuring the U concentrations of the Kinetics experiments.

500 ppb = 487.80 ppb
400 ppb = 390.24 ppb
300 ppb = 292.68 ppb
200 ppb = 195.12 ppb
100 ppb = 97.56 ppb
50 ppb = 48.78 ppb
30 ppb = 29.27 ppb

1054 PM

Expt K1-2 continued
Two 0.95 g of Na-clinoptilolite were quickly tared onto weighing paper.

a 0.9519 g
b 0.9512 g

1106 PM The pH of K1-2A & B were remeasured,
(K1-2-D:) & the Na-clingitolate added to each solution.

K1-2A pH = 5.18 @ 24.1
K1-2B pH = 5.19 @ 24.1°C

1115 PM The solutions were covered with Kimwipes & placed on a gyratory shaker set at ~100 rpm.

3 10-mL aliquots of solution K1-2X10 were taken by Eppendorf max. pipettor (pre-rinsed with solution) & put in 15-mL PP bottles labeled K1-2X10a (or b, c). 500 µl of 1.05 M tartaric acid/triethanolamine solution was added to each solution. The 1.05 M TT solution used is from GC-05-252.

K1-2-E: At Δ time = 2, 4, 22, 27, 46, 70, 94, 166, 214, 262, 286, & 334 hours two 10-mL samples will be taken with a max pipet from solutions K1-2A & B. The aliquots will be put in 15-mL PP bottles labeled K1-2AXia (or b) and K1-2BXia (or b), where i is the sampling time number (1, 2, 3...12). 500 µl of 1.05 M TT will be added to each sample.

Also, the pH & temperature of each solution will be read & recorded.

A fresh portion of pH buffer (Fisher 5.0 lot #58102-500) was measured after each sampling to monitor the condition of the buffers in the bath.

| date | time | pH |
|------|------|---------------|
| 4/21 | 1328 | 5.02 @ 25.7°C |
| 4/23 | 0927 | 5.01 @ 22.5°C |
| 4/24 | 1108 | 5.02 @ 22.9°C |
| 4/25 | 0937 | 5.01 @ 23.1°C |
| 4/30 | 0939 | 5.03 @ 21.7°C |

solution K1-2A

| sampling number | time | Δ hours | pH | soln temp °C | U concentration, ppb 1 | 2 |
|--------------------|-------------------------|----------------|------------------------|-----------------|---------------------------|---|
| 1 | 4/21 1320 | 2.1 | 5.42 | 25.5 | | |
| 2 | 4/21 1516 | 4.0 | 5.55 | 26.6 | | |
| 3 | 4/22 0913 | 22.0 | 5.87 ^{PM} | 24.2 | | |
| 4 | 4/22 1412 | 27.0 | 5.90 ^{8/4/99} | 24.9 | | |
| 5 | 4/23 ^{PM} 0917 | 46.0 | 6.20 | 23.1 | | |
| 6 | 4/24 ^{PM} 1056 | 71.7 | 6.32 | 23.6 | | |
| 7 | 4/25 0918 | 94.1 | 6.51 | 23.3 | | |
| 8 | 4/28 0915 | 166.0 | 6.52 | 23.2 | | |
| 9 | 4/30 0915 | 214.0 | 6.63 | 21.9 | | |
| 10 | 5/2 0918 | | 6.68 | 21.9 | | |
| 11 | 5/3 0918 | | 6.63 | 22.3 | | |
| 12 | 5/5 0917 | | 6.65 | 22.1 | | |

solution K1-2B

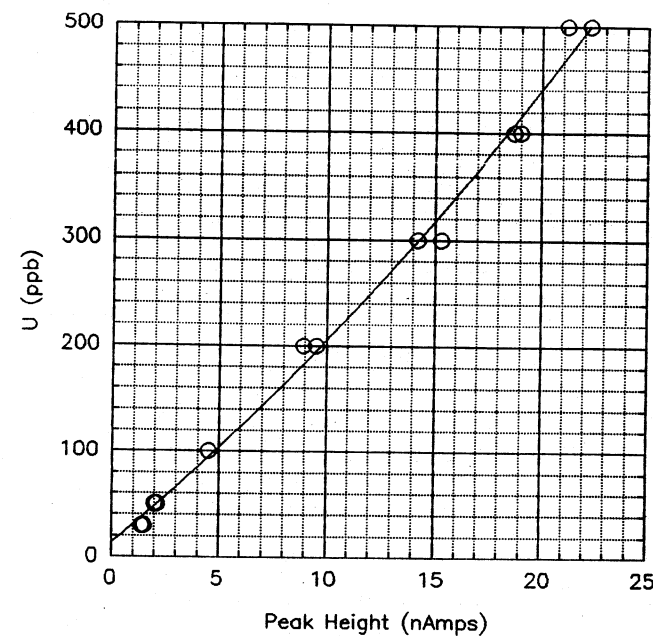
| sampling number | time | hours | pH | soln temp °C | U concentr. ppb |
|--------------------|-----------|-------|------|-----------------|--------------------|
| | | | | | 1 2 |
| 1 | 4/21 1324 | 2.2 | 5.44 | 25.6 | |
| 2 | 4/21 1518 | 4.1 | 5.50 | 26.5 | |
| 3 | 4/22 0915 | 22.0 | 5.88 | 24.2 | |
| 4 | 4/22 1414 | 27.0 | 6.04 | 24.9 | |
| 5 | 4/23 0922 | 46.1 | 6.30 | 23.1 | |
| 6 | 4/24 1059 | 71.7 | 6.26 | 23.5 | |
| 7 | 4/25 0924 | 94.2 | 6.52 | 23.2 | |
| 8 | 4/28 0920 | 166.1 | 6.54 | 23.0 | |
| 9 | 4/30 0920 | 214.1 | 6.64 | 21.9 | |
| 10 | 5/2 0922 | | 6.71 | 21.9 | |
| 11 | 5/3 0923 | | 6.68 | 22.1 | |
| 12 | 5/5 0919 | | 6.68 | 22.2 | |

1230 TD

Began to calibrate using the standards with .1M NaNO₃. After Reading the Procedure written I switched to standards without Na⁺. The calibration values and curve are presented below.

UNA0421.SPG: Tue, 21-Apr-92

| U (ppm) 1 | Pk Ht (nAmps) 2 | Regress-20rd 3 |
|--------------|--------------------|-------------------|
| 500 | 21.2 | 13.34 |
| 500 | 22.3 | 16.964 |
| 400 | 18.7 | 0.22099 |
| 400 | 19 | 0 |
| 300 | 14.2 | 0 |
| 300 | 15.3 | 0 |
| 200 | 8.9 | 0 |
| 200 | 9.5 | 0 |
| 100 | 4.5 | 0 |
| 100 | 4.5 | 0 |
| 50 | 2.1 | 0 |
| 50 | 2 | 0.99713 |
| 30 | 1.5 | 0 |
| 30 | 1.4 | 0 |



INITIAL

The solutions were run, and their U concentrations, as well as a check standard, were ^{TD 4/14/92} more than 5% off and will be rerun.

| | RUN1 | RUN2 | U conc(ppb) |
|----------|------|------|-------------|
| KI-1*1UA | 19.5 | 20 | 435 |
| *1UB | 20 | 19.1 | 429 |
| *1UC | 19.2 | 20 | 431 |

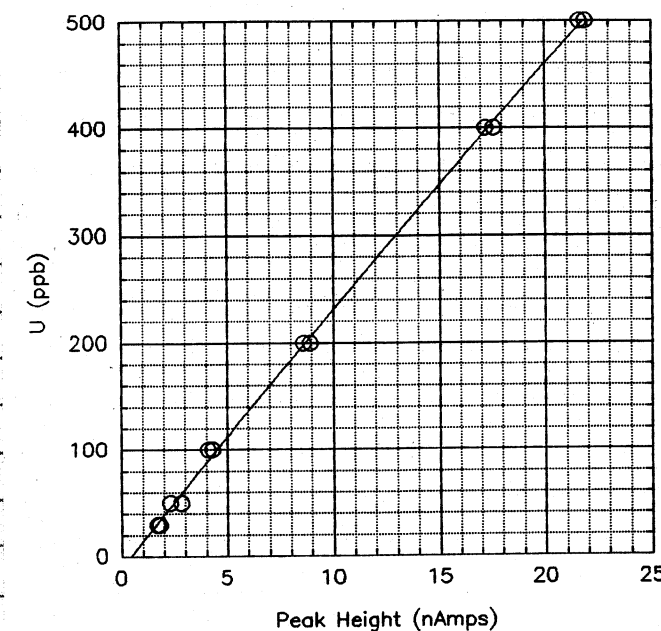
4/22/92 0930 TD

Began analyzing solutions from the kinetics experiment KI-1 on the Polarograph using DPS. The Polarograph was calibrated with the Uranium Standards from 30 ppb - 500 ppb prepared 4-20-92. Plots for the runs can be found in Polarographic Analyses of Uranium Vol II.

CALIBRATION VALUES AND CALIBRATION CURVE

UDPS0422.SPG: Wed, 22-Apr-92

| U (ppm) 1 | Pk Ht (nAmps) 2 | Regress-20rd 3 |
|--------------|--------------------|-------------------|
| 30 | 1.7 | -10.291 |
| 30 | 1.8 | 24.77 |
| 50 | 2.8 | -0.062835 |
| 50 | 2.3 | 0 |
| 100 | 4.3 | 0 |
| 100 | 4.1 | 0 |
| 200 | 8.6 | 0 |
| 200 | 8.9 | 0 |
| 400 | 17.6 | 0 |
| 400 | 17.2 | 0 |
| 500 | 21.6 | 0 |
| 500 | 21.9 | 0.99957 |



U CONCENTRATION ANALYSIS

KI-1_A.SPG: Wed, 22-Apr-92

| KI-1*A* 1 | Pk Ht1 (nA) 2 | Pk Ht2 (nA) 3 | U conc (ppb) 4 | Avg conc(ppb) 5 |
|--------------|------------------|------------------|-------------------|--------------------|
| IUA | 19.2 | 19.1 | 441 | -- |
| IUB | 15.6 | 16.3 | 369 | -- |
| IUC | 22 | 21.4 | 498 | 445 |
| 1A | 18.8 | 19.6 | 442 | -- |
| 1B | 17.8 | 17.2 | 404 | 423 |
| 2A | 16.4 | 15.5 | 369 | -- |
| 2B | 19.2 | 18.9 | 439 | 404 |
| 3A | 16.9 | 17.8 | 401 | -- |
| 3B | 18.1 | 16.2 | 396 | 399 |
| 4A | 16.9 | 17.1 | 393 | -- |
| 4B | 15.8 | 16.2 | 370 | 382 |
| 5A | 12.3 | 12.3 | 285 | -- |
| 5B | 13.8 | 13.7 | 318 | 302 |
| 200 PPB | 8.9 | 8.9 | (205) | |

4/24/92 PM Kinetics experiment K1-1 continued
1320

K1-1-F: After the last samples for K1-1XA and K1-1XB were taken, three 10-ml aliquots of solution K1-1KV were taken by Eppendorf Maxipettor (pre-rinsed with the solution being sampled) and put in 15-ml polypropylene bottles. 500 μ l of 1.05 M tartaric acid/triethanolamine solution (GC-05-252) was added to each bottle, and the bottles labeled K1-1XFUxa (or b, C).

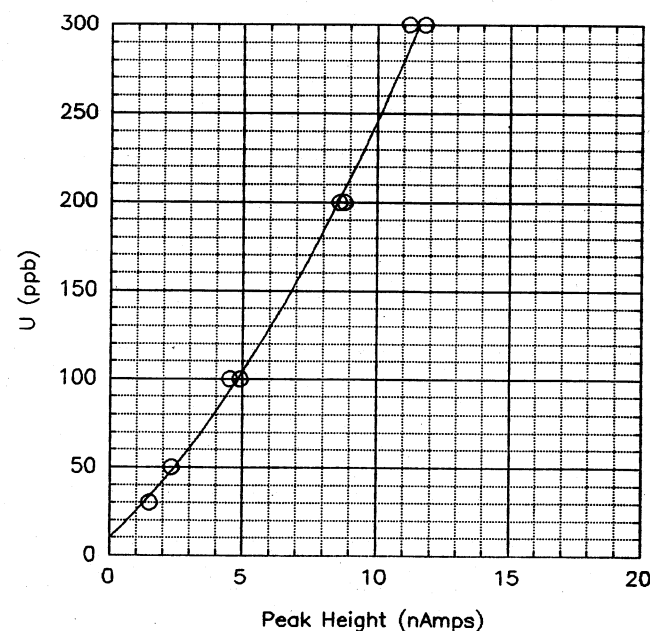
0800 TD

Analyzed more of the K1-1XA solutions for U concentration on the Polarograph using DPS. Standards were prepared on 4-20-92. Plots can be found in Polarographic Analyses of Uranium, Vol II.

CALIBRATION VALUES AND CURVE

UDPS0424.SPG: Fri, 24-Apr-92

| U (ppm) | Pk Ht (nAmps) | Regress-20rd |
|---------|---------------|--------------|
| 1 | 2 | 3 |
| 30 | 1.5 | 10.285 |
| 50 | 2.3 | 14.168 |
| 100 | 4.9 | 0.93686 |
| 100 | 4.5 | 0 |
| 200 | 8.8 | 0 |
| 200 | 8.6 | 0 |
| 300 | 11.2 | 0 |
| 300 | 11.8 | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0.99761 |



U CONCENTRATION ANALYSIS

KI-1_AI.SPG: Fri, 24-Apr-92

| KI-1*A* | Pk Ht1 (nA) | Pk Ht2 (nA) | U conc (ppb) | Avg conc (ppb) |
|---------|-------------|-------------|--------------|----------------|
| 1 | 2 | 3 | 4 | 5 |
| 6A | 7.9 | 7.3 | 172 | -- |
| 6B | 9.2 | 10.1 | 234 | -- 203 |
| 7A | 7.9 | 7.8 | 179 | -- |
| 7B | 6.6 | 7.2 | 153 | -- 166 |
| 8A | 3.6 | 3.7 | 74.5 | -- |
| 8B | 4.1 | 4.4 | 87.4 | -- 90 |
| 9A | 1.9 | 1.8 | 39.7 | -- |
| 9B | 2.3 | 2.3 | 47.8 | -- 43.8 |
| 10A | 1.4 | 1.8 | 35.4 | -- |
| 10B | 1.5 | 1.5 | 33.6 | -- 34.5 |
| 200 PPB | 8.4 | 8.2 | (192) | |

27 APRIL 1992 0830 TD

Prepared a new set of U standards in Tartaric Acid/Triethanolamine (1.05M). The amount of the standard given below and 5ml 1.0M Tartaric Acid/Triethanolamine (2/19/92) was diluted to 100ml in a volumetric flask with Ultrapure H₂O. The RICCA U standard, 1000 ppm, lot #D145, exp. date June 92 was used to prepare the 10 ppm - 50 ppm standard and these prepared standards were used to prepare the others.

| | |
|-----------------------------------|-----------------------------------|
| 50 ppm \rightarrow 5ml 1000 ppm | 500 ppb \rightarrow 1ml 50 ppm |
| 40 ppm \rightarrow 4ml 1000 ppm | 400 ppb \rightarrow 1ml 40 ppm |
| 30 ppm \rightarrow 3ml 1000 ppm | 300 ppb \rightarrow 1ml 30 ppm |
| 20 ppm \rightarrow 2ml 1000 ppm | 200 ppb \rightarrow 1ml 20 ppm |
| 10 ppm \rightarrow 1ml 1000 ppm | 100 ppb \rightarrow 1ml 10 ppm |
| 5 ppm \rightarrow 10ml 50 ppm | 50 ppb \rightarrow 0.1ml 50 ppm |
| 4 ppm \rightarrow 10ml 40 ppm | 30 ppb \rightarrow 0.1ml 30 ppm |
| 3 ppm \rightarrow 10ml 30 ppm | 10 ppb \rightarrow 0.1ml 10 ppm |
| 2 ppm \rightarrow 10ml 20 ppm | |
| 1 ppm \rightarrow 10ml 10 ppm | |

Glass volumetric pipets were used for volumes \geq 5ml and fixed volume Eppendorf pipets were used for other volumes.

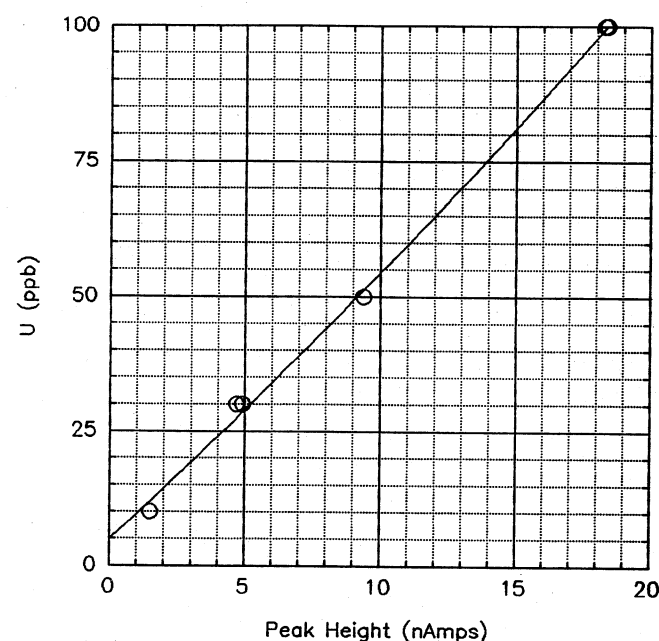
1200 TD

Continued U concentration analysis of KI-1*~~A~~
 Solutions using DPS on the Polarograph.
 The standards from 10ppb-100ppb prepared earlier today
 were used for calibration. 60 s deposition time was
 used. Plots can be found in Polarographic Analyses of
 Uranium vol 12.

CALIBRATION VALUES AND CURVE

UDPS0427.SPG: Mon, 27-Apr-92

| U (ppb) | Pk Ht (nAmps) | Regression |
|---------|---------------|------------|
| 1 | 2 | 3 |
| 10 | 1.5 | 4.8077 |
| 30 | 4.7 | 4.7001 |
| 30 | 4.9 | 0.025951 |
| 50 | 9.4 | 0 |
| 50 | 9.4 | 0 |
| 100 | 18.3 | 0 |
| 100 | 18.4 | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0.99891 |



RESULTS OF U ANALYSES

KI-1_AII.SPG: Mon, 27-Apr-92

| KI-1*A* | Pk Ht1 (nA) | Pk Ht2 (nA) | U conc (ppb) | Avg conc (ppb) |
|---------|-------------|-------------|--------------|----------------|
| 1 | 2 | 3 | 4 | 5 |
| 8A | 14.3 | 15.9 | 81.7 | -- |
| 8B | 16.9 | 17.2 | 92.5 | 87.1 |
| 9A | 6.2 | 6.5 | 35.7 | -- |
| 9B | 7.6 | 5 | 35.5 | 35.6 |
| 10A | 3.8 | 3.5 | 22.3 | -- |
| 10B | 3.3 | 3.4 | 20.8 | 21.6 |
| 11A | 2.6 | 2.9 | 17.9 | -- |
| 11B | 2.8 | 2.9 | 18.4 | 18.2 |
| 12A | 2 | 1.4 | 12.9 | -- |
| 12B | 1.4 | 1.8 | 12.4 | 12.7 |
| 50 PPB | 7.1 | 6.7 | (38.5) | |

1630P~~PM~~ Expt KI-1 continued

The polarographic analysis of the KI-1 samples
 indicate that the amount of U in solution had not
 yet equilibrated, although the pH had. So one more
 set of samples (labeled KI-1*~~A~~*13a,b + KI-1*~~B~~*13a,b)
 were taken as described on pages 22-23.

5 seep. 58 PM

4/28/92 P~~PM~~ Expt K2 prep - Calibration of glass dropper
0936

A glass dropper for use with 0.05 M HNO₃ was
 calibrated by adding 0.05 M HNO₃ solution
 (GC-05-301) dropwise to a 5 mL volumetric
 flask.

$$\frac{127 \text{ drops}}{5 \text{ mL}} = \frac{1 \text{ drop}}{x \text{ mL}} \quad x = 0.0394 \text{ mL/drop}$$

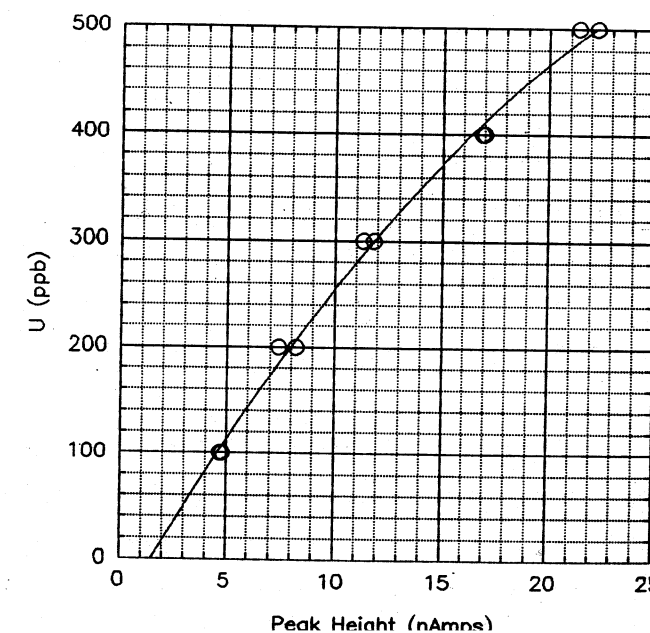
0800 TD

Began analyzing the U concentrations of the
 samples from KI-1*B on the Polarograph using DPS.
 The standards, from 100-500ppb, were prepared on 27 APRIL 1992.
 Plots of these runs can be found in Polarographic
 Analyses of Uranium, vol 12.

CALIBRATION VALUES AND CURVES

UDPS0428.SPG: Tue, 28-Apr-92

| U (ppm) | Pk Ht (nAmps) | Regress-20rd |
|---------|---------------|--------------|
| 1 | 2 | 3 |
| 100 | 4.8 | -51.454 |
| 100 | 4.7 | 35.265 |
| 200 | 7.4 | -0.46939 |
| 200 | 8.2 | 0 |
| 300 | 11.8 | 0 |
| 300 | 11.3 | 0 |
| 400 | 16.9 | 0 |
| 400 | 17 | 0 |
| 500 | 21.4 | 0 |
| 500 | 22.3 | 0 |
| | | 0.99766 |



28 APRIL 1992

RESULTS OF U CONCENTRATION ANALYSES

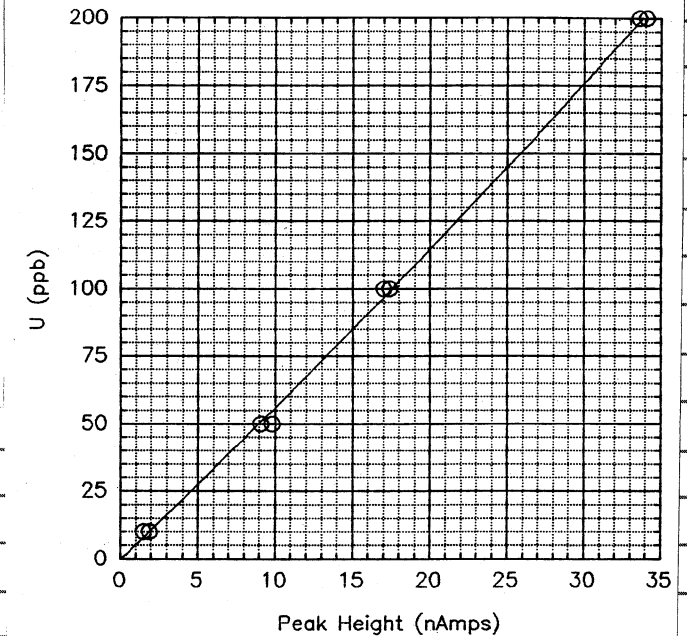
KI-1_B.SPG: Tue, 28-Apr-92

| | KI-1*B* 1 | Avg Pk Ht(nA) 2 | U conc (ppb) 3 | Av conc(ppb) 4 |
|----|--------------|--------------------|-------------------|-------------------|
| 1 | IUA | 19.7 | 460 | -- |
| 2 | IUB | 19 | 449 | -- |
| 3 | IUC | 18.9 | 447 | -- |
| 4 | FUA | 20.6 | 476 | 452 |
| 5 | FUB | 19.8 | 463 | -- |
| 6 | FUC | 21 | 482 | 474 |
| 7 | 1A | 16.6 | 405 | -- |
| 8 | 1B | 21 | 482 | 444 |
| 9 | 2A | 19.9 | 464 | -- |
| 10 | 2B | 18.2 | 435 | 450 |
| 11 | 3A | 17.2 | 416 | -- |
| 12 | 3B | 16.1 | 395 | 406 |
| 13 | 400 PPB | 16 | (393) | -- |
| 14 | 4A | 17.3 | 418 | -- |
| 15 | 4B | 14.1 | 352 | 385 |
| 16 | 5A | 14.3 | 357 | -- |
| 17 | 5B | 14.9 | 370 | 364 |
| 18 | 6A | 12.1 | 307 | -- |
| 19 | 6B | 13.1 | 330 | 319 |
| 20 | 7A | 10.4 | 265 | -- |
| 21 | 7B | 10.4 | 265 | 265 |
| 22 | 400 PPB | 15.6 | (385) | -- |

CALIBRATION VALUES AND CURVE

UDPS0429.SPG: Wed, 29-Apr-92

| U (ppb) 1 | Pk Ht (nAmps) 2 | Regression 3 |
|--------------|--------------------|-----------------|
| 10 | 1.9 | -0.36578 |
| 10 | 1.5 | 5.5002 |
| 50 | 9.8 | 0.012615 |
| 50 | 9.1 | 0 |
| 100 | 17 | 0 |
| 100 | 17.4 | 0 |
| 200 | 34.1 | 0 |
| 200 | 33.6 | 0 |
| | | 0 |
| | | 0.99946 |



29 APRIL 1992 0800 TD

Continued the U concentration analysis of the solutions from KI-1*B on the Polarograph using DPS and 60 second deposition time. The Polarograph was calibrated from 10ppb-200ppb using the standards prepared on 27 April 1992. Plots of these runs can be found in Polarographic Analyses of Uranium, Vol. 12.

RESULTS OF U CONCENTRATION ANALYSIS

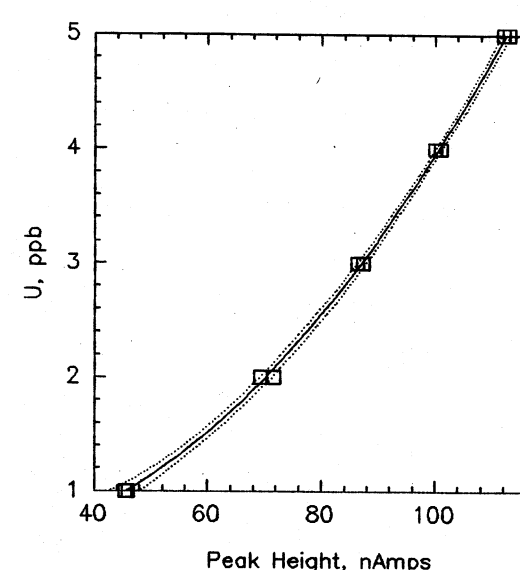
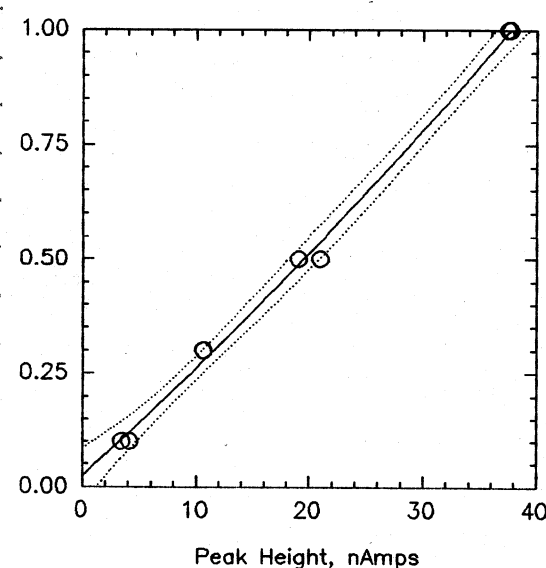
KI-1_BI.SPG: Wed, 29-Apr-92

| KI-1*B* 1 | Avg Pk Ht(nA) 2 | U conc (ppb) 3 | Av conc(ppb) 4 |
|--------------|--------------------|-------------------|-------------------|
| 8A | 13 | 73.3 | -- |
| 8B | 17 | 96.8 | 85.1 |
| 9A | 9.4 | 52.5 | -- |
| 9B | 9.7 | 54.2 | 53.4 |
| 10A | 3.2 | 17.4 | -- |
| 10B | 3.3 | 17.9 | 17.7 |
| 11A | 2.4 | 12.9 | -- |
| 11B | 1.8 | 9.6* | 11.3 |
| 12A | 1.6 | 8.5* | -- |
| 12B | 1.4 | 7.4* | 8.0* |
| 13A | 1 | 5.2* | -- |
| 13B | 1.1 | 5.7* | 5.5* |
| *A*13A | 1.3 | 6.8* | -- |
| *A*13B | 1.3 | 6.8* | 6.8* |
| 100 PPB | 16 | (90.1) | -- |

1 MAY 1992 1000 TD

Analyzed solutions A-I-2*PHI*VR2 on the Polarograph using DPS. Cell one was calibrated from 1 ppm - 1 ppm and cell 2 was calibrated from 1 ppm - 5 ppm. The standards used were prepared 4/27/92. Plots for these runs can be found in Polarographic Analyses of Uranium, vol 12.

CALIBRATION



UDPS0501.SPG: Fri, 1-May-92

| U ppm 1 | Pk Height 2 | Regress-Ord2 3 |
|------------|----------------|-------------------|
| 0.1 | 3.4 | 0.024979 |
| 0.1 | 4.1 | 0.022787 |
| 0.3 | 10.6 | 8.1406e-005 |
| 0.3 | 10.7 | 0 |
| 0.5 | 19.1 | 0 |
| 0.5 | 21 | 0 |
| 1 | 37.5 | 0 |
| 1 | 37.7 | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0.99823 |

UDPS0501.SPG: Fri, 1-May-92

| U ppm 5 | Pk Height 6 | Regress-Ord2 7 |
|------------|----------------|-------------------|
| 1 | 45.9 | 0.56698 |
| 2 | 45.4 | -0.011111 |
| 2 | 71.7 | 0.00044948 |
| 3 | 69.3 | 0 |
| 3 | 86.3 | 0 |
| 3 | 87.3 | 0 |
| 4 | 100 | 0 |
| 4 | 101 | 0 |
| 5 | 113 | 0 |
| 5 | 112 | 0 |
| | | 0 |
| | | 0.99953 |

RESULTS OF U CONCENTRATION ANALYSIS

AI2R2.SPG: Fri, 1-May-92

| A-I-2*PHI*R2 1 | Pk Ht1(nAmps) 2 | Pk Ht2(nAmps) 3 | U (ppm) 4 |
|-------------------|--------------------|--------------------|--------------|
| 2.5 | 76.6 | 80.6 | 2.47 |
| 3 | 93.6 | 92.1 | 3.42 |
| 3.5 | 103 | 103 | 4.2 |
| 4 | 100 | 101 | 4 |
| 4.5 | 73 | 76.6 | 2.25 |
| 5 | 20.3 | 19 | 0.504 |
| 5.5 | 18.3 | 18.5 | 0.471 |
| 6 | 25.5 | 24.4 | 0.643 |
| 6.5 | 28.8 | 28.3 | 0.741 |
| 7 | 72.5 | 66.9 | 1.98 |
| 7.5 | 62.9 | 63.9 | 1.67 |
| 8 | 74.4 | 77.3 | 2.31 |
| 8.5 | 65.5 | 67.1 | 1.81 |
| 9 | 69.1 | 71.1 | 2 |
| IU4 | 107 | 109 | 4.62 |
| IU5 | 106 | 107 | 4.49 |
| IU6 | 105 | 107 | 4.45 |
| 3 PPM | 86.9 | 88.9 | (3.07) |
| .2 PPM | 8.4 | 9.6 | (.237) |

4 MAY 1992 0930 TD

Prepared new standards of U in .05 M Tartaric Acid/Triethanolamine. The amount of standard given below and 5 ml of the 1.0M TT prepared 2/19/92 were diluted to 100ml with Ultrapure H₂O in a Volumetric flask. The RICCA U std, lot # D145, exp. date Jun 92, was used to prepare the 10ppm-50ppm U stds which were then used in preparing the others. Glass volumetric pipets were used for volumes > 5ml.

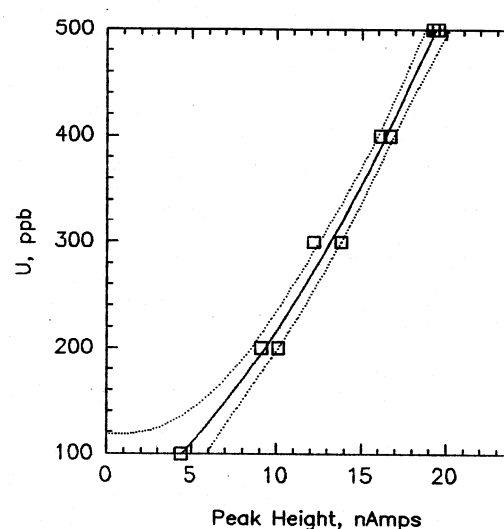
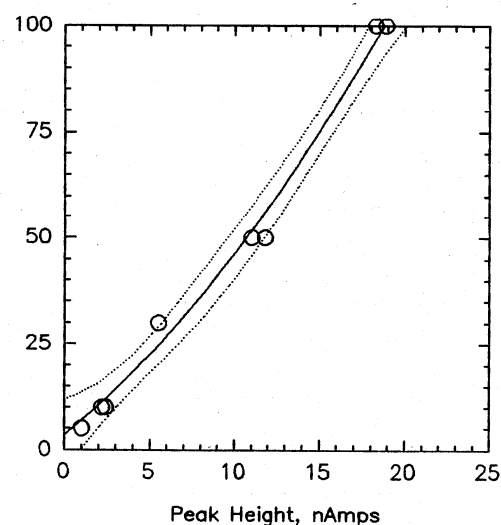
50 ppm → 5ml 1000 ppm
 40 ppm → 4ml 1000 ppm
 30 ppm → 3ml 1000 ppm
 20 ppm → 2ml 1000 ppm
 10 ppm → 1ml 1000 ppm
 5 ppm → 10 ml 50 ppm
 4 ppm → 10 ml 40 ppm
 3 ppm → 10 ml 30 ppm
 2 ppm → 10 ml 20 ppm
 1 ppm → 10 ml 10 ppm

500 ppb → 1ml 50 ppm
 400 ppb → 1ml 40 ppm
 300 ppb → 1ml 30 ppm
 200 ppb → 1ml 20 ppm
 100 ppb → 1ml 10 ppm
 50 ppb → 0.1 ml 50 ppm
 30 ppb → 0.1 ml 30 ppm
 10 ppb → 0.1 ml 10 ppm
 5 ppb → 0.1 ml 5 ppm

1200 TD

Began analyzing U concentrations of solutions
A-I-3*PHI*U on the Polarograph using
DPS. 60s deposition was used for concentrations
below 100 ppb. The standards prepared earlier today
were used for calibration. Plots can be found in
Polarographic Analyses of Uranium, vol. 12.

CALIBRATION CURVES



UDPS0504.SPG: Mon, 4-May-92

| U ppb 1 | Pk Height 2 | Regress-Ord2 3 | 4 |
|------------|----------------|-------------------|----------|
| 5 | 1 | 3.4945 | <----- |
| 10 | 2.4 | 3.3108 | ANALYZED |
| 10 | 2.2 | 0.097556 | ON |
| 30 | 5.5 | 0 | CELL #1 |
| 30 | 5.5 | 0 | WITH 60S |
| 50 | 11.8 | 0 | DEPOSIT |
| 50 | 11 | 0 | |
| 100 | 18.9 | 0 | |
| 100 | 18.3 | 0 | |
| | | 0 | |
| | | 0 | |
| | | 0.99437 | |

UDPS0504.SPG: Mon, 4-May-92

| U ppb 5 | Pk Height 6 | Regress-Ord2 7 | 8 |
|------------|----------------|-------------------|----------|
| 100 | 4.4 | 34.711 | <----- |
| 200 | 9.1 | 11.891 | ANALYZED |
| 200 | 10.1 | 0.62696 | ON |
| 300 | 13.8 | 0 | CELL #2 |
| 300 | 12.2 | 0 | WITH 60S |
| 400 | 16.1 | 0 | DEPOSIT |
| 400 | 16.7 | 0 | |
| 500 | 19.5 | 0 | |
| 500 | 19.2 | 0 | |
| | | 0 | |
| | | 0.99456 | |

RESULTS OF U ANALYSIS

AI3R2.SPG: Tue, 5-May-92

| A-I-3*PHI*R1 1 | Pk Ht1(nAmps) 2 | Pk Ht2(nAmps) 3 | U (ppm) 4 |
|-------------------|--------------------|--------------------|--------------|
| 2.5 | 12.7 | 13.2 | 294 |
| 3 | 12.9 | 15 | 323 |
| 3.5 | 8.2 | 8.2 | 174 |
| 4 | 9.8 | 9.6 | 44.8 |
| 4.5 | 1.1 | 0.73 | 6.6 |
| 5 | 1.2 | 1.2 | 7.6 |
| 5.5 | -- | -- | -- |
| 6 | 1.3 | 1.1 | 7.6 |
| 6.5 | 1.6 | 4.2 | 13.9 |
| 7 | 1.8 | 2.1 | 10.3 |
| 7.5 | 4.1 | 3.4 | 17.3 |
| IU4 | 17.5 | 17.7 | 438 |
| IU5 | 14.5 | 14.6 | 340 |
| IU6 | 17 | 17 | 418 |
| 400 PPB | 15.2 | 16.2 | (376) |

NOTE: no peak was found for solution 5.5. It will
be reanalyzed with the remaining solutions.

6MAY 1992 0900 TD

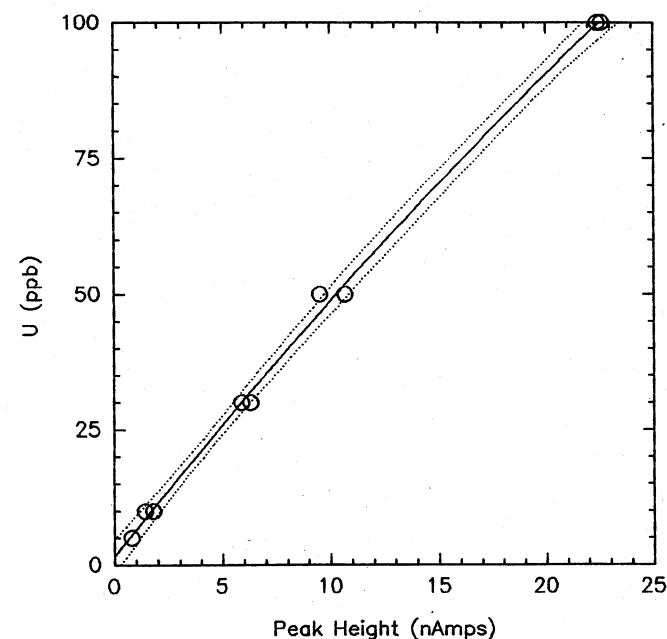
U to 5162
Made a new set of standards in the same
manner as on 4 May using the same
equipment, reagents and method.

1200 TD

Began to finish analysing the remaining A-I-3 reverse solutions. The standards prepared earlier were used in calibration. DPS was once again used with 60s deposition time. Plots can be found in Polarographic Analyses of Uranium vol 12.

UDPS0506.SPG: Wed, 6-May-92

| U (ppb) 1 | Pk Ht (nAmps) 2 | Regression 3 |
|--------------|--------------------|-----------------|
| 5 | 0.8 | 1.5546 |
| 10 | 1.8 | 5 |
| 10 | 1.4 | -0.027734 |
| 30 | 6.3 | 0 |
| 30 | 5.9 | 0 |
| 50 | 9.5 | 0 |
| 50 | 10.7 | 0 |
| 100 | 22.6 | 0 |
| 100 | 22.4 | 0 |
| | | 0.99894 |



AI32R2.SPG: Wed, 6-May-92

| A-I-3*PH1*R1 1 | Pk Ht1(nAmps) 2 | Pk Ht2(nAmps) 3 | U (ppb) 4 |
|-------------------|--------------------|--------------------|--------------|
| 8 | 3.1 | 4.5 | 20.2 |
| 8.5 | 8.9 | 8.4 | 42.7 |
| 9 | 11.9 | 14.4 | 62.5 |
| 50 PPB | 10.3 | 10.5 | 50.6 |

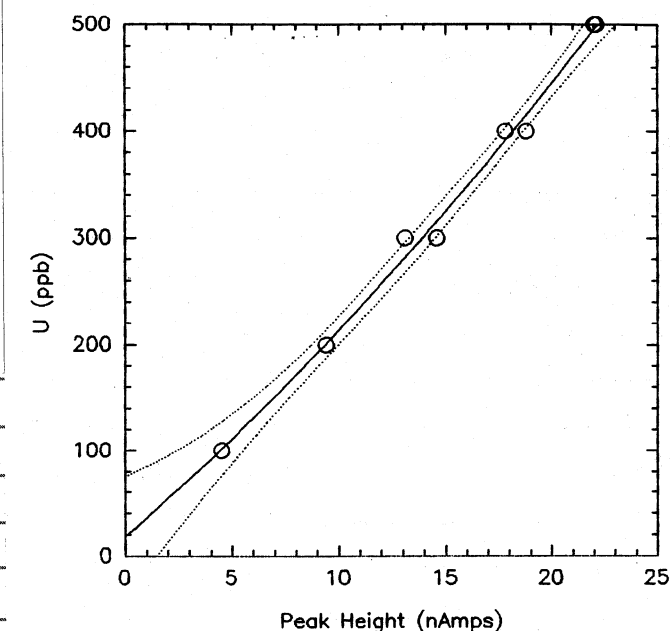
7 MAY 1992 0800 TD

Began analysing samples from K1-2 x A. Calibration from 100 to 500 ppb was done using the standards prepared on 6 MAY 1992 (p.56). DPS with 0.5 deposition time was the method used. Plots can be found in Polarographic Analyses of Uranium, vol 12.

CALIBRATION

UDPS0507.SPG: Thu, 7-May-92

| U (ppm) 1 | Pk Ht (nAmps) 2 | Regress-20rd 3 |
|--------------|--------------------|-------------------|
| 1 | 100 | 4.5 |
| 2 | 200 | 9.4 |
| 3 | 200 | 9.4 |
| 4 | 300 | 13.1 |
| 5 | 300 | 14.6 |
| 6 | 400 | 17.8 |
| 7 | 400 | 18.8 |
| 8 | 500 | 22 |
| 9 | 500 | 22.1 |
| 10 | | 0 |
| 11 | | 0 |
| 12 | | 0.99701 |



RESULTS OF U ANALYSIS

| SAMPLE | PK HT 1 (nA) | PK HT 2 (nA) | U CONC (ppb) |
|--------|-------------------------------------|-------------------------------------|------------------------------|
| 1A | 19.1 18.0 TO 19.1 ^{5/1/92} | 19.3 20.0 TO 19.3 ^{5/1/92} | 441 TO 425 ^{5/1/92} |
| 1B | 19.1 20.4 TO 19.1 ^{5/1/92} | 20.3 | 438 ^{5/1/92} |
| 2A | 18.0 | 20.0 | 420 |
| 2B | 18.5 | 17.8 | 399 |
| 3A | 14.5 | 17.4 | 347 |
| 3B | 16.5 | 17.9 | 377 |
| 4A | 13.3 | 15.0 | 305 |
| 4B | 13.5 | 13.9 | 294 |
| 5A | 8.3 | 9.4 | 189 |
| 5B | 8.3 | 9.2 | 187 |

K1-2+A U conc ANALYSES

| SAMPLE | PKH+1 (nA) | PKH+2 (nA) | U conc (ppb) |
|--------|------------|------------|----------------|
| 6A | 5.6 | 6.3 | TD 130 5142 |
| 6B | 6.9 | 5.4 | 134 |
| 14A | 18.8 | 20.9 | 441 |
| 14B | 20.4 | 20.1 | 451 |
| 14C | 21.5 | 22.9 | 501 |
| 200ppb | 9.9 | 9.3 | (194) |

5/8/92 PM Kinetics Experiment K2
(Initial pH ~ 4.5, 5.5; N₂ atmosphere)

Objective: Determine the time it takes to reach sorption equilibrium between uranimum solutions + clinophibolite

K2-1: Solution K2-1 (2 bottles)
- Initial $\Sigma U = 500$ ppb
Initial $\Sigma Na = 0$
Initial pH = 4.5
Initial solution mass ~ 950g
Wt. zeolite used = 0.95g
N₂ atmosphere

5/10/92 PM The Labconco Protector Controlled Atmosphere Glove Box Model 50600 will be used in this experiment to provide a controlled N₂ atmosphere. The glove box pressure (\pm inches H₂O) varies slightly with room temperature, pressure + humidity, but returns to the original value when the T, P, & h return to previous values. eg,

reading: 0906 hrs, 5/10/92, 1.0 in H₂O 163.3°F 64%
1905 " 1.5 164.6°F 67%

Three liters of degassed ultra pure water will be used for the experiment. The water will be

degassed by boiling the water a liter at a time for 30+ minutes, while purging the water with N₂ gas. The water will then be capped with a rubber stopper + transferred into the glove box.

11 MAY 1992 0600 TD

Prepared new U standards according to the table below. The 1000 ppm U standard is from the ²³⁸U RICA, lot # D145, exp. date Jun 92. The 1.0M TT was prepared 2/19/92.

| | | |
|-----------|--|---|
| 50 ppm = | 5 ml 1.0M TT, 5 ml 1000 ppm U std, dilute to 100ml w/ ultrapure H ₂ O | |
| 40 ppm = | " 4 ml 100 ppm U std, | " |
| 30 ppm = | " 3 ml 1000 ppm U std, | " |
| 20 ppm = | " 2 ml 1000 ppm U std, | " |
| 10 ppm = | " 1 ml 1000 ppm U std, | " |
| 5 ppm = | " 5 ml 1000 ppm U std, | " |
| 500 ppb = | " 1 ml 50 ppm U std, | " |
| 400 ppb = | " 1 ml 40 ppm U std, | " |
| 300 ppb = | " 1 ml 30 ppm U std, | " |
| 200 ppb = | " 1 ml 20 ppm U std, | " |
| 100 ppb = | " 1 ml 10 ppm U std, | " |
| 50 ppb = | " 1 ml 50 ppm U std, | " |
| 30 ppb = | " 1 ml 30 ppm U std, | " |
| 10 ppb = | " 1 ml 10 ppm U std, | " |
| 5 ppb = | " 1 ml 5 ppm U std, | " |

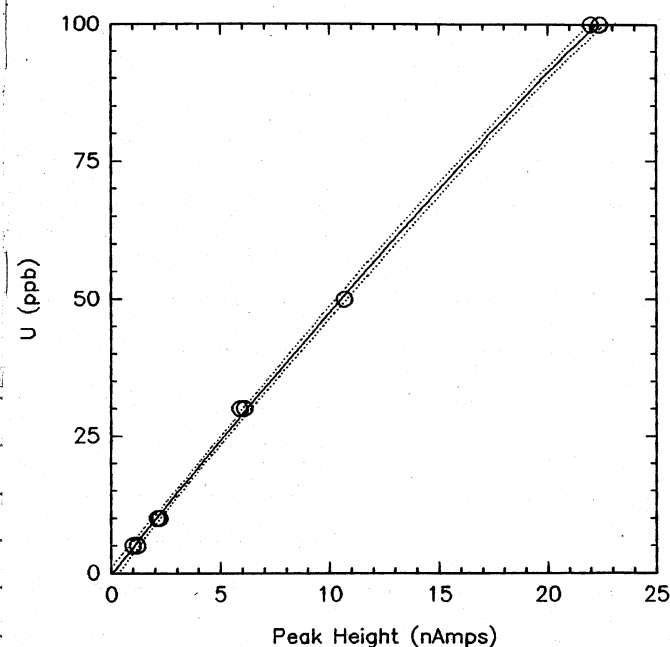
1030 TD

Continued U concentration analysis of K1-2+A solutions. Calibration was done with the standards prepared earlier today. The method used was DPS with 60 s deposition. Plots can be found in Polarographic Analysis of Uranium, vol 12.

CALIBRATION

UDPS0511.SPGMon, 11-May-92

| U (ppb) 1 | Pk Ht (nA) 2 | Regression 3 |
|--------------|-----------------|-----------------|
| 5 | 1 | -0.35232 |
| 5 | 1.2 | 5.014 |
| 10 | 2.1 | -0.022456 |
| 10 | 2.2 | 0 |
| 30 | 5.9 | 0 |
| 30 | 6.1 | 0 |
| 50 | 10.7 | 0 |
| 50 | 10.7 | 0 |
| 100 | 22.4 | 0 |
| 100 | 22 | 0 |
| | | 0.99975 |



RESULTS OF U ANALYSIS

KI-2_A7.SP5: Mon, 11-May-92

| KI-2*A* 1 | Pk Ht1 (nA) 2 | Pk Ht2 (nA) 3 | U conc (ppb) 4 |
|--------------|------------------|------------------|-------------------|
| 7A | 12.7 | 12.5 | 59.6 |
| 7B | 12.7 | 11.7 | 57.8 |
| 8A | 5.3 | 5.1 | 25.4 |
| 8B | 5.2 | 4.5 | 23.8 |
| 9A | 3.2 | 2.8 | 14.8 |
| 9B | 2.7 | 2.8 | 13.6 |
| 10A | 2.2 | 2.3 | 11.1 |
| 10B | 2.2 | 2.3 | 11.1 |
| 50 PPB | 10.8 | 10.1 | (49.8) |

5/12/92 PM Kinetics Expt K2 continued

0804 PM As a check on how oxygen-free the nitrogen-purged / boiled water in the glove box is the pH will be measured. The Orion 920 pH/BCE meter will be used, with a Ross glass

electrode + automatic temperature compensator (ATC) probe.

The pH meter was calibrated using two Fisher pH buffers (~~5B102-500~~, pH 5 + ~~5B108-1~~ pH 7) which were put in the box yesterday. ⁹¹⁸²⁸⁴⁻²⁴ ⁹¹⁰⁹⁴³⁻²⁴ PM 5/11/92

0.041 M NaNO₃ solution was made as described on p. 4 from the same 4 M stock solution. ~15 ml was transferred into an appropriately marked PP bottle, + the bottle + a 250 µl Eppendorf micropipet were put in the box.

10 ml of the deoxygenated ultrapure water in the box was taken by max pipet + put in a 15 ml PP bottle + the bottle labeled. 250 µl of the 0.041 M NaNO₃ solution was added to the water (final Na concentration: 1×10^{-3} M) + the bottle shaken to mix. The pH + temperature of the solution were read.

0842 PM pH = 5.29 @ 21.8 °C

pH closer to 7.0 is expected of water without O₂ in it. The previously degassed water will be removed from the box, + another liter boiled + stirred + purged with N₂ gas for an hour's duration. This water will be put in the box, all wed to cool, + measured for pH as before.

0600 TO

The polarograph was calibrated from 5-100 ppb with the standards prepared yesterday. The analysis proved unreproducible and was stopped. It will be continued tomorrow.

1315 PM $1 \times 10^{-3} \text{ M NaNO}_3$ w 1 hour degassed water: 5.49 pH @ 29.2°C
 w 1/2 hour degassed water: 5.30 @ 24.1°C
 see ↑ 0842 today: pH = 5.29 @ 21.8°C

1533 PM The Mettler DL21 titrator used with the Mettler DG11-5C combination pH electrode was calibrated using Fisher pH buffers 4.0 & 7.0. A $1 \times 10^{-3} \text{ M}$ solution of ultrapure water was made by adding 2 mL of the 0.041 M NaNO_3 solution made earlier today to 80 mL of ultrapure water. The pH was read.

pH = 5.60 @ 68.4°F room temp
 After 1/2 hour N_2 gas purge:
 pH = 6.37 @ 68.6°F room temp

5/13/92 PM

0803 The liter of water degassed yesterday afternoon was read for pH after meter calibration with the pH 5 & 7 buffers used yesterday. Then 10 mL of the water was taken & had 250 μL of the 0.041 M NaNO_3 soln from yesterday afternoon added. The 10 mL $1 \times 10^{-3} \text{ M NaNO}_3$ solution was read for pH as well.

0822 PM pH degassed water: 6.16 @ 20.3°C
 pH $1 \times 10^{-3} \text{ M NaNO}_3$: 5.58 @ 23.2°C

Boiling, purging with N_2 gas & stirring for an hour appears to be removing an appreciable amount of dissolved oxygen from the water. The same procedure will be used for the rest of the water to be used in kinetics experiment K2.

5/14/92 PM Expt K2-1 continued

0803 PM 100 mL of 0.05 M HNO_3 was prepared by diluting one mL of 2 M HNO_3 (GC-05-301) to 100 mL in a volumetric flask. ~30 mL of the 0.05 M solution was put in a labeled PP bottle & put in the glove box. This will be used for adjusting the pH of the 500 ppb U solution (see next step).

Step K2-1-A: 2000 mL of 500 ppb ($2 \times 10^{-6} \text{ M}$) U solution was prepared by diluting 1 mL of a 1000 ppm U stock solution (Barco, lot # LQ-10, exp. date Dec '92, $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in water) to 2000 mL in a volumetric flask using ultrapure de oxygenated water.

Step K2-1-B: Approximately 950 g aliquots of the 500 ppb solution were poured into two 1000 mL PP bottles labeled K2-1A and K2-1B. The remaining 500 ppb solution was poured into a 125 mL PP bottle labeled K2-1C.

The pH meter was calibrated using the Fisher pH buffers w. pH = 4.0, 5.0, & 7.0 (lot # 5B9824, 5B102-500, 910359-24, 913284-24, & 910943-24).
 8/4/94

Step K2-1-C: The initial pH of solutions K2-1A & KB were measured & recorded. ~ 3.14×10^{-5} moles HNO_3 need to be added to the 950 mL U solution according to EQ3 calculations, so that the resulting solution pH will be ~4.5. 152 drops (5/14/92 @ 0.0394 mL/drop, p49) of the 0.05 M HNO_3 prepared earlier today was added to each 500 ppb solution. The solutions were then put on the New Brunswick gyratory shaker set at 120 rpm.

initial pH values

K2-1X A 5.66 @ 21.4°C

K2-1X B 5.58 @ 21.3°C

0940 PM Solutions K2-1X A and K2-1X B were set on the shaker.

1022 PM Two 0.95g portions of Na-clinoptilolite were weighed onto weighing papers. The papers were carefully folded & put in the glove box.

wt paper A: 0.9506g
wt paper B: 0.9496g

1507 PM Step K2-1-D: After at least an hour has passed, the pH of the solutions was remeasured

K2-1X A 4.74 @ 24.0°C

K2-1X B 4.63 24.1

1515 PM The 0.95g clinoptilolite was added, & the solutions put back on the gyratory shaker.

Three 10-mL aliquots of solution K2-1X U was taken by Eppendorf maxipipettor (pre-rinsed with solution) and transferred into 15 mL PP bottles labeled K2-1X ~~U~~ 10X a (or b, c). 500 μ L of the 1.05 M tartaric acid/triethanolamine solution (GC-05-252) was added to each bottle.

At Δ time = 2, 4; 22, 27; 46; 70; 94; 166; 214; 262; 286; & 334 hours, two 10 mL samples will be taken from solutions K2-1X A + X B and put in 15 mL PP bottles labeled K2-1X A X i a (or b)

and K2-1X B X i a (or b), where i is the sampling time number (1, 2, 3...). 500 μ L of 1.05 M tartaric acid/triethanolamine solution will be added to each sample by Eppendorf micro pipet.

Also, the pH & temperature of solutions K2-1X A and X B will be measured & recorded at each sampling time.

solution K2-1X A

| sampling time number | sampling time | Δ hours | pH | soln temp °C |
|----------------------|---------------|----------------|------|--------------|
| 1 | 5/14/92 1703 | | 4.87 | 24.5 |
| 2 | 1913 | | 5.02 | 24.8 |
| 3 | 5/15/92 1725 | | 5.91 | 24.0 |
| 4 | 5/16/92 1330 | | 6.27 | 21.3 |
| 5 | 5/17/92 1320 | | 6.60 | 21.7 |
| 6 | 5/22/92 1428 | | 6.74 | 22.2 |
| 7 | 5/24/92 1730 | | 6.87 | 20.3 |
| 8 | 5/18/92 1259 | | 6.65 | 21.9 |
| 9 | 5/25/92 1310 | | 6.70 | 22.2 |
| 10 | 5/27/92 1330 | | 6.62 | 21.6 |
| 11 | 5/29/92 1320 | | 6.60 | 21.8 |
| 12 | 5/30/92 1222 | | 6.76 | 19.5 |
| 13 | 6/10/92 0855 | | 6.81 | 20.2 |

solution K2-1XB

| sampling number | sampling time | Δ hours | pH | solu temp °C |
|--------------------|------------------|----------------|------|-----------------|
| 1 | 5/14/92 1705 | | 4.86 | 24.6 |
| 2 | 1915 | | 5.21 | 24.8 |
| 3 | 5/15/92 1730 | | 5.97 | 23.9 |
| 4 | 5/16/92 1333 | | 6.51 | 21.4 |
| 5 | 5/17/92 1322 | | 6.63 | 21.8 |
| 6 | 5/22/92 1432 | | 6.72 | 22.3 |
| 7 | 5/24/92 1734 | | 6.77 | 20.3 |
| 8 | 5/18/92 1407 | | 6.66 | 20.1 |
| 9 | 5/25/92 1312 | | 6.67 | 22.1 |
| 10 | 5/27/92 1332 | | 6.69 | 21.7 |
| 11 | 5/29/92 1322 | | 6.62 | 22.0 |
| 12 | 5/30/92 1224 | | 6.74 | 19.6 |
| 13 | 6/10/92 0858 | | 6.77 | 20.3 |

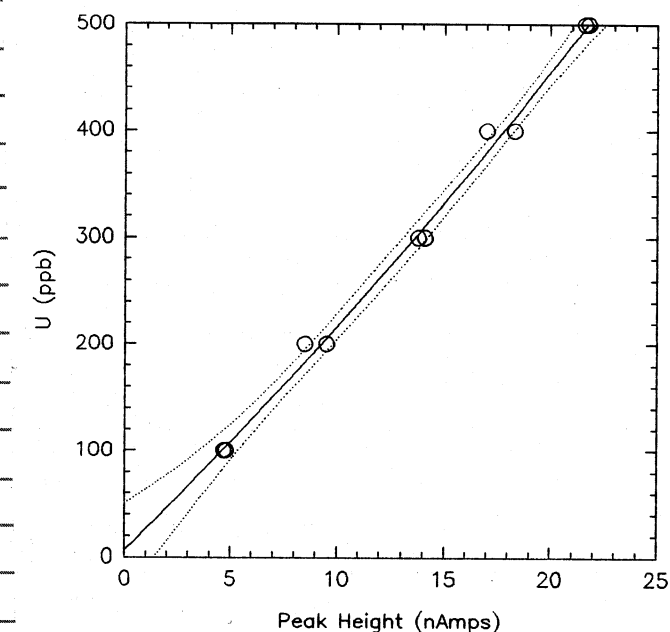
0860 TD

Analyzed U concentration of KI-2XB solutions on the Polarograph using DPS with no deposition time. The standards prepared 11 MAY 1992, p. 59, were used for calibration. Plots for these runs can be found in Polarographic Analyses of Uranium, vol 12.

CALIBRATION VALUES

| UDPS0514.SP5: Thu, 14-May-92 | | | |
|------------------------------|--------------------|-------------------|--|
| U (ppb) 1 | Pk Ht (nAmps) 2 | Regress-20rd 3 | |
| 100 | 4.8 | 6.611 | |
| 100 | 4.7 | 19.539 | |
| 200 | 8.5 | 0.14508 | |
| 200 | 9.5 | 0 | |
| 300 | 13.8 | 0 | |
| 300 | 14.1 | 0 | |
| 400 | 17 | 0 | |
| 400 | 18.3 | 0 | |
| 500 | 21.6 | 0 | |
| 500 | 21.8 | 0 | |
| | | 0.99746 | |

CALIBRATION CURVE



RESULTS OF U CONC. ANALYSIS

KI-2_B1.SP5: Thu, 14-May-92

| KI-2*B* | Pk Ht1 (nA) | Pk Ht2 (nA) | U conc (ppb) | |
|---------|-------------|-------------|--------------|--------|
| 1 | 2 | 3 | 4 | |
| 1 | 1A | 17.2 | 17.9 | 394 |
| 2 | 1B | 17.3 | 18.7 | 405 |
| 3 | 2A | 17.2 | 17.4 | 388 |
| 4 | 2B | 17 | 17.4 | 386 |
| 5 | 3A | 14.5 | 14.7 | 323 |
| 6 | 3B | 15.4 | 15.1 | 338 |
| 7 | 4A | 13.4 | 13.9 | 300 |
| 8 | 4B | 12.8 | 14.7 | 303 |
| 9 | 300PPB | 13.3 | 13.1 | (290) |
| 10 | 5A | 8.3 | 8.4 | 180 |
| 11 | 5B | 7.6 | 8.1 | 169 |
| 12 | 6A | 6.1 | 6.9 | 140 |
| 13 | 6B | 6 | 7.3 | 143 |
| 14 | 300PPB | 13.1 | 14.5 | (304). |
| 15 | -1*B*5A | 14 | 14.4 | 313 |
| 16 | -1*B*5B | 14.5 | 14.1 | 316 |
| 17 | -1*B*6A | 11.4 | 11.9 | 254 |
| 18 | -1*B*6B | 9.1 | 9.4 | 200 |
| 19 | -1*B*7A | 7.1 | 7.2 | 154 |
| 20 | -1*B*7B | 7.2 | 6.4 | 146 |
| 21 | 300PPB | 13.1 | 12.3 | (278) |

5/15/92 PM Expt K2 continued 5/14/94
carbon dioxide ion selective

A carbonate sensitive electrode will be used to check the amount of oxygen contamination in the glove box. A 0.01 M NaHCO₃ solution is used to pre-soak the electrode. 100 mL will be made by dissolving 0.0840 g NaHCO₃ (lot # 897186A) in a 100 mL volumetric flask, using ultrapure water. for 5/14/94

wt used: 0.0839g

1050 PM 1x10⁻⁴ 1x10⁻³ & 1x10⁻² M carbon dioxide standards will be prepared by doing serial dilution, using 0.1 M Sodium Bicarbonate Carbon Dioxide Standard (lot # 73464). 10 mL of the 0.1 M standard brought to volume in a 100 mL volumetric flask will ⇒ 1x10⁻⁴ M solution, + so on.

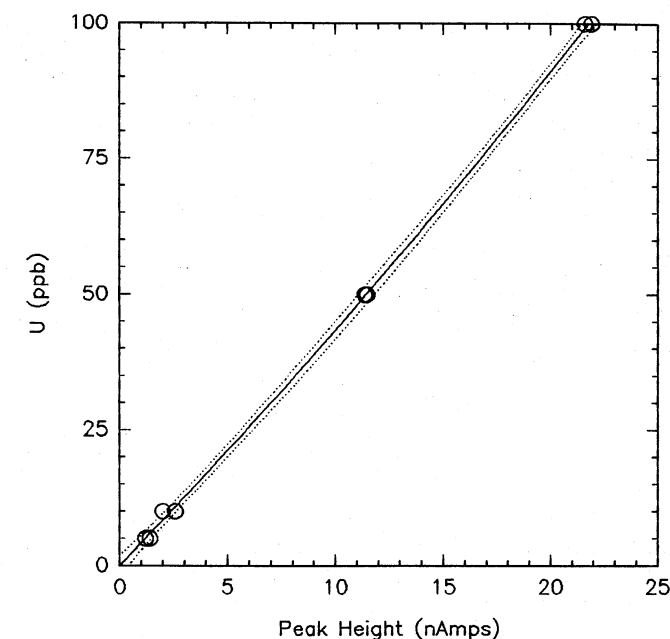
0900 TD

Finished analyzing the U concentrations of the solutions from K1-2-B on the polarograph using DPS and 80 s deposition. The standards prepared on 11 May 1992 were used for calibration. The Polarograph was calibrated twice, as the drift in the electrode was greater than 5% after half the samples were run. The sample 12A did not ^{5/15/92} yield a peak for Uranium. Plots can be found in Polarographic Analyses of Uranium, Vol 13.

1st CALIBRATION CURVE

UDPS0515.SP5: Fri, 15-May-92

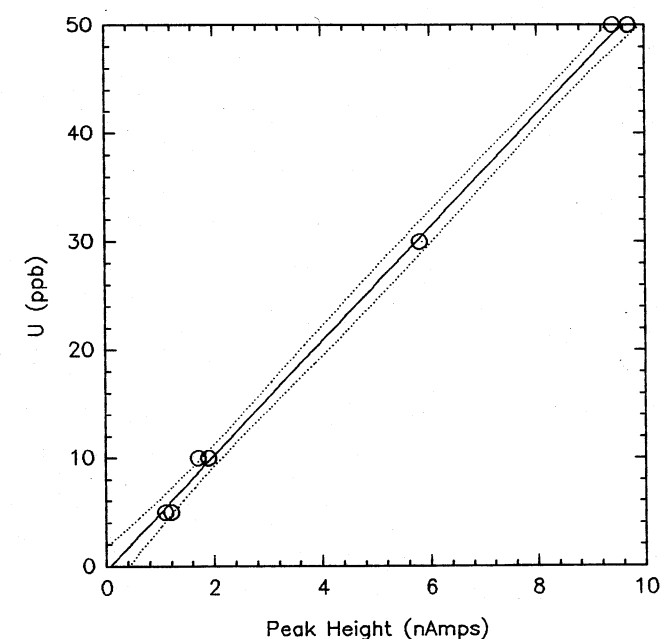
| U (ppb) 1 | Pk Ht (nAmps) 2 | Regression 3 |
|--------------|--------------------|-----------------|
| 1 | 5 | 1.4 |
| 2 | 5 | 1.2 |
| 3 | 10 | 2 |
| 4 | 10 | 2.6 |
| 5 | 50 | 11.5 |
| 6 | 50 | 11.4 |
| 7 | 100 | 21.9 |
| 8 | 100 | 21.6 |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | 0.99977 |



2ND CALIBRATION CURVE

UDPS515A.SP5: Fri, 15-May-92

| U (ppb) 1 | Pk Ht (nAmps) 2 | Regression 3 |
|--------------|--------------------|-----------------|
| 50 | 9.4 | -0.43573 |
| 50 | 9.7 | 5.3338 |
| 30 | 5.8 | -0.0066869 |
| 30 | 5.8 | 0 |
| 10 | 1.7 | 0 |
| 10 | 1.9 | 0 |
| 5 | 1.2 | 0 |
| 5 | 1.1 | 0 |
| | | 0 |
| | | 0 |
| | | 0 |
| | | 0.99911 |



RESULTS OF U CONC. ANALYSIS

KI-2_B7.SP5: Fri, 15-May-92

| KI-2*B* 1 | Pk Ht1 (nA) 2 | Pk Ht2 (nA) 3 | U conc (ppb) 4 |
|--------------|------------------|------------------|-------------------|
| 7A | 13 | 12.8 | 56.8 |
| 7B | 14.5 | 15.3 | 66.2 |
| 8A | 6.6 | 6.8 | 28.6 |
| 8B | 6.5 | 6 | 26.6 |
| 9A | 4.1 | 4 | 17 |
| 9B | 3.6 | 4.5 | 17 |
| 50 PPB | 9.4 | 9.7 | (41.3) |
| 10A | 2.9 | 2.6 | 14.2 |
| 10B | 2.9 | 2.5 | 13.9 |
| 11A | 2.4 | 2.4 | 12.3 |
| 11B | 2 | 2.6 | 11.8 |
| 12A | -- | -- | -- |
| 12B | 2 | 1.9 | 9.9 |
| 30 PPB | 4.6 | 4.8 | (24.5) |

5/18/92 PM
1008

To check the pH of CO_2 equilibrated water + the resulting pH when it is purged with N_2 gas over a period of time, the Mettler DC21 titrator was calibrated using the same pH buffers as before (4.0 + 7.0 lot # 910359-24 + 910943-24). Ultra pure water that has been kept in a carboy with a porous top was measured for pH, and then steadily purged with N_2 gas while being stirred.

pH of water equilibrated with atmospheric CO_2 :

| | |
|------|------------------------|
| 1025 | pH = 5.49 |
| 1035 | 5.78 (10 min purge) |
| 1045 | 5.82 |
| 1055 | 5.89 |
| 1102 | 5.78 (gas turned up) - |
| 1115 | 6.00 |
| 1125 | 6.65 |
| 1129 | 6.46 |
| 1135 | 6.54 |
| 1140 | 6.53 (gas turned up) |
| 1145 | 6.70 |

1150 pH = 6.99
1155 7.16

Sample cup was left open to atmosphere for 30s + remeasured. pH = 7.16

1015 ID

Prepared 1000 ml of 1.0M Tartaric Acid/Triethanolamine by dissolving 150.09g Tartaric Acid and 149.14g Triethanolamine in 1000 ml Ultrapure H_2O in a volumetric flask. The resulting solution was placed in a clean, labeled PP bottle.

wt. Tartaric Acid used 150.0911g (lot # 920136)
wt. Triethanolamine used 149.1420g (lot # 911834)

1200PM pH buffers re read on titrator:
pH 4.00 : 4.01
7.00 : 7.01

1335PM The ultrapure water will be repurged to remove the oxygen again, the pH read, the water quickly capped + then transferred into the glove box. The pH will then be re-read, + re read again at a later time.

pH before put in glove box: 6.95
(1407: pH inside : 6.66 @ 20.1°C)

1200

Prepared new U standards in .05M Tartaric Acid/Triethanolamine by diluting a certain amt of standard (given on next page) and 5ml 1.0M TT prepared earlier today to 100ml in a volumetric flask using ultrapure H_2O . The 1000 ppm std is RICA lot # D145.

Std

50 ppm → 5 ml 1000 ppm
 40 ppm → 4 ml 1000 ppm
 30 ppm → 3 ml 1000 ppm
 20 ppm → 2 ml 1000 ppm
 10 ppm → 1 ml 1000 ppm
 500 ppb → 1 ml 50 ppm
 400 ppb → 1 ml 40 ppm
 300 ppb → 1 ml 30 ppm

200 ppb → 2 ml 100 ppm
 100 ppb → 1 ml 10 ppm
 50 ppb → 0.1 ml 50 ppm
 30 ppb → 0.1 ml 30 ppm
 10 ppb → 0.1 ml 10 ppm
 5 ppb → 1 ml 500 ppb

1956 PM pH of carbon water in glove box: 6.31 @ 21.8 °C

19 MAY 1992 0900 TD

Ran standards made yesterday on the Polarograph using DPS. No peaks were found, so new standards will be made.

1015 TD

Prepared new U standards in 0.05M TT. The 10M TT was prepared 18 MAY 1992 (p. 71). The 1000 ppm U std is from BANCO, Lot #10-10. The prep. was the same as yesterday (shown above). A 15 ppb standard was made by diluting 0.1 ml 10 ppm and 0.1 ml 5 ppm as done with the others. The 5 ppm was made by diluting 5 ml 1000 ppm in the same way.

1515 PM - Checking CO₂ content of water -

A 1×10^{-3} M solution of KCl was made by weighing 0.0752g KCl (lot #885 967) + dissolving in water (ultrapure) allowed to equilibrate with the atmosphere. The solution was brought to volume in a 100 mL volumetric flask. 10 mL of this were brought to volume in a 100 mL volumetric flask w. ultrapure water.

1525 PM The Mettler DL21 titrator was again used to measure the pH of the solution (see p 70). N₂ gas will be bubbled through the solution, + the pH monitored.

| | initial pH | @ 67.1 °F room temp | time |
|---------|------------|---------------------|------|
| w purge | 5.62 | | 1527 |
| | 5.68 | | 1537 |
| | 5.71 | | 1604 |
| | 5.73 | | 1620 |
| | 5.74 | | 1627 |
| | | | 1637 |

1639 PM Solution will be re-purged tomorrow due to time factor.

5/20/92 0815 PM - Checking CO₂ content of water -

Another 1×10^{-3} M solution of KCl was made by weighing 0.0743g KCl (lot #885 967) + dissolving in CO₂ equilibrated ultrapure water in a 100 mL volumetric flask. The flask was brought to volume, giving a 1×10^{-3} M solution. 10 mL of this were taken by volumetric pipet + brought to volume in a 100 mL volumetric flask.

Resulting solution: 1×10^{-3} M KCl

0830 PM The Mettler DL21 titrator will be used again to monitor the pH of the solution (see p 70). N₂ gas will be bubbled through the solution, + the pH monitored. Vol. soln: 80 mL.

| | initial pH | @ 64.2 °F room temp | time |
|---------|------------|---------------------|------|
| w purge | 5.43 | | 0846 |
| | 5.96 | | 0902 |
| | 6.78 | | 0934 |

pH: 6.93 @ 65.0°F room temp 0945
 6.96 1000
 6.96 1010

1155 PM The vacuum from the transfer chamber repeatedly pulled the lid from the solution container, & the solution was spilled. A new 1×10^{-3} M KCl soln was made up, using 0.0752g KCl (lot # 885967) + same procedure as on p. 73.

5/21/92 PM The solution will be measured for pH by using both the Mettler DL21 titrator + glass electrode and the 290A Orion pH meter + Ross electrode at the same time.

| beginning pH | mettler | Orion | temp |
|--------------|---------|-------|---------------|
| 9036 | 5.59 | 5.57 | 19.3°C (soln) |
| time 1042 | 5.65 | 5.61 | 19.4 |
| 1057 | 5.65 | 5.60 | " |
| 1117 | 5.63 | 5.57 | |

Purge + reading was stopped. The solution will be remade.

1100 PM 1×10^{-3} KCl solution remade, using 0.0746g KCl (lot # 885967) for the 1×10^{-2} M solution + carboy water as before for solution dilution.

1120 PM Both the DL21 + the pH meter were re-calibrated using Fisher pH buffers 4.0 + 7.0, lot # 5 910359-24 + 910943-24.

| | DL21 pH | Orion pH | time | temp |
|---------------|---------|----------|------|---------------|
| beginning pH: | 5.46 | 5.45 | 1130 | 19.7°C (soln) |
| | 5.77 | 5.72 | 1137 | |
| | 5.93 | 5.86 | 1142 | |
| | 6.11 | 6.12 | 1154 | 19.8°C |
| | 6.24 | 6.21 | 1215 | |
| | 6.49 | 6.45 | 1245 | |
| | 6.54 | 6.52 | 1314 | |
| | 6.64 | 6.62 | 1348 | |
| | 6.80 | 6.77 | 1408 | |
| | 6.58 | 6.54 | 1435 | |
| | 6.93 | 6.91 | 1450 | |
| | 7.13 | 7.22 | 1454 | |
| | 7.20 | 7.19 | 1502 | |
| | 6.99 | 6.97 | 1532 | |
| | 6.74 | 6.72 | 1602 | |
| | 7.18 | 7.18 | 1637 | |
| | 7.21 | 7.21 | 1647 | |
| | 7.03 | 7.03 | 1705 | |

1706 PM Gas was shut off + stirring stopped
 pH buffer 4.0: 4.09 @ 1711

5/22/92 PM Experiment B-IV (1 bottle per pH)

- Control experiment to determine pH change of 0.1 M NaNO_3 after addition of clinoptilolite

Initial $\Sigma U = 0$
 Initial pH = 2.0 to 9.5, every 0.5 pH unit
 $I = 0.1$ M NaNO_3
 volume = 100 mL
 wt zeolite = 0.200g
 $p\text{CO}_2 = 10^{-3.48}$ bars
 pH adjusted with HNO_3 or NaHCO_3
 according to EQ 3 calculations

0752PM 5kp B-IV-1: 1.0, 0.5, 0.05, 0.005 + 0.0005 M HNO_3 solutions were made up from concentrated HNO_3 (lot # A-200-212). 6.3 mL of conc. HNO_3 was diluted to 100 mL in a volumetric flask to give a 1.0 M solution; 50 mL of the 1.0 M solution was diluted to 100 mL to give a 0.5 M solution; 10 mL was taken from the 0.5 M solution and diluted to 100 mL to give a 0.05 M solution, & so on.

0832PM 5kp B-IV-2: 2000 mL of 0.1 M NaNO_3 solution was prepared by dissolving 17.0002 g of NaNO_3 (lot # 7808 KODJE) in water & bringing to volume in a 2000 mL volumetric flask.

100 mL aliquots were taken from the 0.1 M NaNO_3 solution by volumetric pipet & placed in 125 mL PP bottles labeled B-IV * i, where $i = 2.0, 2.5 \dots 9.5$.

1001PM The pH meter was calibrated using Fisher pH buffers pH = 4.0, 5.0 + 7.0 (lot # 5910359-24, 910284-24 + 910743-24). The initial pH of each solution was recorded, along with soln. temp.

| soln. B-IV * i | pH / temp | | pH / temp |
|----------------|---------------|---------|-------------|
| i = 2.0 | 5.69 / 24.2°C | i = 6.5 | 5.71 / 24.4 |
| 2.5 | 5.68 " | 7.0 | 5.69 " |
| 3.0 | 5.72 24.1 | 7.5 | 5.69 " |
| 3.5 | 5.65 " | 8.0 | 5.67 " |
| 4.0 | 5.70 24.2 | 8.5 | 5.66 " |
| 4.5 | 5.71 " | 9.0 | 5.69 " |
| 5.0 | 5.72 24.3 | 9.5 | 5.65 " |
| 5.5 | 5.71 " | | |
| 6.0 | 5.73 " | | |

EQ3 calculations were used to determine how much HNO_3 or NaHCO_3 is needed to adjust the 0.1 M NaNO_3 solutions (eq. pH predicted = 5.6 by EQ3) to i values. The following amounts of HNO_3 were added as listed:

| | | |
|---------|-------------------------|---------------|
| 3 drops | 1.0 M HNO_3 | to B-IV * 2.0 |
| 2 | 0.5 | 2.5 |
| 7 | 0.05 | 3.0 |
| 2 | 0.05 | 3.5 |
| 7 | 0.005 | 4.0 |
| 2 | 0.005 | 4.5 |
| 6 | 0.0005 | 5.0 |
| 1 drop | 0.0005 M HNO_3 | to B-IV * 5.5 |

The following amounts of NaHCO_3 were added as listed:

| | | |
|-----------|------------------|---------------|
| 0.00009 g | NaHCO_3 | to B-IV * 6.0 |
| 0.00018 | | * 6.5 |
| 0.00056 | | * 7.0 |
| 0.00211 | | * 7.5 |
| 0.00629 | | * 8.0 |
| 0.02128 | | * 8.5 |
| 0.07333 | | * 9.0 |
| 0.33583 g | NaHCO_3 | to B-IV * 9.5 |

1232PM The solution bottles were covered with Kimwipes & set on a gyratory shaker at 100 rpm.

5/25/92 PM Expt A-IV-2 cont

Solutions A-IV-2 * pH_i will be remeasured for pH & then sampled as in GC-07-26.

See page 80
for correction
5/25/92

1345 PM The pH meter was calibrated using Fisher pH buffers 2.0, 4.0, 5.0, 7.0, & 9.0 from the water bath.

| sample | pH | temp °C | Δ pH (4/11/92) |
|----------------|------|---------|-----------------------|
| A-II-2k pH 2.5 | 2.09 | 23.3 | .04 |
| 3.0 | 2.69 | 23.3 | .03 |
| 3.5 | 3.32 | 23.3 | .01 |
| 4.0 | 4.06 | 23.4 | .04 |
| 4.5 | 4.91 | 23.4 | .09 |
| 5.0 | 5.80 | 23.5 | .23 |
| 5.5 | 6.08 | 23.5 | .18 |
| 6.0 | 6.43 | 23.4 | .09 |
| 6.5 | 6.36 | 23.3 | .18 |
| 7.0 | 6.81 | 23.4 | .02 |
| 7.5 | 7.03 | 23.4 | .09 |
| 8.0 | 7.79 | 23.3 | .08 |
| 8.5 | 8.46 | 23.3 | .51.16 |

A-II-2k pH 9.0 9.15 23.3
8.94 23.3 .09
(pH meter recalibrated between pH = 9.0 & 10.0)

Compare with pH values taken GC-07-26.

0900

Began to calibrate the Polarograph from 100-500 ppb U but had to stop the analysis because a satisfactory calibration curve was not plotted. The first sample run was ~150 ppb too high. New standards will be made and the analysis begun again.

1200 TD

Prepared new U standards in 0.05 M Tartaric Acid / Triethanol amine by diluting a certain amount of standard (given below) with 5 ml 1.0 M TT (prepared 5/10/92) to 100 ml in a volumetric flask using Ultrapure H₂O. The lot # of the 1000 ppm Ustd is L0-10, from BANCO.

| | |
|-----------------------------|------------------------------|
| 50 ppm → 5 ml 1000 ppm Ustd | 300 ppb → 1.0 ml 30 ppm Ustd |
| 40 ppm → 4 ml 1000 ppm " | 200 ppb → 1 ml 20 ppm " |
| 30 ppm → 3 ml 1000 ppm " | 100 ppb → 1 ml 10 ppm " |
| 20 ppm → 2 ml 1000 ppm " | 50 ppb → 0.1 ml 50 ppm " |
| 10 ppm → 1 ml 1000 ppm " | 30 ppb → 0.1 ml 30 ppm " |
| 5 ppm → 0.5 ml 1000 ppm " | 10 ppb → 0.1 ml 10 ppm " |
| 500 ppb → 1.0 ml 50 ppm " | 5 ppb → 0.1 ml 5 ppm " |
| 400 ppb → 1.0 ml 40 ppm " | |

+31423 PM Expt A-II-2 cont.

One 10-ml aliquot was taken by Eppendorf max pipet from each solution A-II-2k pH_i and 3 10-ml aliquots were taken from solution A-II-2k 10⁰. The aliquots were put in 15 ml PP bottles labeled A-II-2k pH_i & UR3 and A-II-2k 10⁰ & 10¹, 10².

500 µl of 1.05 M tartaric acid / triethanolamine (GC-05-252) was added to each sample taken.

1540 PM Expt B-IV continued

The pH meter was recalibrated using Fisher pH buffers pH = 2.0, 4.0, 7.0, 9.0, & 10.0 from the water bath.

Solutions B-IV Ki were measured for pH.

4/4/94

| solution M | pH | temp °C |
|-------------|-------|---------|
| B-IV * 2.0 | 2.89 | 23.2 |
| 2.5 | 3.33 | 23.2 |
| 3.0 | 3.78 | 23.2 |
| 3.5 | 4.34 | 23.2 |
| 4.0 | 4.83 | 23.2 |
| 4.5 | 5.26 | 23.2 |
| 5.0 | 5.51 | 23.2 |
| 5.5 | 5.56 | 23.2 |
| 6.0 | 6.29 | 23.3 |
| 6.5 | 6.31 | 23.3 |
| 7.0 | 6.74 | 23.3 |
| 7.5 | 7.33 | 23.3 |
| 8.0 | 7.84 | 23.3 |
| 8.5 | 8.39 | 23.3 |
| 9.0 | 8.93 | 23.3 |
| 9.5 | 9.16 | 23.3 |
| buffer 10.0 | 10.00 | 23.3 |

1623 PM Solutions B-IV Ki were returned to the gyratory shaker.

5/26/92 PM 0940 Expt B-IV continued

Due to a calculation error using moles instead of molarity, enough HNO_3 was not added to the B-IV Ki solutions < 5.5. Allowing for the HNO_3 already added the amounts of HNO_3 that will be added to correct the problem are as follows:

29 d of 1.0 M HNO_3 to B-IV * 2.0
 9 d of 1.0 2.5

4/4/94

26 d of 0.5 M HNO_3 to solution B-IV * 3.0
 9 0.5 M 3.5

26 d of 0.05 M 4.0

9 d of 0.05 M 4.5

27 d of 0.005 M 5.0

1 d of 0.005 M HNO_3 to solution B-IV * 5.5

HNO_3 solutions used are the same prepared 5/22/92.

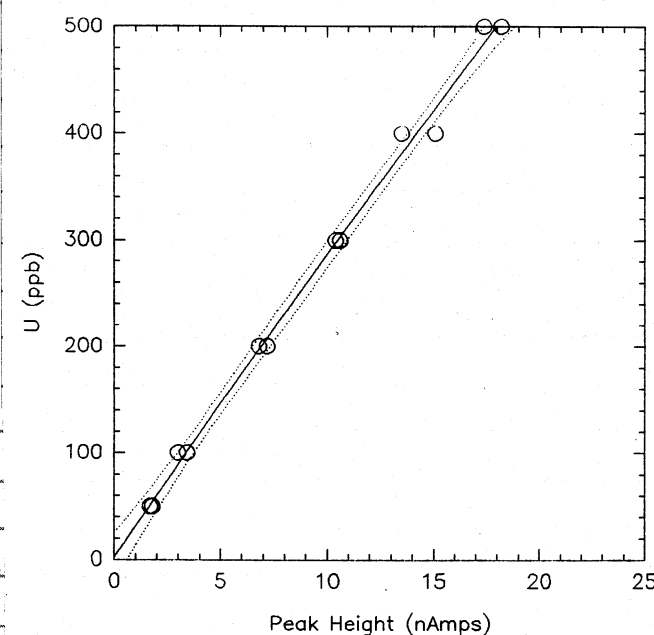
0800^{TD} Began analyzing samples from K2-1x4 on the Polarograph using DPS with no deposition. The standards prepared 5/25/92 were used for calibration. Plots can be found in Polarographic Analyses of Uranium, vol 13.

CALIBRATION CURVE

UDPS0526.SP5: Tue, 26-May-92

| U (ppb) | Pk Ht (nAmps) | Regress-20rd |
|---------|---------------|--------------|
| 1 | 50 | 1.7 |
| 2 | 50 | 1.8 |
| 3 | 100 | 3.4 |
| 4 | 100 | 3 |
| 5 | 200 | 7.2 |
| 6 | 200 | 6.8 |
| 7 | 300 | 10.4 |
| 8 | 300 | 10.6 |
| 9 | 400 | 13.5 |
| 10 | 400 | 15.1 |
| 11 | 500 | 17.4 |
| 12 | 500 | 18.2 |

2.2698
29.293
-0.0886
0
0
0
0
0
0
0
0.99765



1520 PM Expt B-IV continued

Since there is some doubt as to whether the HNO_3 solutions are of the correct concentrations, an extra 1000 mL of →

RESULTS OF U CONC ANALYSIS

K2-1_A1.SP5: Tue, 26-May-92

| K2-1* | A | Pk Ht1 (nA) | Pk Ht2 (nA) | U conc (ppb) |
|-------|---------|-------------|-------------|--------------|
| 1 | TD | 2 | 3 | 4 |
| 1 | 5/26/92 | | | |
| 1 | IUA | 17.9 | 20.9 | 537* |
| 2 | IUB | 18.9 | 19.6 | 533* |
| 3 | IUC | 19.4 | 19.4 | 537* |
| 4 | 1A | 18.6 | 18.7 | 518* |
| 5 | 1B | 18.6 | 18.8 | 519* |
| 6 | 2A | 18 | 18.1 | 502* |
| 7 | 2B | 17 | 18.8 | 498 |
| 8 | 300 PPB | 10.2 | 10.7 | (299) |
| 9 | 3A | 9.1 | 10 | 274 |
| 10 | 3B | 10.8 | 10.4 | 303 |
| 11 | 4A | 6.1 | 6 | 176 |
| 12 | 4B | 5.9 | 6.1 | 175 |
| 13 | 5A | 2.8 | 3 | 86.5 |
| 14 | 5B | 3.5 | 3.2 | 99.4 |
| 15 | 300 PPB | 10.5 | 9.7 | (289) |

* = OUT OF CALIBRATION RANGE

No PM 4/4/94
 ⇒ 0.1 M HNO_3 solution will be made up
 + acidified with freshly made up HNO_3
 solutions.

One liter of 0.1 M NaNO_3 solution is
 prepared by weighing 8.4995g NaNO_3
 and diluting to volume in a 1000 mL
 volumetric flask.

wt used: 8.5006g lot 7808 KDTE 4/4/94
 No PM 4/4/94

1615 PM 0.5 M HNO_3 solution prepared 1/14/92 (GC-05
 -179) was diluted 1/10 to make 100 mL of
 0.05 M HNO_3 in a volumetric flask.
 10 mL of the 0.05 M HNO_3 was taken by volumetric
 pipet + put in a 100 mL volumetric flask, then
 diluted to volume to yield a 0.005 M HNO_3 solution.

1640 PM Eight 125-mL PP bottles were labeled
 B-IVX*i*a (where *i* = 2.0...5.5). 100 mL of
 the 0.1 M NaNO_3 solution was taken by
 volumetric pipet and put in each bottle.

5/27/92 PM Expt B-IV continued

The meter was calibrated using 5 Fisher buffers
 (pH = 2.0 4.0 7.0 9.0 + 10.0) from the water bath.
 0752 PM The pH of solutions B-IVX*i* were recorded.

| solution | pH | temp °C | solu | pH | temp |
|----------|------|---------|----------------|-------|------|
| B-IVX2.0 | 1.91 | 20.4 | B-IVX6.0 | 6.38 | 20.6 |
| 2.5 | 2.37 | 20.5 | 6.5 | 6.42 | 20.6 |
| 3.0 | 2.41 | 20.5 | 7.0 | 6.83 | 20.6 |
| 3.5 | 2.86 | 20.4 | 7.5 | 7.41 | 20.6 |
| 4.0 | 3.32 | 20.5 | 8.0 | 7.93 | 20.7 |
| 4.5 | 3.81 | 20.5 | 8.5 | 8.45 | 20.6 |
| 5.0 | 4.39 | 20.5 | 9.0 | 8.92 | 20.6 |
| 5.5 | 5.46 | 20.5 | 9.5 | 9.23 | 20.6 |
| | | | pH buffer 10.0 | 10.01 | 20.7 |

0827 PM The pH of the B-IVX*i*a solutions made yesterday
 will be measured, after calibrating with
 pH buffers 4.0, 5.0, + 7.0 from the water bath.

| solution | pH | temp °C |
|-----------|------|---------|
| B-IVX2.0a | 5.56 | 20.7 |
| X2.5a | 5.59 | 20.7 |
| X3.0a | 5.56 | 21.0 |
| X3.5a | 5.58 | 20.8 |
| X4.0a | 5.59 | 20.8 |
| X4.5a | 5.59 | 20.8 |
| X5.0a | 5.60 | 20.8 |
| B-IVX5.5a | 5.60 | 20.8 |

0920 PM The amounts of HNO_3 needed to
 adjust the B-IVX*i*a solutions are
 as follows:

| solution | desired molarity NO_3^- | amount HNO_3 (~0.1M) |
|-----------|----------------------------------|-------------------------------|
| B-IVX2.0a | 1.2129×10^{-2} | 65 drops 0.5M HNO_3 |
| 2.5a | 3.8173×10^{-3} | 20 0.5 |
| 3.0a | 1.2053×10^{-3} | 6 0.5 |
| 3.5a | 3.8096×10^{-4} | 20 0.05 |
| 4.0a | 1.2035×10^{-4} | 6 0.05 |
| 4.5a | 3.7866×10^{-5} | 20 0.005 |
| 5.0a | 1.1335×10^{-5} | 6 0.005 |
| 5.5a | 1.5615×10^{-6} | 1 0.005 |

0950PM The B-IVXia solutions were covered with kumipies & put on the gyratory shaker.

1131PM The pH meter was calibrated using four Fisher pH buffers (2.0, 4.0, 5.0, + 7.0) from the water bath.

Solutions B-IVXia were measured for pH

| solution | pH | temp °C |
|-----------|------|---------|
| B-IVX2.0a | 2.09 | 21.8 |
| 2.5a | 2.62 | 21.7 |
| 3.0a | 3.09 | 21.7 |
| 3.5a | 3.63 | 21.8 |
| 4.0a | 4.20 | 21.7 |
| 4.5a | 4.67 | 21.7 |
| 5.0a | 5.09 | 21.7 |
| B-IVX5.5a | 5.45 | 21.8 |

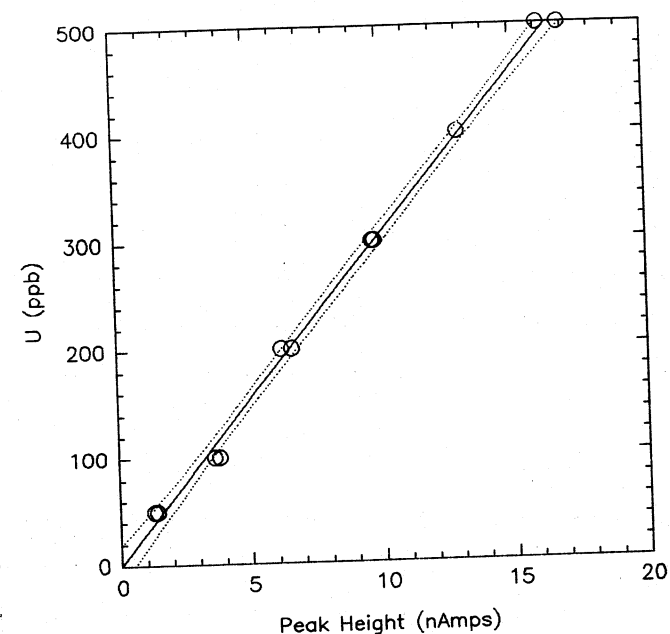
0400 TD

Continued analyzing the K2-1 solutions on the Polarograph using DPS with no deposition. The calibration was done with the standards prepared on 5/25/92.

CALIBRATION CURVE VALUES

| UDPS0527.SP5: Wed, 27-May-92 | | | |
|------------------------------|---------------|--------------|-----------|
| U (ppb) | Pk Ht (nAmps) | Regress-20rd | |
| 1 | 2 | 3 | |
| 1 | 50 | 1.4 | 0.11983 |
| 2 | 50 | 1.3 | 30.958 |
| 3 | 100 | 3.8 | -0.034212 |
| 4 | 100 | 3.6 | 0 |
| 5 | 200 | 6.6 | 0 |
| 6 | 200 | 6.2 | 0 |
| 7 | 300 | 9.8 | 0 |
| 8 | 300 | 9.7 | 0 |
| 9 | 400 | 13 | 0 |
| 10 | 400 | 13 | 0 |
| 11 | 500 | 16.9 | 0 |
| 12 | 500 | 16.1 | 0.9984 |

CALIBRATION CURVE



RESULTS OF U CONCENTRATION ANALYSIS

| K2-1_B1.SP5: Wed, 27-May-92 | | | | |
|-----------------------------|------------------|------------------|-------------------|-------|
| K2-1*B* 1 | Pk Ht1 (nA) 2 | Pk Ht2 (nA) 3 | U conc (ppb) 4 | |
| 1 | 1A | 17.4 | 16.6 | 517* |
| 2 | 1B | 16.8 | 17.5 | 521* |
| 3 | 2A | 15.6 | 15.3 | 472 |
| 4 | 2B | 15.7 | 15.6 | 476 |
| 5 | 3A | 8.2 | 8.6 | 258 |
| 6 | 3B | 8.7 | 8.3 | 261 |
| 7 | 300 PPB | 10.1 | 10.9 | (321) |
| 8 | 4A | 4.5 | 4.3 | 136 |
| 9 | 4B | 4 | 5.7 | 150 |
| 10 | 5A | 2.9 | 2.3 | 80.5 |
| 11 | 5B | 2.5 | 2.1 | 71.2 |
| 12 | IUA | 17 | 17.1 | 518* |
| 13 | IUB | 18.6 | 17.9 | 554* |
| 14 | IUC | 18.2 | 18.2 | 552* |
| 15 | 300 PPB | 10.2 | 11.4 | (331) |
| 16 | * MEANS | VALUE | OUT OF | RANGE |

1158 PM

Expt B-IV continued

The HNO_3 solution change seems to have improved how close the target pH values for solutions B-IV X_i can be approximated. Compare pH values for B-IV X_i (p 83) + those for B-IV X_{ca} (p 84), where $i \leq 5.5$.

→ The B-IV X_i solutions ($i \leq 5.5$) prepared 5/22/92 will be thrown out, and the B-IV X_{ca} solutions used in their place. The "a" on the label will no longer be used, as there will be no other set of solutions to confuse this set with.

28 MAY 1992 TD 0800

There have ^{are} been ^{TD 5/29/92} some problems in analyzing the low concentrations of U on the polarograph. Different parameters were tried with no change in our lack of ability to ^{TD 5/29/92} analyze these concentrations. A new set of standards, made with a new ^{0.25 M} Tartaric Acid Triethanolamine matrix will be made, and hopefully this will allow analysis to continue.

1530 TD

Prepared 500 mL 1.0M TT by dissolving 74.57 gm Triethanolamine and 75.045 gm Tartaric Acid in water and diluting to 500 mL in a volumetric flask.

WT. Tartaric Acid used 74.5725 gm

WT.

5/29/92

WT. Triethanolamine used 74.5725 gm

WT. Tartaric Acid used 75.0467 gm

This solution will be used to prepare new U standards.

5/29/92 PM

Expt B-IV continued

0828 PM The B-IV X_i solutions will be measured for pH, using the same 2.0, 4.0, 7.0, 9.0, + 10.0 pH buffers for calibration.

| solution | pH | temp °C |
|------------|------|---------|
| B-IV X 2.0 | 2.11 | 20.5 |
| X 2.5 | 2.66 | 20.5 |
| X 3.0 | 3.13 | 20.5 |
| X 3.5 | 3.69 | 20.5 |
| X 4.0 | 4.21 | 20.6 |
| X 4.5 | 4.66 | 20.6 |
| X 5.0 | 5.12 | 20.7 |
| X 5.5 | 5.49 | 20.7 |
| X 6.0 | 6.38 | 20.7 |
| X 6.5 | 6.38 | 20.8 |
| X 7.0 | 6.78 | 20.8 |
| X 7.5 | 7.34 | 20.8 |
| X 8.0 | 7.89 | 20.9 |
| X 8.5 | 8.45 | 20.9 |
| X 9.0 | 8.94 | 20.9 |
| B-IV X 9.5 | 9.30 | 20.9 |

29 MAY 1992 TO 0800

Prepared new U standards in the same manner as before (see pg 79). The 1.0 MTT prepared 5-28-92 was used in place of the other 1.0 M IT solution.

5/30/92 PM Expt B-IV cont.

10:55 PM Solutions B-IV X i will be measured for pH as before.

| solution | pH | temp °C |
|------------|------|-----------|
| B-IV X 2.0 | 2.11 | 20.8 |
| X 2.5 | 2.63 | 20.8 |
| X 3.0 | 3.10 | 20.9 |
| X 3.5 | 3.64 | 20.9 |
| X 4.0 | 4.19 | 20.8 |
| X 4.5 | 4.65 | 20.8 |
| X 5.0 | 5.48 | 5.09 20.8 |
| X 5.5 | 5.48 | 20.8 |
| X 6.0 | 6.42 | 20.9 |
| X 6.5 | 6.44 | 20.9 |
| X 7.0 | 6.84 | 20.8 |
| X 7.5 | 7.42 | 20.8 |
| X 8.0 | 7.94 | 20.8 |
| X 8.5 | 8.47 | 20.8 |
| X 9.0 | 8.93 | 20.8 |
| X 9.5 | 9.31 | 20.8 |

5/31/92 PM Expt B-IV cont.

Solutions B-IV X i will be remeasured for pH as before.

| 11:20 PM solution | pH | temp °C |
|-------------------|-------|---------|
| B-IV X 2.0 | 2.11 | 21.1 |
| 2.5 | 2.63 | 21.1 |
| 3.0 | 3.09 | 21.1 |
| 3.5 | 3.61 | 21.1 |
| 4.0 | 4.14 | 21.1 |
| 4.5 | 4.62 | 21.1 |
| 5.0 | 5.09 | 21.1 |
| 5.5 | 5.50 | 21.1 |
| 6.0 | 6.43 | 21.1 |
| 6.5 | 6.44 | 21.1 |
| 7.0 | 6.84 | 21.1 |
| 7.5 | 7.42 | 21.1 |
| 8.0 | 7.93 | 21.1 |
| 8.5 | 8.47 | 21.1 |
| 9.0 | 8.94 | 21.1 |
| 9.5 | 9.33 | 21.1 |
| pH 10.0 buffer | 10.01 | 21.1 |

12:03 PM If the pH readings have stabilized, 0.200g of clindamycin will be added to each B-IV X i solution (CDV X 10% 200 X UC X WA X HL X CPT X NaF)

| solution | wt added | solution | wt added |
|------------|----------|------------|----------|
| B-IV X 2.0 | .2007 | B-IV X 6.5 | .2001 |
| 2.5 | .2001 | 7.0 | .2001 |
| 3.0 | .2007 | 7.5 | .2002 |
| 3.5 | .2000 | 8.0 | .2008 |
| 4.0 | .2006 | 8.5 | .2005 |
| 4.5 | .2002 | 9.0 | .2008 |
| 5.0 | .2004 | 9.5 | .2005 |
| 5.5 | .2004 | | |
| 6.0 | .2002 | | |

12:50 PM The B-IV X i solutions were set back in the gyratory shaker.

6/1/92 PH Expt K2-1 continued

0845 PH 3 10-ml aliquots were taken from solution K2-1XU by Eppendorf max pipette after the last samples were taken from solutions K2-1XA + K2-1XB. The aliquots were put in 3 15 ml PP bottles labeled K2-1XEUKa (or b, c). 500 μ l of 1.05 M tartaric acid / triethanolamine solution (GC-05-252) were added to each bottle.

1330 TD

Prepared 1ppm-5ppm U stds by diluting the amounts of 100ppm U std (given below) and 5ml 1.0M TT (p. 86-87) to 100 ml in a volumetric flask with Ultrapure H₂O. The 100ppm U std was prepared by diluting 10 ml 1000ppm U (lot LO-10) to 100 ml in a volumetric flask with Ultrapure H₂O.

1 ppm \rightarrow 1ml 100ppm
 2 ppm \rightarrow 2ml 100ppm
 3 ppm \rightarrow 3ml 100ppm
 4 ppm \rightarrow 4ml 100ppm
 5 ppm \rightarrow 5ml 100ppm

1340 PH Expt A-II-2 continued

The A-II-2 solutions will be vacuum filtered through a 0.45 μ m membrane filter, with the solution repeatedly swirled in the sample bottle + quickly poured through the filter to remove as much clinoptilolite from the solution as possible. The retained clinoptilolite will be air dried on the filter paper, then transferred into labeled glass vials. The solution

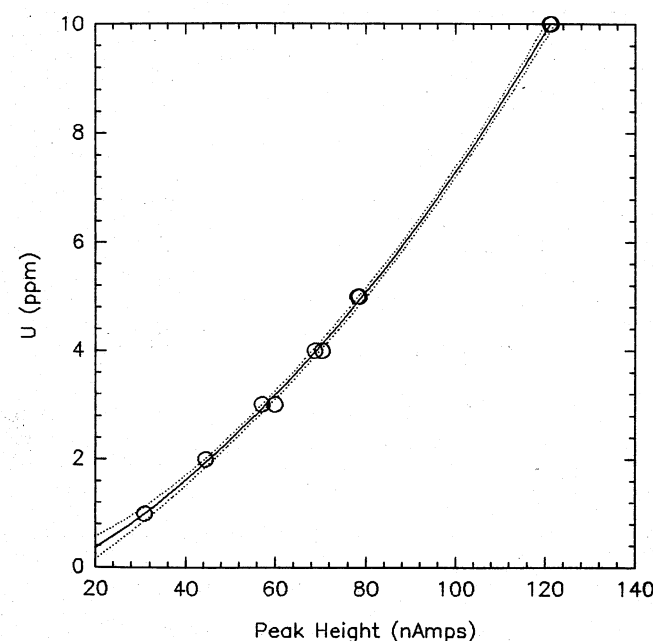
will be saved and analyzed for sodium content on the atomic absorption spectrophotometer later. The vials will be labeled A-II-2XpHi, where i is the target pH listed on the solution bottle.

6/2/92 PH 0814 The remaining A-II-2XpHi solutions will be filtered + saved as described above.

0800 TD

Analyzed the U concentrations of the A-II-2XpHiXUR3 solutions. DPS with 0s deposition time was used.

The Polarograph was calibrated with the standards prepared on p. 90. Plots can be found in Polarographic Analysis of Uranium, vol 10.13.



CALIBRATION
CURVE

UDPS0602.SP5: Tue, 2-Jun-92

| U (ppm) 1 | Pk Ht (nA) 2 | Regression 3 |
|--------------|-----------------|-----------------|
| 1 | 1 | 30.9 |
| 2 | 1 | 30.9 |
| 3 | 2 | 44.6 |
| 4 | 3 | 57.2 |
| 5 | 3 | 59.9 |
| 6 | 4 | 68.7 |
| 7 | 4 | 70.5 |
| 8 | 5 | 78.3 |
| 9 | 5 | 78.6 |
| 10 | 10 | 121.1 |
| 11 | 10 | 121.4 |
| 12 | | |

0.99963

RESULTS OF U ANALYSIS OF A-II-2 KPHIXUR3

| i | PK He(nA) | | U conc (ppm) |
|-------|-----------|------|--------------|
| IU 10 | 96 | 98.7 | 7.03 |
| IU 11 | 97.8 | 96.7 | 7.02 |
| IU 12 | 98.9 | 101 | 7.34 |
| 2.5 | 80 | 82.8 | 5.25 |
| 3 | 109 | 108 | 8.39 |
| 3.5 | 114 | 114 | 9.11 |
| 4 | 118 | 115 | 9.44 |
| 4.5 | 103 | 108 | 8.02 |
| 5 | 64.4 | 65 | 3.62 |
| 5.5 | 58.6 | 58.5 | 3.07 |
| 6 | 55.3 | 56.1 | 2.83 |
| 6.5 | 53.1 | 55.8 | 2.73 |
| 7 | 58.5 | 59.2 | 3.1 |
| 7.5 | 83.9 | 87 | 5.68 |
| 8 | 85.7 | 90.5 | 5.97 |
| 8.5 | 110 | 114 | 8.84 |
| 9 | 117 | 112 | 9.17 |
| 4 PPM | 66.7 | 66.1 | (3.77) |

3 June 1992 TDS 1000

Prepared new U standards according to table below. The 1000 ppm U standard is from Banco, lot # L0-10. The 1.0M TT was prepared 28 May 1992 (p. 86-87). All were diluted in 100 ml volumetric flasks. 5 ml measurements and 10 ml measurements were made with volumetric pipets. All others were made with 1 ml fixed volume Eppendorf pipets.

| | | |
|--------|---------------------------------|----------|
| 50 ppm | → 5 ml 1000 ppm U, 5 ml 1.0M TT | → 100 ml |
| 40 ppm | → 4 ml 1000 ppm U, " | " |
| 30 ppm | → 3 ml 1000 ppm U, " | " |
| 20 ppm | → 2 ml 1000 ppm U, " | " |
| 10 ppm | → 1 ml 1000 ppm U, " | " |
| 5 ppm | → 10 ml 50 ppm, " | " |
| 4 ppm | → 10 ml 40 ppm, " | " |
| 3 ppm | → 10 ml 30 ppm, " | " |
| 2 ppm | → 10 ml 20 ppm, " | " |
| 1 ppm | → 10 ml 10 ppm, " | " |

0728PK Expt A-II-2 continued
The A-II-2 solutions filtered previously (see p. 90-91) will be analyzed using

atomic absorption to determine the amount of Na present in the solutions. The solutions will be analyzed on a Perkin Elmer 3100 Atomic Absorption Spectrometer.

Na standards of 1 ppm and 0.05 ppm (50 ppb) were made by taking 2 ml of 100 ppm Na standard (NaCl in deionized water; lot # 94-11-07) Orion Application Solution) and diluting to volume in a 200 ml volumetric flask with ultra pure water to yield a 1 ppm Na standard and by taking 250 μ l of the 100 ppm Na standard and diluting to volume in a 500 ml volumetric flask with ultra pure water.

CsCl (lot # 896658A) was added to the standards, sample solutions & the blank (0.1% or more by weight) to control ionization.

AA parameters included

λ (nm) = 589.5

slit (nm) = 0.7

flame = air/acetylene

The machine was run according to instructions in the Running the 3100 Operating Instructions manual. The blank (nanopure water + ~0.1% CsCl) was run first.

Parameters entered included

lamp current = 8 mA

integration time = 3 s

replicates = 4

calibration = linear

AA technique = flame

standard = 1.000 ppm

Solutions A-II-2X_i were diluted 1/10 in volumetric flasks, poured into 15 mL PP beakers, and had 0.1% GCL added to each.

readings:

| | |
|------------------------|-----------|
| A-II-2X 2.5 (1/10 dil) | 8.52 ppm |
| 3.0 " | 6.451 ppm |
| 3.5 " | 3.977 |
| 4.0 " | 2.283 |
| 4.5 " | 2.307 |
| 5.0 " | 2.307 |
| 5.5 " | 1.574 |
| 6.0 " | 1.72 |
| 6.5 " | 1.442 |
| 7.0 " | 1.500 |
| 7.5 " | 1.915 |
| 8.0 " | 2.508 |
| 8.5 " | 4.979 |
| 9.0 " | 8.163 |
| 0.05 ppm std | 0.046 |

All readings were ± 0.003 (SD). All readings were above the 1 ppm standard used for calibration + out of the linear range, so the solutions were diluted differently.

readings:

| | |
|-------------------------|-----------|
| A-II-2X 2.5 (1/200 dil) | 2.699 ppm |
| 3.0 (1/200) | 0.994 |
| 3.5 (1/100 dil) | 0.482 |
| 4.0 " | 0.253 |
| 4.5 " | 0.244 |
| 5.0 " | 0.156 |
| 5.5 " | 0.161 |

readings:

| | |
|-------------------------|-------|
| A-II-2X 6.0 (1/100 dil) | 0.176 |
| 6.5 " | 0.139 |
| 7.0 " | 0.146 |
| 7.5 " | 0.191 |
| 8.0 " | 0.263 |
| 8.5 (1/200 dil) | 0.633 |
| 9.0 " | 1.999 |
| 0.05 ppm std | 0.050 |

The 1/200 dilutions for A-II-2X 2.5 and A-II-2X 9.0 were diluted an extra 1/10 and read on the AA.

| | |
|--------------------------|-------|
| A-II-2X 2.5 (1/2000 dil) | 0.317 |
| X 9.0 " | 0.208 |

ppm present in solutions A-II-2X_i:

| | | | |
|-------------|------|-------------|------|
| A-II-2X 2.5 | 634 | A-II-2X 6.0 | 17.6 |
| 3.0 | 994 | 6.5 | 13.9 |
| 3.5 | 482 | 7.0 | 14.6 |
| 4.0 | 25.3 | 7.5 | 19.1 |
| 4.5 | 24.4 | 8.0 | 26.3 |
| 5.0 | 15.6 | 8.5 | 63.3 |
| 5.5 | 16.1 | 9.0 | 416 |

NaHCO₃ was added to solutions A-II-2X 5.0 through A-II-2X 9.0 to adjust the pH previously (see GC-05-216); ppm Na added =

| | |
|-------------------------|--------------------------|
| 0.4 ppm for A-II-2X 5.0 | 49.2 ppm for A-II-2X 8.0 |
| 1.9 | 5.5 |
| 3.0 | 6.0 |
| 4.0 | 6.5 |
| 7.5 | 7.0 |
| 17.0 | 7.5 |

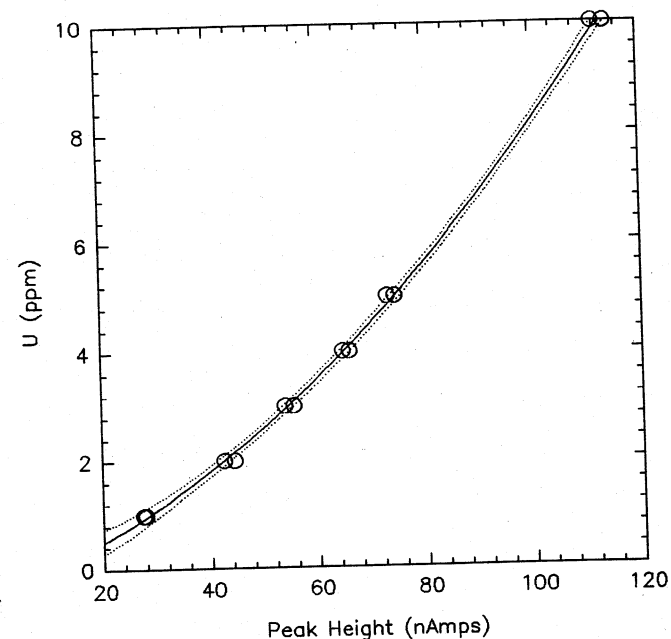
1200 D

Reanalyzed the U concentration in several of the A-II-2*PHI*UR3 solutions on the Polarograph. The polarograph was calibrated using the standards prepared earlier today. Plots can be found in Polarographic Analyses of Uranium Vol 13.

CALIBRATION

UDPS0603.SP5: Wed, 3-Jun-92

| U (ppm) 1 | Pk Ht (nA) 2 | Regression 3 |
|--------------|-----------------|-----------------|
| 1 | 27.5 | -0.38114 |
| 1 | 27.8 | 0.034196 |
| 2 | 42.5 | 0.00051213 |
| 2 | 44.4 | 0 |
| 3 | 53.8 | 0 |
| 3 | 55.5 | 0 |
| 4 | 64.6 | 0 |
| 4 | 65.9 | 0 |
| 5 | 72.9 | 0 |
| 5 | 74.4 | 0 |
| 10 | 111.8 | 0 |
| 10 | 114 | 0.99936 |



RESULTS OF U ANALYSIS

AII2R34.SP5: Thu, 4-Jun-92

| AII2*PHI*UR3 1 | Pk Ht (nA) 2 | Pk Ht (nA) 3 | U conc (ppm) 4 |
|-------------------|-----------------|-----------------|-------------------|
| 1 IU 10 | 90.2 | 91.6 | 6.9417 |
| 2 IU 12 | 94 | 97 | 7.5363 |
| 3 3 | 103 | 107 | 8.8326 |
| 4 4 | 116 | 116 | 10.449 |

6/5/92 PM Expt A-II-2 continued

Some of the A-II-2 *PHI* UR3 samples were re-analyzed on the polarograph.

0908 PM New 2, 3, 5, + 10 ppm U standards were prepared. A 100 ppm U standard was prepared by diluting 10 mL of 1000 ppm U standard (lot L0-10) to 100 mL in a volumetric flask using nanopure water.

2 mL of the 100 ppm std was diluted to 100 mL to yield a 2 ppm standard. The 2 mL was diluted with 5 mL 5.0 M tartaric acid / triethanolamine (GC-07-86, 87) and then to volume with ultrapure water.

In the same manner, 3, 5, + 10 mLs of the 100 ppm U std were taken by volumetric pipet + diluted in 100 mL volumetric flasks to yield 3, 5, + 10 ppm U standards.

Parameters used:

polarographic method: differential pulse stripping
 electrode # : 2
 deposition time : 0 s
 scan increments : 8 mV
 drop/step time : 0.5 s
 blank subtract : no
 purge time : 360 s
 drop size : med
 range : 0.1 to -0.5 V
 equil. time : 5 s

| standard | pk hgt 1 (nA) | pk hgt 2 (nA) | temp, °F |
|----------|---------------|---------------|----------|
| 2 ppm | 44.1 | 43.8 | 71.2 |
| 3 ppm | 54.5 | 55.2 | 71.5 |
| 5 ppm | 75.7 | 76.9 | 71.6 |
| 10 ppm | 115.5 | 114.6 | 71.5 |

| solution | pk hgt 1 | pk hgt 2 | ppm* | temp °F |
|-------------|----------|----------|------|---------|
| A-T-2X10X10 | 84.90 | 83.10 | 5.93 | 71.6 |
| XpH25XUR3 | 77.80 | 77.40 | 5.20 | 71.6 |
| XpH4.5X | 97.30 | 102.5 | 7.92 | 71.9 |
| XpH7.5X | 79.60 | 80.50 | 5.48 | 72.0 |
| 10 ppm Ustd | 111.7 | 115.0 | 9.80 | 72.0 |

* Second order regression line, calculated with Sigmaplot:

$$y = 0.00050x^2 + 0.03305x - 0.37166$$

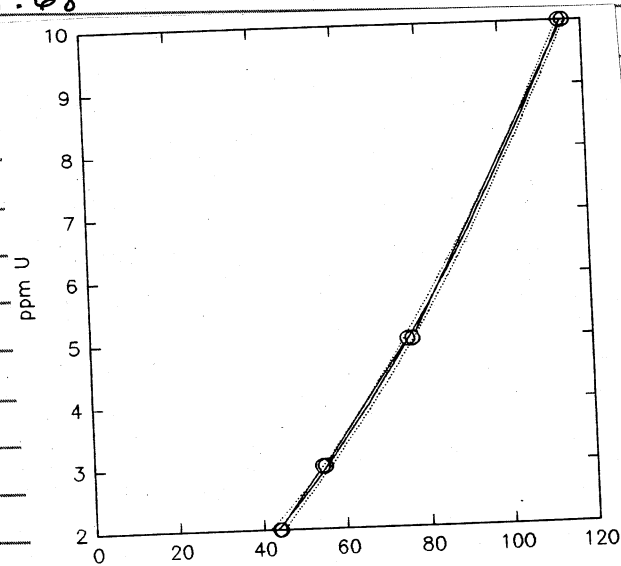
$$\text{corr. coeff} = 0.99978$$

Compare ppm calculated above to calculated ppm from 6/2/92 (GC-07-92):

| | |
|-------------|--------|
| A-T-2X10X10 | : 7.03 |
| XpH25XUR3 | : 5.25 |
| XpH4.5XUR3 | : 8.02 |
| XpH7.5XUR3 | : 5.68 |

Plots can be found in
XPL PA00 Vol 13
6/9/92

Calibration →
Curve



6/9/92 XPL 1052 Expt A-II cont

The polarograph was not giving satisfactory peak readings for low (< 100 ppb) U standards in II. 10, 30, 50, + 100 ppb U standards prepared 13/6/92 were run in Cell #2 using the following parameters:

Method: Differential pulse stripping

Depos. time: 60 s

Scan rate: 8 mV/.5 s

drop size: medium

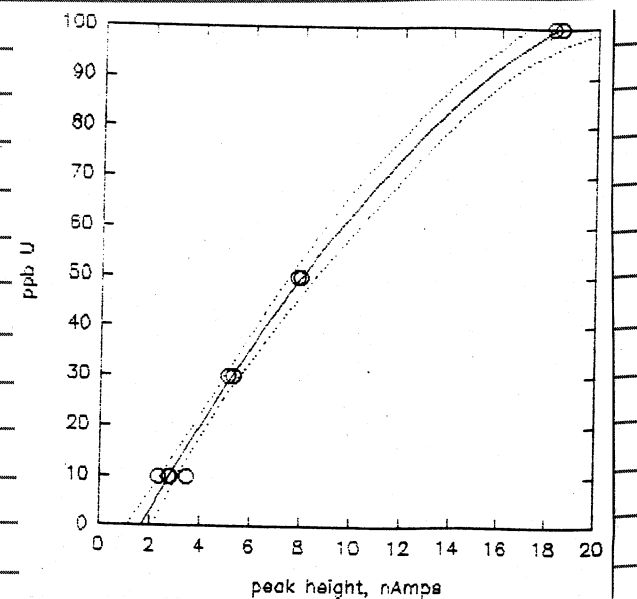
blank sub: no

scan range: 0.100 to -0.400 V

equil. time: 10 s

purge time: 360 s

| ppb | pk hgt |
|-----|--------|
| 10 | 2.771 |
| 10 | 3.461 |
| 10 | 2.786 |
| 10 | 2.345 |
| 30 | 5.29 |
| 30 | 5.11 |
| 50 | 7.98 |
| 50 | 7.83 |
| 100 | 18.53 |
| 100 | 18.18 |



Second order regression line, calculated with Sigmaplot:

$$y = -0.18276x^2 + 9.6503x - 15.494$$

$$\text{corr. coeff} = 0.99774$$

Compare to analysis run 5/6/92 (GC-07-56)
The 5 ppb standard was not run due to noise trouble.

6/10/92 PM 1424 Expt B-IV continued

The pH meter was calibrated with the Fisher pH buffers 2.0, 4.0, 7.0, 9.0 + 10.0 from the bath. The B-IV $\times i$ solutions were then read for pH.

| solution | pH | temp, °C |
|-------------------|------|----------|
| B-IV $\times 2.0$ | 2.11 | 23.3 |
| $\times 2.5$ | 2.65 | 23.3 |
| $\times 3.0$ | 3.15 | 23.4 |
| $\times 3.5$ | 3.72 | 23.4 |
| $\times 4.0$ | 4.38 | 23.6 |
| $\times 4.5$ | 4.94 | 23.6 |
| $\times 5.0$ | 5.42 | 23.5 |
| $\times 5.5$ | 5.68 | 23.6 |
| $\times 6.0$ | 6.37 | 23.5 |
| $\times 6.5$ | 6.38 | 23.7 |
| $\times 7.0$ | 6.81 | 23.8 |
| $\times 7.5$ | 7.40 | 23.8 |
| $\times 8.0$ | 7.90 | 23.9 |
| $\times 8.5$ | 8.43 | 23.9 |
| $\times 9.0$ | 8.92 | 23.9 |
| $\times 9.5$ | 9.39 | 23.9 |

1528 PM pH buff 10.0 10.00 24.0

6/11/92 PM Expt B-IV cont

Expt B-IV started on 5/22/92 (GC-07-75) 6/11/92 will be discontinued. The NaNO_3 added to in the stock solution interferes with possible H^+ exchange, which is what the experiment is supposed to check.

B-IV will be restarted, but without NaNO_3 present in the stock solution.

6/11/92 0930 TD

Experiment B-V

Control experiment to determine the pH change of ultrapure water after addition of clinoptilolite.

- Initial $\Sigma U = 0$
- Initial pH = 2.0 - 9.0, every 0.5 pH Units
- volume = 100 ml Ultrapure H_2O
- $p\text{CO}_2 = 10^{-3.48}$ bars
- wt. zeolite = 0.200 g
- pH adjusted with HNO_3 or NaHCO_3 according to EQ 3 calculations

B-V STEP 1

100 ml aliquots of Ultrapure H_2O were taken by volumetric pipet and placed in clean, dry 125 ml PP bottles. The bottles were labbed B-V $\times i$, where $i = 2.0, 2.5, \dots, 9.5$.

B-V STEP 2

The initial pH of the solutions was measured using the Orion 920A meter, an ^{TD 6/11/92} Automatic Temperature Compensation Probe, and a Ross combination glass pH electrode.

B-V STEP 3

^{TD 6/11/92} The pH of the solutions was adjusted according to the calculation using the adjustments found on GC-07-84 for $i = 2.0 - 5.5$ and GC-07-77 for solutions 6.0 - 9.5. The bottles were covered with a Kim wipe and placed on a gyratory shaker. The pH will be monitored until it reaches equilibrium with atmospheric $\text{CO}_2(\text{g})$. This should take ~10 days.

B-V STEP 4

0.200 g clinoptilolite was added to each solution (after reaching equilibrium). The pH

will be measured periodically ^{6/11/92} until to determine when equilibrium is reached. This should take ~2 weeks.

0945 Experiment B-V - pH measurements (initial)

| NAME | pH(initial)/T(°C) | adjustment |
|------|-------------------|---|
| 2.0 | 5.57/22.6 | 65 drops 0.5M HNO ₃ |
| 2.5 | 5.75/22.5 | 20 drops " |
| 3.0 | 5.59/22.4 | 6 drops " |
| 3.5 | 5.60/22.4 | 20 drops 0.05M HNO ₃ |
| 4.0 | 5.72/22.4 | 6 drops " |
| 4.5 | 5.70/22.5 | 20 drops 0.005M HNO ₃ |
| 5.0 | 5.68/22.6 | 6 drops " |
| 5.5 | 5.91/22.6 | 1 drop " |
| 6.0 | 5.83/22.6 | 10 ^{6/11/92} 0.00019g .00013g NaHCO ₃ |
| 6.5 | 5.66/22.6 | .00018g |
| 7.0 | 5.62/22.6 | .00061g |
| 7.5 | 5.67/22.6 | .00205g |
| 8.0 | 5.56/22.7 | .00673g |
| 8.5 | 5.68/22.5 | .02139g |
| 9.0 | 5.71/22.6 | .07348g |
| 9.5 | 5.60/22.6 | .33571g |

The HNO₃ solutions were prepared on 5/22/92 (see GC-07-76)
The NaHCO₃ comes ~~from~~ ^{6/11/92} lot # 897186A

12 June 1992 1225 TD B-V Cont
Measured the pH of all B-V solutions

| NAME | pH/T(°C) | NAME | pH/T(°C) |
|------|-----------|------|-----------|
| 2.0 | 2.13/22.6 | 6.0 | 6.38/21.9 |
| 2.5 | 2.60/22.6 | 6.5 | 6.70/21.8 |
| 3.0 | 3.03/22.3 | 7.0 | 7.20/22.0 |
| 3.5 | 3.58/22.4 | 7.5 | 7.60/22.4 |
| 4.0 | 4.15/22.0 | 8.0 | 7.85/22.2 |
| 4.5 | 4.63/22.6 | 8.5 | 8.60/22.5 |
| 5.0 | 5.11/22.1 | 9.0 | 8.47/21.9 |
| 5.5 | 5.83/22.5 | 9.5 | 8.94/21.9 |

0950 PM

Expts K1-1 & K1-2 cont.

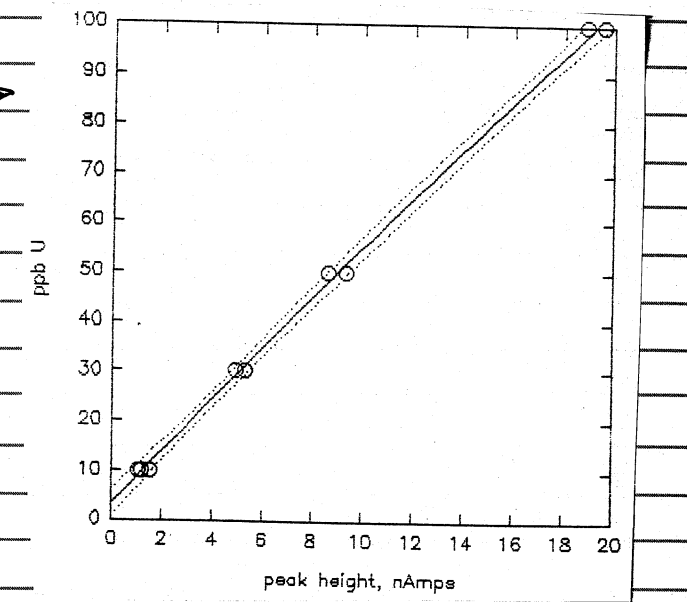
The polarograph was standardized using 10, 30, 50 + 100 ppb U²³⁵ standard solutions with prepared 5/6/92. Parameters used were those on GC-07-99.

| ppb U | pk hgt, nAmps run 1 | pk hgt, nAmps run 2 (run 1) |
|-------|---------------------|-----------------------------|
| 10 | 1.513 | 1.07 |
| 30 | 4.92 | 5.28 |
| 50 | 8.62 | 9.35 |
| 100 | 19.62 | 18.87 |

2nd order regression curve, calculated on →
Sigmaplot:

$$y = -0.01668x^2 + 5.3382x + 3.3392$$

corr. coeff: 0.99913



| solution | pk hgt 1 (nAmps) | pk hgt 2 | calc. ppb U |
|----------------------------|------------------|----------|-------------|
| K1-2XAX 11a | 2.15 | 2.14 | 14.71 |
| 11b | 8.01 | 7.55 | 43.86 |
| K1-2XBX 12a | 27.53 | 26.43 | 135.21 |
| 50ppb U ^{6/11/92} | 9.82 | 8.60 | 95.109 |
| 12b | 1.51 | 1.37 | 11.01 |
| K1-2XAX 12a | 2.14 | 1.88 | 14.01 |
| 12b | no peak | no peak | |
| K1-1XAX 14a | 1.41 | 1.35 | 10.66 |
| 14b | 1.24 | 1.26 | 9.96 |
| BX 14a | 1.28 | 1.24 | 10.04 |
| BX 14b | 1.07 | 1.17 | 9.29 |
| 500nb U std | 9.30 | 8.79 | 50.26 |

All plots from 6/12/92 can be found in PAOU Vol 13.

15 JUNE 1992 845 TD

EXPERIMENT B-V; pH measurements (initial)

| NAME | pH/T(°C) |
|---------|----------------------|
| B-V#2.0 | 2.13/22.2°C |
| 2.5 | 2.57/22.2°C (TD) |
| 3.0 | 3.13/22.0 |
| 3.5 | 3.54/22.0 |
| 4.0 | 4.17/22.0 |
| 4.5 | 4.59/22.1 |
| 5.0 | 5.13/22.0 |
| 5.5 | 5.80/22.0 |
| 6.0 | 6.73/21.8 |
| 6.5 | 6.73/21.7 |
| 7.0 | 7.17/21.9 |
| 7.5 | 7.34/22.1 |
| 8.0 | 7.90/21.9 |
| 8.5 | 9.11/21.8 |
| 9.0 | 8.57/22.2 TD 6/15/92 |
| 9.5 | 9.26/21.8 |

0815 PM Glove-box check

The glovebox was checked for leaks using Snoop leak detector + then Freon and a halogen leak detector. One of the glovebox gloves had two small slits in it, + was replaced. The box was rechecked for leaks and none were detected.

1428 PM A 1×10^{-3} M solution of KCl was made in the same manner described on GC-07-73.

WT KCl used: 0.0748g lot # 885967

100 mL of ultrapure water was purged with N_2 gas for rinsing use in the glove box. The Orion 290 pH meter + a Ross glass electrode were calibrated using fresh 4.0 + 7.0 pH buffers (lot # 910355-24 and 910943-24).
 H_2O initial pH/temp 5.94 / 21.9 °C
 final 7.50 / 21.6 °C
 Δ time for purging 56 minutes

The water was capped + transferred into the glove box.

6/16/92 PM 0805 Glovebox check cont.

The pH meter was recalibrated as before. The 1×10^{-3} M KCl solution was measured for pH while it was purged with N_2 + was stirred. Solution volume was ~ 80 mL.

| | | time |
|--------------|----------------|-----------|
| starting pH: | 5.15 @ 19.8 °C | 0902 |
| w purge | 5.20 | 0905 |
| | 5.25 | 0910 |
| | 5.26 | 19.9 0917 |
| | " | 0922 |
| | 5.25 | 20.0 0927 |

0927 PM Solution volume was decreased to ~ 50 mL, + the N_2 gas was turned up.

| | | |
|------|------|------|
| 5.24 | 20.0 | 0936 |
| 5.23 | 20.1 | 0946 |
| 5.22 | 20.2 | 0952 |

The electrode fill solution was changed the meter/electrode recalibrated, + an ultra pure water aliquot (~ 80 mL) purged.

| | | | |
|--------------|------|------|------|
| starting pH: | 5.62 | 20.7 | 1053 |
| | 5.70 | " | 1056 |
| | 5.78 | " | 1058 |

| pH | temp | time |
|------|------|------|
| 5.89 | 20.7 | 1100 |
| 6.01 | 20.8 | 1103 |
| 6.13 | " | 1107 |
| 6.21 | " | 1110 |
| 6.28 | 20.9 | 1115 |
| 6.34 | 20.9 | 1118 |
| 6.43 | 21.1 | 1147 |
| 6.36 | 21.3 | 1240 |

N₂ gas was turned up

| | | |
|------|------|------|
| 6.49 | 21.4 | 1256 |
| 6.48 | 21.4 | 1304 |
| 6.47 | " | 1309 |

pH buffer 4.0; pH = 4.04 / 21.6°C @ 1324

The water was capped + transferred into the glovebox

1300PM The meter/glass electrode inside the glovebox was calibrated using Fisher pH 4.0 + 7.0 buffers (same lots as used outside) put in the box yesterday.

| Water pH | temp | time |
|----------|------|------|
| 6.10 | 21.3 | 1326 |

17 JUNE 1992 10 B-V (cont)

| TD 6/17/92 pH NAME | pH / T(°C) | NAME | pH / T(°C) |
|-----------------------|-------------|------|-------------|
| B-V* 2.0 | 2.01 / 22.5 | 2.0 | 6.41 / 22.4 |
| 2.5 | 2.40 / 22.4 | 6.5 | 6.72 / 22.5 |
| 3.0 | 2.99 / 22.5 | 7.0 | 6.83 / 22.5 |
| 3.5 | 3.52 / 22.5 | 7.5 | 7.37 / 22.6 |
| 4.0 | 4.08 / 22.5 | 8.0 | 7.80 / 22.6 |
| 4.5 | 4.48 / 22.6 | 8.5 | 8.50 / 22.5 |
| 5.0 | 5.08 / 22.5 | 9.0 | 9.06 / 22.4 |
| 5.5 | 5.84 / 22.6 | 9.5 | 9.34 / 22.4 |

Measurements made with Triode electrode

18 JUNE 1992 1100 10 EXP B-V (CONT)
pH MEASUREMENTS (CONT)

| NAME) TD 6/19/92 SAMPLE NAME | pH / T(°C) |
|---------------------------------|-------------|
| B-V* 2.0 | 2.11 / 22.7 |
| 2.5 | 2.56 / 22.7 |
| 3.0 | 3.18 / 22.7 |
| 3.5 | 3.64 / 22.7 |
| 4.0 | 4.16 / 22.7 |
| 4.5 | 4.61 / 22.7 |
| 5.0 | 5.10 / 22.8 |
| 5.5 | 5.76 / 22.8 |
| 6.0 | 6.02 / 22.8 |
| 6.5 | 6.46 / 22.8 |
| 7.0 | 6.92 / 22.8 |
| 7.5 | 7.38 / 22.8 |
| 8.0 | 7.80 / 22.8 |
| 8.5 | 8.52 / 22.8 |
| 9.0 | 9.02 / 22.8 |
| 9.5 | 9.39 / 22.8 |

The ROSS COMBINATION GLASS pH electrode with fresh filling solution was used for the measurements.

0900PM Tartaric Acid / Triethanolamine Solution
Check

The 1.0 M TT from 5/18/92, the 1.0 M TT from 5/28/92 + the 1.05 M TT from 2/19/92 were used to make low concentration U standard solutions, which were run on the polarograph using the parameters listed on GC-07-99.

First, a 100 ppm U std solution was made by taking 1 ml of 1000 ppm U standard solution (lot # 40-10) by Eppendorf micropipet and

diluting to volume in a 100 mL volumetric flask with ultra pure water.

Final std solution concentration:
100 ppm

47 ppb U solutions were made by taking 500 μ l of the given TI solution, 50 μ l of the 10 ppm U standard solution, and 10 mL of ultra pure water put directly in the sample cup.

Results: $\frac{5}{18}$ matrix 1.385 / 1.054 uAmp pk hgt
 $\frac{5}{28}$ matrix 1.127 / 1.160 " "
 $\frac{2}{19}$ matrix 7.640 / 7.470 " "

The 1.05 M $\frac{2}{19}$ TI was adjusted down to a final solution concentration of 0.0475 M (to match the solution M of the $\frac{5}{18}$ + $\frac{5}{28}$ matrices).

A 19.1 ppb U solution was made at this concentration by taking 475 μ l of the given 1.05 M TI (by taking 200 μ l, 200 μ l, + 75 μ l by Eppendorf pipet), 20 μ l of the 10 ppm U soln, + 10 mL of the ultra pure water put directly into the sample cup.

A 19 ppb U solution using the $\frac{5}{18}$ TI matrix was made by taking 500 μ l of the TI, 20 μ l of the 10 ppm U soln, + 10 mL of the ultra pure water.

Results: $\frac{2}{19}$ matrix 3.015 / 3.044 uAmp pk hgt
 $\frac{5}{18}$ matrix no peak

Plots can be found in PAOU vol 13.

19 JUNE 1992 1000 TD

EXP. BV (CONT.)

CONTINUED MONITORING THE pH OF SOLUTIONS

| SOLUTION NAME | pH/T(°C) |
|---------------|-----------|
| 2.0 | 2.11/22.4 |
| 2.5 | 2.56/22.4 |
| 3.0 | 3.15/22.4 |
| 3.5 | 3.58/22.4 |
| 4.0 | 4.15/22.4 |
| 4.5 | 4.60/22.4 |
| 5.0 | 5.11/22.4 |
| 5.5 | 5.74/22.5 |
| 6.0 | 5.91/22.5 |
| 6.5 | 6.49/22.6 |
| 7.0 | 6.98/22.6 |
| 7.5 | 7.34/22.6 |
| 8.0 | 7.78/22.6 |
| 8.5 | 8.50/22.6 |
| 9.0 | 9.03/22.6 |
| 9.5 | 9.44/22.7 |

0753 PM

Expt K2-1 continued

New U standards were prepared using the 1.05 M TI solution from 2/19/92 (GC-05-252).

First, a 100 ppm U solution was made by taking 10 mL of 1000 ppm U standard solution (lot # 40-10) and diluting to volume in a 100 mL volumetric flask with ultra pure water.

A 1 ppm U solution was made by diluting 2 mL of the 100 ppm U solution to volume in a 200 mL volumetric flask with ultra pure water.

Next, the following concentrations of standard solutions were made up by adding the following amounts of the 1 ppm U standard solution + 5 mL of 1.05 MTT to a 100 mL volumetric flask and brought to volume with ultrapure water.

| | |
|-----------|-------------------------|
| 500 ppb U | 50 mL 1 ppm U std |
| 400 | 40 |
| 250 | 25 |
| 100 | 10 |
| 50 | 5 |
| 30 | 3 |
| 10 | 1 |
| 5 ppb U | 500 μ L 1 ppm U std |

The 5, 10, 30, 50, & 100 ppb U std solutions were used to calibrate the polarograph using the parameters listed on GC-07-99.

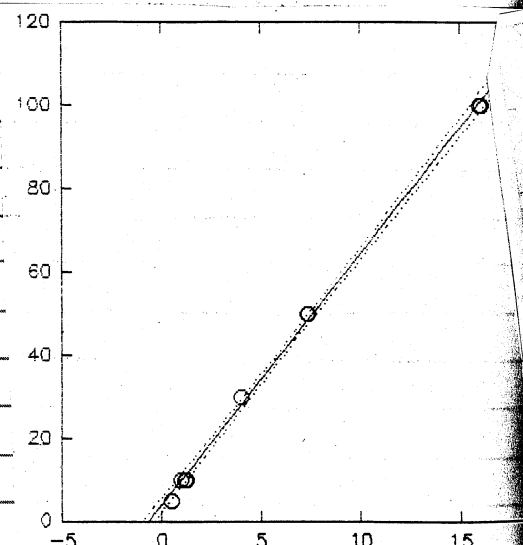
| ppb U std | pk Hgt, nAmps |
|-----------|---------------|
| 5.0 | 0.53 |
| 10.0 | 1.23 |
| 10.0 | 1.00 |
| 10.0 | 1.199 |
| 30.0 | 4.013 |
| 30.0 | 3.981 |
| 50.0 | 7.36 |
| 50.0 | 7.39 |
| 100.0 | 15.94 |
| 100.0 | 16.08 |

1st order regression plot \rightarrow

$$y = 6.0641x + 3.8859$$

$$\text{corr. coeff} = 0.9991$$

pk Hgt, nAmps



| Sample | pk Hgt 1 | pk Hgt 2 | calc. ppb | temp °F |
|--------------------|---------------|----------|-----------|---------|
| 1042 PM K2-1 KBK6a | 2.980 | 2.929 | 17.56 | 73.2 |
| *K6b | 2.843 | 2.848 | 17.13 | 73.4 |
| *7a | 1.029 | 1.111 | 10.23 | 73.5 |
| *7b | 0.899 | 0.809 | 9.39 | 73.6 |
| *8a | peak obscured | | | |
| *8b | 0.847 | 0.696 | 9.07 | 73.9 |

1245 PM The polarograph was recalibrated, using the same standard solutions.

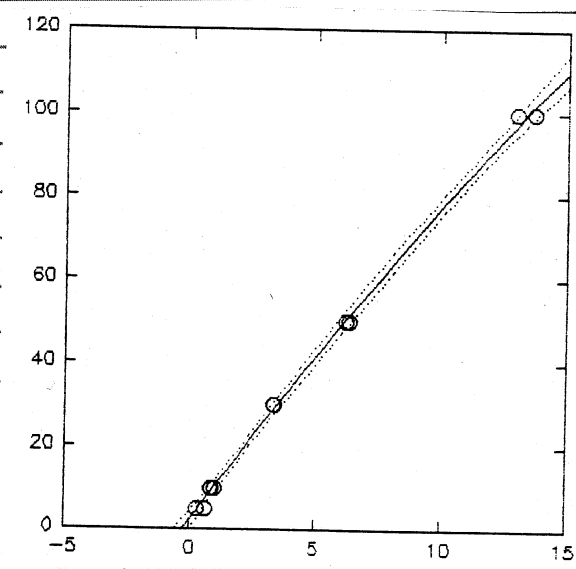
The parameters used were the same EXCEPT that the ~~scanning~~ ^{PKC 6/19/92} initial E was changed to 0.000V.

| ppb U std | pk Hgt, nAmps |
|-----------|---------------|
| 5.0 | 0.633 |
| 5.0 | 0.304 |
| 10.0 | 0.846 |
| 10.0 | 1.010 |
| 10.0 | 0.852 |
| 30.0 | 3.359 |
| 30.0 | 3.323 |
| 50.0 | 6.340 |
| 50.0 | 6.230 |
| 100.0 | 13.69 |
| 100.0 | 12.98 |

2nd order regression plot \rightarrow

$$y = -0.0652x^2 + 8.1805x + 2.2296 \quad \text{pk Hgt, nAmps}$$

$$\text{corr. coeff} = 0.9991$$



| sample | pk hgt1 | pk hgt2 | calc ppb | temp °F |
|------------------|---------|---------|----------|---------|
| K2-1A*6a | 4.220 | 4.120 | 35.21 | 75.3 |
| *6b | 4.340 | 4.410 | 36.77 | |
| *7a | 0.988 | 0.917 | 9.96 | 75.4 |
| *7b | 1.098 | 1.137 | 11.29 | 75.5 |
| *8a | 1.252 | 1.057 | 11.59 | 75.6 |
| *8b | 1.068 | 1.127 | 11.13 | 75.5 |
| *9b | 1.222 | 1.026 | 11.54 | |
| 30 ppb | 3.395 | 3.164 | 28.36 | 76.4 |
| 1544 PM K2-1B*9a | 0.574 | 0.455 | 6.42 | 76.8 |

All plots can be found in PoUA Vol. 13

6/22/92 PM Expt K2-1 continued

0814 PM The standards made up 6/19/92 (6C-07-109, 110) were used to calibrate the polarograph using the parameters

| | | |
|---------------------------------|------------------------------|--------|
| Method | differential pulse stripping | |
| cell # | 2 | |
| range | 0.0 to -0.4 V | |
| scan rate | 8 mV/5.5 | |
| deposit time | 60 s | |
| drop size | medium | |
| blank sub. | no | |
| purge time (N ₂ gas) | 360 s | 4/4/94 |
| equilibration time | 10 s | |

The 5 ppb U standards did not show peaks.

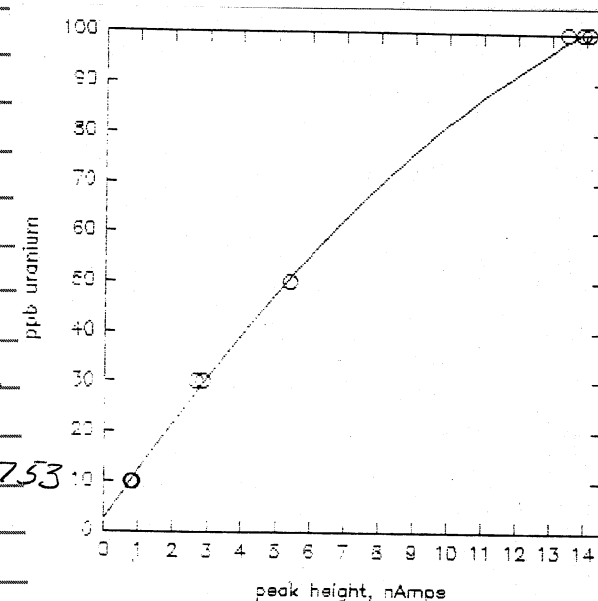
| ppb U std | pk Hgt, nAmps |
|-----------|---------------|
| 10.0 | 0.767 |
| | 0.810 |
| | 0.843 |
| 30.0 | 2.849 |
| | 2.648 |

| ppb U std | pk Hgt |
|-----------|--------|
| 50.0 | 5.420 |
| | 5.420 |
| 100.0 | 13.91 |
| | 13.45 |
| | 14.08 |

2nd order calibration curve:

$$y = -2.2489x^2 + 10.168x + 2.3753$$

corr. coeff = 0.9996



| sample | pk Hgt1 | pk Hgt2 | calc. ppb |
|----------|---------|---------|-----------|
| K2-1B*9b | 1.041 | 0.633 | 10.73 |
| A*9a | 1.073 | 0.538 | 12.37 |
| A*10b | 1.443 | 1.215 | 15.49 |
| A*10a | 0.945 | 0.785 | 11.00 |
| B*10b | 0.771 | 0.918 | 10.80 |
| B*10a | 0.737 | 0.917 | 10.63 |
| B*11a | 1.042 | | |

1121 PM 50 ppb U std 5.520 5.200 50.41

1310 PM The electrode was recalibrated as before. Again, the 5 ppb didn't register a peak.

| ppb U std | pk Hgt, nAmps |
|-----------|---------------|
| 10.0 | 0.877 |
| | 0.611 |
| | 0.871 |
| 30.0 | 3.313 |
| | 3.425 |
| 50.0 | 6.460 |
| | 6.650 |
| 100.0 | 14.63 |
| | 14.06 |

6/22/92 0830 TD EXP B-V (cont)

Remeasured pH of B-V solutions.

| SOLUTION NAME | pH/T(°C) |
|---------------|--------------------------------------|
| B-V * pH 2.0 | 2.11/21.8 |
| 2.5 | 2.56/21.4 |
| 3.0 | 3.16/21.5 |
| 3.5 | 3.59/21.6 |
| 4.0 | 4.15/21.6 |
| 4.5 | 4.60/21.7 |
| 5.0 | 5.13/21.7 |
| 5.5 | 5.76/21.7 |
| 6.0 | 6.15/21.8 |
| 6.5 | 6.50 ^{TD 4/21/92} 6.70/21.8 |
| 7.0 | 6.90/21.8 |
| 7.5 | 7.34/21.9 |
| 8.0 | 7.84/21.9 |
| 8.5 | 8.48/21.9 |
| 9.0 | 9.03/21.9 |
| 9.5 | 9.45/21.9 |

1015 TD EXP. BV (cont)

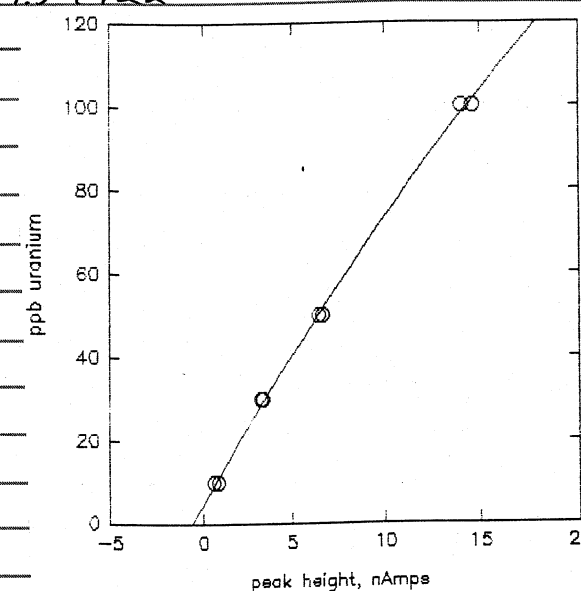
Since the pH of the solutions has stabilized, ~2.00 gm CDV * 100/200 * UC * WA * HL * CDT * NaF was added to each solution. The solutions were placed on a gyratory shaker for approximately two weeks to reach equilibrium. Their pH was again measured at this time.

| NAME | WT CDV ADDED (gm) | NAME | WT CDV ADDED (gm) |
|------|-------------------|------|-------------------|
| 2.0 | .1999 | 6.0 | .1997 |
| 2.5 | .1994 | 6.5 | .1992 |
| 3.0 | .2003 | 7.0 | .2008 |
| 3.5 | .2009 | 7.5 | .1998 |
| 4.0 | .2002 | 8.0 | .2001 |
| 4.5 | .2001 | 8.5 | .2007 |
| 5.0 | .1994 | 9.0 | .2007 |
| 5.5 | .2000 | 9.5 | .2000 |

1420 PM Expt K2-1 continued

calibration curve (2nd order):

$$y = -7.05295x^2 + 7.40148x + 4.54922$$



| sample | pk list 1 | pk list 2 | calc ppb | temp °F |
|-------------------|-----------|-----------|----------|---------|
| K2-1 * AK11a | 1.126 | 1.302 | 13.46 | 74.7 |
| B*11b | 0.678 | 0.670 | 9.51 | 74.8 |
| AK11b | 1.197 | 1.155 | 13.18 | |
| B*12b | 0.949 | 1.297 | 12.79 | 75.2 |
| AK12b | 1.121 | 1.296 | 13.42 | |
| AK12a | 1.353 | 1.334 | 14.54 | 75.5 |
| B*12a | 1.145 | 1.115 | 12.85 | |
| 1613 PM 50ppb std | 6.320 | 6.340 | 49.28 | 75.9 |

All plots can be found in PAU vol. 14.

4/23/92 PM Expt K2-1 cont.

0914 PM The 5.0 10.0 30.0 50.0 + 100.0 ppb U standards made up 6/19/92 (GC-07-109, 110) were used to calibrate the polarograph with the parameters listed in GC-07-112.

| ppb Ustd | pk list, nAmps |
|----------|----------------|
| 5.0 | 0.624 |
| | 0.427 |
| 10.0 | 0.749 |
| | 0.682 |
| 30.0 | 3.033 |
| | 2.974 |
| 50.0 | 6.170 |
| | 6.010 |
| 100.0 | 14.22 |
| | 13.92 |

6/23/92

10 Second order calibration curve:

$$y = -0.13884x^2 + 8.86955x + 2.51212$$

$$\text{corr coeff} = .9988$$

| solution | pk Hgt 1 | pk Hgt 2 | calc ppb U | temp °F |
|--------------------|----------|----------|------------|---------|
| K2-1KAX13b | 1.193 | 1.036 | 12.23 | 73.0 |
| *13a | 1.463 | 1.164 | 13.92 | 73.1 |
| B*13a | 1.558 | 1.118 | 14.13 | |
| *13b | 0.811 | — | 9.61 | 73.6 |
| 1144 PM 50ppb Ustd | 6.710 | 6.710 | 55.78 | 74.0 |

1310 PM The polarograph was recalibrated using the 100 250 400 + 500 ppb U standards made 6/19/92 and the same parameters.

| ppb Ustd | pk list, nAmps |
|----------|----------------|
| 100 | 15.08 |
| | 13.17 |
| | 13.44 |
| 250 | 32.15 |
| | 32.20 |
| 400 | 40.23 |
| | 40.28 |
| 500 | 45.30 |
| | 45.60 |

6/23/92 TD 1030 EXPT B-IV (cont)

Measured the Na⁺ concentration of the B-IV solutions using the 920A ORION meter and the Na⁺ Ion SELECTIVE ELECTRODE. Samples were prepared by taking 1 ml aliquots from each of the B-IV solutions and diluting it to 10 ml with Ultrapure water in a volumetric flask. 1 ml of the Na⁺ IONIC STRENGTH ADJUSTER was added to each ^{10 ml} sample. 100, 500, and 1000 ppm Na⁺ standards were used for calibration.

| NAME | Na ⁺ CONC. MEASURED (ppm) | Na ⁺ CONC. CORRECTED (ppm) | ADJUSTMENT (ppm) |
|------|--------------------------------------|---------------------------------------|------------------|
| 2.0 | 225 | 2250 | NA |
| 2.5 | 224 | 2240 | NA |
| 3.0 | 224 | 2240 | NA |
| 3.5 | 227 | 2270 | NA |
| 4.0 | 225 | 2250 | NA |
| 4.5 | 224 | 2240 | NA |
| 5.0 | 223 | 2230 | NA |
| 5.5 | 223 | 2230 | NA |
| 6.0 | 224 | 2240 | TD 1/142.9 0.24 |
| 6.5 | 225 | 2250 | 1.8 0.49 |
| 7.0 | 226 | 2260 | 5.6 1.53 |
| 7.5 | 226 | 2260 | 24.1 5.77 |
| 8.0 | 225 | 2250 | 62.4 17.2 |
| 8.5 | 227 | 2270 | 212.8 58.2 |
| 9.0 | 239 | 2390 | 233.3 201 |
| 9.5 | 312 | 3120 | 335.8 919 |

The adjustment is from the addition of NaHCO₃ to adjust the pH.

1420 PM Expt K2-1 cont

2nd order calibration curve:

$$y = 0.32879x^2 - 6.696x + 129.1$$

$$\text{corr. coeff: } 0.99951$$

| Sample | pk list 1 | pk list 2 | calc. ppb U | temp °F |
|-------------|-----------|-----------|-------------|---------|
| Y2-1XFXa | 42.20 | 42.60 | 436.3 | 76.5 |
| Kb | 39.16 | 39.10 | 370.5 | |
| Kc | 42.60 | 43.20 | 447.0 | 76.8 |
| 400ppb Ustd | 40.81 | 41.10 | 406.4 | 77.0 |

26 JUNE 1992 TD 0930

EXP B-V (CONT)

MEASURED THE pH OF ALL B-V SOLNS. THESE RESULTS ARE
MIGHT ~~PROBABLY~~ ^{TD 26 June 1992} NOTE THE EQUILIBRIUM pH VALUES.

| SOLUTION NAME | pH/T(°C) |
|---------------|-----------|
| 2.0 | 2.38/23.6 |
| 2.5 | 3.02/23.6 |
| 3.0 | 3.93/23.6 |
| 3.5 | 4.94/23.7 |
| 4.0 | 5.90/23.7 |
| 4.5 | 6.21/24.1 |
| 5.0 | 6.87/24.1 |
| 5.5 | 6.71/24.2 |
| 6.0 | 6.75/24.2 |
| 6.5 | 6.67/24.3 |
| 7.0 | 6.96/24.3 |
| 7.5 | 7.43/24.4 |
| 8.0 | 7.79/24.5 |
| 8.5 | 8.52/24.6 |
| 9.0 | 9.08/24.6 |
| 9.5 | 9.52/24.7 |

29 JUNE 1992 9:30 TD

EXP B-V (CONT)

REMEASURED THE pH OF ALL SOLNS.

| SOLUTION NAME | pH/T(°C) |
|---------------|-----------|
| 2.0 | 2.37/22.3 |
| 2.5 | 3.00/22.3 |
| 3.0 | 3.94/22.3 |
| 3.5 | 4.89/22.4 |
| 4.0 | 6.12/22.4 |
| 4.5 | 6.33/22.4 |
| 5.0 | 6.67/22.4 |
| 5.5 | 6.79/22.5 |
| 6.0 | 6.76/22.5 |
| 6.5 | 6.81/22.5 |
| 7.0 | 7.00/22.5 |
| 7.5 | 7.40/22.6 |
| 8.0 | 7.80/22.6 |
| 8.5 | 8.53/22.6 |
| 9.0 | 9.08/22.6 |
| 9.5 | 9.54/22.6 |

7/2/92 Zero point of charge (ZPC)

A method for determining the pHzpc of a given mineral is being developed - this experiment is to give practical experience in the method + to find + modify difficulties present in the method.

0920 PM Previously milled quartz is cleaned ultrasonically by repeated washings with ultra pure water. The powder is put in ultra pure water, cleaned for 10 min, the supernatant allowed to set for ~~20~~ ^{40 min} ~~min~~ ^{7/2/92} + then poured off, + the process repeated. When the supernatant is clear, the cleaning is finished.

0925 PM Preparation of 0.1M, 0.01M, + 0.001M NaNO_3
 A 0.1M NaNO_3 solution is made by weighing out 4.2497g NaNO_3 and dissolving it with ultra pure water in a 500 mL volumetric flask.

wt NaNO_3 used: 4.2496g Lot # 7808 KDOE

A 0.01M NaNO_3 solution is made by taking 20 mL of the 0.1M NaNO_3 solution by volumetric pipet, transferring the aliquot to a 200 mL volumetric flask, + diluting to volume with ultra pure water.

A 0.001M NaNO_3 solution is made by taking 20 mL of the 0.01M NaNO_3 solution by volumetric pipet, transferring the aliquot to a 200 mL volumetric flask, + diluting to volume with ultra pure water.

1030 TD EXPERIMENT B.V

MEASURED Na^+ CONCENTRATION OF THE B.V*PHI SOLUTIONS USING THE Na^+ ION SELECTIVE ELECTRODE.

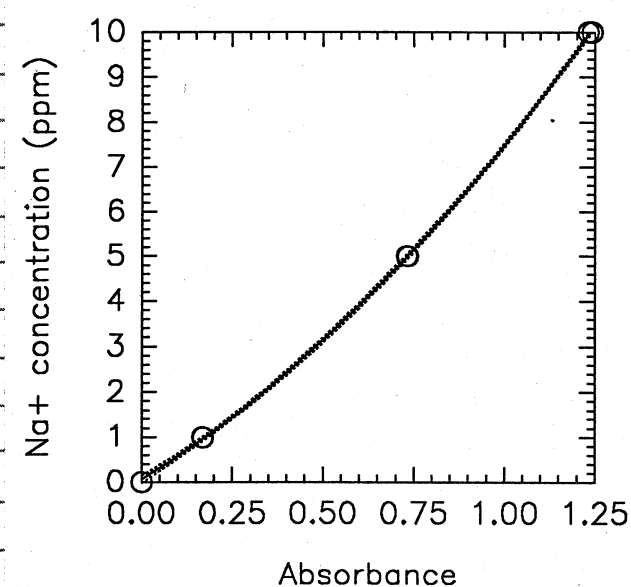
ONLY THOSE SOLUTIONS WHOSE EXPECTED Na^+ CONCENTRATION WAS GREATER THAN 10 ppm WERE ANALYZED. THE REST WILL BE ANALYZED ON THE AA MACHINE.

| SOLN NAME | Na^+ CONC (ppm) | EXPECTED Na^+ CONC (ppm) |
|----------------|--------------------------|-----------------------------------|
| TD B.V*PHI 2.0 | 66.7 | 80.4 |
| 2.5 | 40.3 | 40.3 |
| 3.0 | 16.1 | 13.3 |
| 8.0 | 16.4 | 18.4 |
| 8.5 | 60.1 | 58.5 |
| 9.0 | 201 | 201 |
| 9.5 | 917 | 919 |

6 JULY 1992 0906 TD EXPERIMENT B.V

ANALYZED THE REMAINING B.V SOLUTIONS FOR Na^+ USING THE AA. ONLY THE SOLUTIONS WHOSE Na^+ CONC. WAS EXPECTED TO BE LESS THAN 10 ppm WERE RUN. ABSORBANCES WERE READ DIRECTLY FROM THE MACHINE AND PLOTTED ON SIGMA PLOT. THE SOLUTION CONCENTRATIONS WERE CALCULATED FROM THE SECOND ORDER REGRESSION LINE

CALIBRATION CURVE



CALIBRATION VALUES

| Na^+ STD | ABSORBANCE | | |
|-------------------|------------|-------|-------|
| | (1) | (2) | (3) |
| 0 | 0 | 0 | 0 |
| 1 | .169 | .167 | .168 |
| 5 | .731 | .734 | .735 |
| 10 | 1.243 | 1.232 | 1.233 |

7/6/92

MEASUREMENTS

| NAME | ABSORBANCE | | | Na ⁺ (ppm)(1) | Na ⁺ (ppm)(2) |
|------------|------------|-------|-------|-----------------------------|--------------------------|
| | (1) | (2) | (3) | | |
| B.V. # H35 | 1.087 | 1.085 | 1.082 | 9.36 | 8.33 |
| 4.0 | .659 | .657 | .654 | 4.37 | 4.59 |
| 4.5 | .650 | .651 | .653 | ^{1/6/12} 4.09 4.32 | 4.53 |
| 5.0 | .622 | .624 | .621 | ^{7/1/92} 3.409 | 4.27 |
| 5.5 | .603 | .601 | .598 | 3.92 | 4.01 |
| 6.0 | .719 | .718 | .719 | 4.89 | 5.07 |
| 6.5 | .767 | .766 | .770 | 5.31 | 5.48 |
| 7.0 | .823 | .825 | .822 | 5.81 | 5.92 |
| 7.5 | 1.247 | 1.239 | 1.235 | 10.04 | 10.06 |

THE STANDARD CONDITIONS FOR Na⁺ ANALYSIS WERE TAKEN FROM "ANALYTICAL METHODS FOR ATOMIC ABSORPTION SPECTROPHOTOMETRY." THESE CONDITIONS INCLUDED WAVELENGTH, FLAME TYPE, ETC.

THE OPTIMIZATION AND INSTALLATION OF THE LAMP, FLAME, AND SAMPLE RUN PROCEDURES WERE TAKEN FROM "RUNNING THE 3100 OPERATING INSTRUCTIONS."

THE FIRST Na⁺ MEAS. VALUE IS THE FIRST RUN, CALCULATED BY SIGMA PLOT. THE SECOND VALUE WAS FROM THE CALCULATIONS DONE BY THE AA IN THE SECOND RUN.

July
7 JUNE 1992 0900 TO EXPERIMENT B.V. (CONT)
TO 7/1/92

REMEASURED THE ^{Na⁺ CONC.} ~~PH~~ OF THE B.V. SOLNS THAT WERE MEASURED YESTERDAY ON THE A.A. USING THE ^{1/6/12} 10m SELECTIVE ELECTRODE. 1, 5, AND 10 ppm Na⁺ STANDARDS WERE PREPARED BY ^{TD} SERIAL DILUTION OF THE 1000ppm STANDARD (LOT# VT1) TO 1000 ML IN A VOLUMETRIC FLASK. 1 ML OF 10M STRENGTH ADJUSTER WAS ADDED TO EACH 10ml STANDARD AND SAMPLE.

RESULTS OF Na⁺ CONC. ANALYSIS

| NAME | Na ⁺ CONC. (ppm) |
|------|-----------------------------|
| 3.5 | 8.79 |
| 4.0 | 4.61 |
| 4.5 | 4.54 |
| 5.0 | 4.31 |
| 5.5 | 4.06 |
| 6.0 | 5.07 |
| 6.5 | 5.65 |
| 7.0 | 6.01 |
| 7.5 | 10.6 |

SLOPE = 58.1 mV

1000 TO EXPERIMENT B.V. (CONT)

REMEASURED THE Na⁺ CONCENTRATION OF THE REMAINING B.V. SOLNS. THE 156 WAS AGAIN USED. THE ELECTRODE WAS CALIBRATED WITH 10, 100, AND 1000ppm STANDARDS. THE ^{TD 7/1/92} 1000ppm STANDARD WAS TAKEN DIRECTLY FROM THE PURCHASED 1000ppm SOLUTION (LOT# VVI). LIKEWISE, THE 1000ppm WAS TAKEN DIRECTLY FROM THE 1000ppm SOLUTION (LOT# VT1). THE 10ppm WAS MADE BY DILUTING 1ml 100ppm TO 10ml WITH ULTRAPURE H₂O. 1ml ISA WAS ADDED TO EACH 10ml STANDARD.

RESULTS OF Na⁺ CONC. ANALYSIS

| NAME | Na ⁺ CONC (ppm) |
|------|----------------------------|
| 2.0 | 66.3 |
| 2.5 | 40.1 |
| 3.0 | 15.7 |
| 3.8 | 16.0 |
| 3.5 | 59.9 |
| 9.0 | 201 |
| 9.5 | 910 |

7/7/92 PM ZPC cont

0746 An acid/base titration will be carried out on the titrator.

titrant: 0.111 M HCl, made 2/1/92

(Not standardized by titration previously)

solution to be titrated: 0.01 M NaOH solution

0750 PM 0.01 M NaOH preparation

50 mL of the 0.1 M NaOH solution from 1/28/92 was diluted to volume in a 500.0 mL volumetric flask with ultrapure water.

The titrator was calibrated using Fisher pH buffers 4.0 & 7.0 (lot # 910359-24 & 910870-24).

The titrator used is a Mettler DL21 with a Mettler DG11-5C combination glass electrode. pH buffers 1.0 & 10.0 were also read for reference.

| pH buffer | mV reading |
|-----------|------------|
| 1.00 | 296.90 |
| 4.00 | 147.7 |
| 7.00 | -2.3 |
| 10.00 | -156.7 |

The titrator is configured to output to a matrix printer to CL serial interface 2400 baud, 8 bit, no parity, at least 2K data buffer (P8: . . . 1).

Parameter 1 (titration type) is set to First equiv. pt
 2 (time response) (P1: . . . 1)
 (P2: . . . 1) i.e. standard
 3 (pre-dispensing) is set to 0.5 mL
 4 (burette vol) 10 mL
 6 (stirring time) 0.5

See Operating Instructions for other possible parameter changes.

titration results from printout:

| | |
|-------------------------------|---------|
| starting mV of NaOH solution: | -254.5 |
| mL HCl result | 4.5652 |
| equiv. pt potential in mV | 5.9 |
| calc. conc. of NaOH, M | 0.01014 |

The calculated concentration of the NaOH can be checked by the relation $M_{HCl} V_{HCl} = M_{NaOH} V_{NaOH}$
 $(0.111)(4.5652) = M_{NaOH}(50)$
 $M_{NaOH} = 0.0101$

8 July 1992; Todd Dietrich; 1000 hrs.

Experiment B-V: Silica Analysis

Conducted Silica Analysis on the B-V solutions labeled 3.5 - 7.5. The Milton Roy Spectrometer 1201 was used in the analysis. All solutions had a 20 ml aliquot taken and diluted to 50 ml. The procedure followed, including reagents, materials, and supplies, is recorded as Technical Operating Procedure 14 and is kept in an appropriately labeled binder in the lab office. The dilution factor is $20:57 = 2.85$.

Calibration of Spectrophotometer

| Silica Standard | Absorbance |
|-----------------|------------|
| 44 ppb | .015 |
| 175 ppb | .064 |
| 526 ppb | .118 |
| 1052 ppb | .373 |

LINEAR FIT
 NOT THROUGH 0
 SLOPE 2823
 INTERCEPT -3.310
 CORR COEF 1.000

Results of Silica Analysis

| B-V pH | Conc. (ppb) | Adj. conc (ppb) |
|--------|-------------|-----------------|
| 3.5 | 679 | 1935 |
| 4.0 | 431 | 1228 |
| 4.5 | 300 | 855 |
| 5.0 | 331 | 943 |
| 5.5 | 353 | 1007 |
| 6.0 | 170 | 485 |
| 6.5 | 309 | 881 |
| 7.0 | 284 | 809 |
| 7.5 | 218 | 621 |

7/8/92 PM ZPC cont

0742 The DC21 titrator was calibrated using the samp
4 Fisher buffers as yesterday.

| pH | mV read | room temp: 72.2°F |
|-------|---------|--------------------------------------|
| 4.00 | 144.7 | used to automatically calb. titrator |
| 7.00 | -1.9 | |
| 1.00 | 289.5 | |
| 10.00 | -143.8 | |

The titrator's parameters were set as follows:

| | |
|----|----|
| P1 | 3 |
| 2 | 31 |
| 3 | 0 |
| 4 | 10 |
| 5 | 0 |
| 6 | 0 |
| 7 | 20 |
| 8 | 1 |
| 9 | 0 |
| A | 0 |
| b | 0 |
| C | 0 |

With these parameters the titrator will gradually dispense the entire contents of the burette (10 mL) while printing out the amount dispensed + mV reading.

These parameters were checked using
0.111 M HCl as titrant (same as yesterday) +
0.1 and 0.01 M NaNO₃ solutions to be titrated.

10 JULY 1992 1000 TO EXPERIMENT B-V (CONT)

FINISHED SIUCA ANALYSIS ON THE REMAINING B-V SOLUTIONS. THE MILTON
ROT SPECTROMETER 1201 WAS USED FOR THE ANALYSIS. THE PROCEDURE
FOLLOWED WAS THE SAME AS THAT ON 8 JULY, TECHNICAL OPERATING
PROCEDURE 14. THE DILUTION FACTOR IS $10257 = 5.7$.

CALIBRATION

| STD CONC (ppb) | ABSORPTANCE | |
|----------------|-------------|--------------------|
| 44 | 0.016 | SLOPE = 2870 |
| 175 | 0.063 | |
| 526 | 0.183 | INTERCEPT = -6.186 |
| 1052 | 0.386 | CORR. COEF = 1.008 |

RESULTS OF ANALYSIS

| B-V x pH | CONC (ppb) | CORRECTED CONC (ppb) |
|----------|------------|----------------------|
| 2.0 | 1781 | 10152 |
| 2.5 | 459 | 2616 |
| 3.0 | 370 | 2109 |
| 8.0 | 154 | 979 |
| 8.5 | 174 | 992 |
| 9.0 | 264 | 1505 |
| 9.5 | 548 | 3124 |

Experiment B-V

Aluminum Analysis Using the Spectrophotometer
14 July 1992, Analysis by Todd Dietrich

Analysis of Aluminum concentration was performed on some of the Experiment B-V solutions. The only solutions that remained were those from 3.5 to 7.5. All the other solutions were gone by the time of this analysis. The procedure for this analysis is found in Technical Operating Procedure 013.

Calibration of Spectrophotometer

| Al Std. | Abs. |
|---------|-------|
| 10 | 0.011 |
| 20 | 0.034 |
| 100 | 0.246 |
| 200 | 0.509 |
| 280 | 0.660 |

SLOPE = 405.7
INTERCEPT = 3.528
CORR. COEF. = .9982

Results of Analysis of Al concentration

| B-V* | Al conc. (ppb) | Corrected conc (ppb) |
|------|----------------|----------------------|
| 3.5 | 3.9 | 6.50 |
| 4.0 | 3.9 | 6.50 |
| 4.5 | 3.9 | 6.50 |
| 5.0 | 2.8 | 4.67 |
| 5.5 | 3.2 | 5.33 |
| 6.0 | 3.0 | 5.00 |
| 6.5 | 2.7 | 4.50 |
| 7.0 | 2.9 | 4.83 |
| 7.5 | 3.9 | 6.5 |

All the measured concentrations are below the detection limit of this method. The dilution is 30 : 50 = 1.67.

+ 910870-24).

| 0832 PM | pH | mV read | room temp °F |
|---------|-------|---------|-----------------------------|
| | 4.00 | 140.1 | used to auto-calibrate 72.6 |
| | 7.00 | -1.8 | titrator |
| | 1.00 | 282.7 | |
| | 10.00 | -148.6 | |

added 8/4/94

0.0500g of alumina, previously washed in ultrasound cleaner was added to each of 3 titration cups. The cups were labeled 0.1 M NaNO_3 or 0.01 M NaNO_3 or 0.001 M NaNO_3 . 50 mL of the appropriate NaNO_3 solution was added to each titration cup.

The titrator was set to the following parameters:

| | |
|-------|----|
| 1 | 3 |
| 2 | 26 |
| 3 | 0 |
| 4 | 10 |
| 5 | 0 |
| 6 | 20 |
| 7 | 20 |
| 8 | 1 |
| 9 | 0 |
| a,b,c | 0 |

PM 7/16/92

0.102 0.1014 M NaOH (see 6C-07-125) was used to titrate the 0.001 M NaNO_3 / alumina mixture up to ~ -223.2 mV.

The buret was cleaned, along with the suction + delivery tubes with deionized ultra pure water, then air-dried.

0.111 M HCl (from 2/11/92) was then used to titrate the solution down to ~ 40 mV.

7/16/92 PM NaNO_3 / alumina titration

0.1 M, 0.01 M + 0.001 M NaNO_3 solutions were made as described on 6C-07-120

wt NaNO_3 used: 4.2496 g Lot # 7808 KDOE

The titrator was calibrated using 4 Fisher pH buffers, pH = 1.00, 4.00, 7.00, + 10.00 (lot #s 910191-18, 910359-24, 910943-24,

The titration with NaOH + HCl was then repeated with the 0.01 + 0.1 M NaNO₃/alumina mixtures.

Sample print-out (reduced-size) of 0.1014M NaOH titration of alumina in 0.001 M NaNO₃:

----- METTLER DL21 Titrator date 07-16-92 -----

TITRATION 3 operator

METHOD 3 26 07-16-92

WEIGHT g .05

IDENT .001 remarks

conc mol/L .11100

const 1.0000

START mV 58.7

| VOLUME mL | INCREMENT mL | SIGNAL mV | S.CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|----------------|---------------------|
| .00000 | .00000 | 58.7 | | |
| .00500 | .00500 | 17.7 | -41.0 | -8208.9 |
| .01000 | .00500 | -82.2 | -99.8 | -19969. |
| .01500 | .00500 | -102.5 | -20.3 | -4069.7 |
| .02500 | .01000 | -123.9 | -21.4 | -2140.0 |
| .04000 | .01500 | -139.0 | -15.1 | -1006.5 |
| .05000 | .01000 | -144.7 | -5.7 | -573.26 |
| .07000 | .02000 | -153.6 | -8.9 | -444.09 |
| .09500 | .02500 | -161.3 | -7.6 | -305.67 |
| .14000 | .04500 | -170.0 | -8.7 | -193.83 |
| .23000 | .09000 | -181.4 | -11.4 | -126.88 |
| .32500 | .09500 | -188.6 | -7.2 | -76.283 |
| .51000 | .18500 | -198.5 | -9.8 | -53.021 |
| .73000 | .22000 | -206.0 | -7.5 | -34.308 |
| 1.1700 | .44000 | -216.1 | -10.1 | -22.881 |
| 1.6700 | .50000 | -223.2 | -7.1 | -14.182 |

0902

7/21/92 PKC

NaNO₃ / quartz titration

The titration will be modified by the bubbling of N₂ gas through the mixture before + during titration.

0.1M, 0.01M, + 0.001M NaNO₃ solutions were prepared as described in GC-07-120.

wt NaNO₃ used: 4.2516g lot# 7808 KDOE

pH buffers 4.0, 7.0, 1.0, + 10.0 (lot#s 910191-18, 910359-24, 910943-24 + 910870-24) were each purged for 10 minutes with N₂ gas + the mV reading checked.

1115 PM pH 4.0 mV = 119.9

1125 PM 126.8

1128 PM pH 7.0 mV = -2.5

1138 PM -2.7

1140 PM pH 1.0 mV = 265.4

1150 PM 268.4

1151 PM pH 10.0 mV = -142.1

1201 PM -140.8

1317 PM The titrator / pH electrode was calibrated using the 4.0 + 7.0 pH buffers (automatic) + the pH = 1.0 + 10.0 buffers were measured + the mV reading of each noted.

pH 4.0 132.8

7.0 -1.1

1.0 265.1

10.0 -134.6

pH calibration line (1st order regression):

$$mV = -44.43 (pH) + 309.93$$

0.04 g of previously ultrasonically cleaned quartz is weighed, + 40 mL of the 0.1M NaNO₃ solution is added to the powder.

wt powder used: 0.0405 g

1332 PM The NaNO_3 / quartz solution is stirred + purged with N_2 gas while it is monitored for mV reading. When a constant mV reading is achieved, the solution will be titrated.

init mV reading: 67.2 mV room temp: 75.5 °F
(purge begun)

1339 PM 47.6 mV 75.6 °F

1400 PM 45.2 mV "

(N_2 turned up)

1410 PM 44.7 mV

1415 PM 45.4 mV

1416 PM Titration was started with 30 s stirring, w. purge continued throughout.

Reduced copy of
printer output from
titration

(0.111 M HCl titrant)

The signal change
is down to ~15 mV
per 90 seconds, which
is probably too slow.

Parameters:

1 type 3
2 control 4090
3 predosing 0
4 bur/max vol 10
5 0
6 stir time 30
7 output 20
8 system 1

| ----- METTLER DL21 Titrator date 07-21-92 ----- | | | | | |
|---|--------------|-----------|-------------|------------------|--|
| TITRATION | 1 | operator | | | |
| METHOD | 3 | 4090 | 07-21-92 | | |
| WEIGHT g | .0405 | | | | |
| IDENT | .1 | remarks | | | |
| conc mol/L | .11100 | | | | |
| const | 1.0000 | | | | |
| START mV | 46.0 | | | | |
| VOLUME mL | INCREMENT mL | SIGNAL mV | S.CHANGE mV | DERIVATIVE mV/mL | |
| .00000 | .00000 | 46.0 | | | |
| .01000 | .01000 | 99.2 | 53.2 | 5320.4 | |
| .02000 | .01000 | 110.7 | 11.5 | 1152.1 | |
| .03000 | .01000 | 117.7 | 7.0 | 704.43 | |
| .04000 | .01000 | 123.2 | 5.4 | 543.09 | |
| .05000 | .01000 | 127.4 | 4.2 | 422.42 | |
| .06000 | .01000 | 130.7 | 3.3 | 332.56 | |
| .07000 | .01000 | 133.1 | 2.4 | 241.06 | |
| .08000 | .01000 | 135.5 | 2.4 | 241.36 | |
| .09000 | .01000 | 138.3 | 2.7 | 273.99 | |
| .10000 | .01000 | 140.6 | 2.3 | 228.09 | |
| .11000 | .01000 | 142.4 | 1.8 | 182.48 | |
| .12000 | .01000 | 143.5 | 1.1 | 110.93 | |
| .13000 | .01000 | 144.9 | 1.4 | 142.26 | |
| .14000 | .01000 | 146.8 | 1.9 | 185.89 | |
| .15000 | .01000 | 148.5 | 1.7 | 170.27 | |
| .16000 | .01000 | 149.9 | 1.5 | 146.18 | |
| .17000 | .01000 | 150.6 | .7 | 67.183 | |
| .18000 | .01000 | 151.8 | 1.2 | 122.00 | |
| .19000 | .01000 | 153.6 | 1.7 | 173.78 | |
| .20000 | .01000 | 155.2 | 1.7 | 167.44 | |
| .21000 | .01000 | 156.4 | 1.2 | 118.94 | |
| .22000 | .01000 | 156.9 | .5 | 46.033 | |
| .23000 | .01000 | 157.6 | .7 | 73.496 | |
| .24000 | .01000 | 159.0 | 1.4 | 139.29 | |
| .25000 | .01000 | 160.5 | 1.5 | 152.85 | |
| .26000 | .01000 | 161.5 | .9 | 91.724 | |
| .27000 | .01000 | 161.7 | .2 | 19.156 | |
| .28000 | .01000 | 162.2 | .6 | 55.908 | |

7/22/92 PM

NaNO_3 / quartz titration

The titration will be rerun like yesterday's, but the addition of titrant will be increased to 0.02 mL per 90 s interval.

0803 PM

Preparation of 0.121 M HCl solution for titration

5 mL of concentrated HCl (~12.1 M), lot # FL-02-0786 were diluted to volume in a 500 mL volumetric flask with ultrapure water. The contents were mixed + transferred into a 500 mL polypropylene bottle.

0210 PM

The titrator / electrode was calibrated using pH = 4.0, 7.0, 1.0, + 3.0 (lot # 910886-24) Fisher brand buffers.

pH mV read

4.0 132.0

7.0 3.0

1.0 264.5

3.0 176.8 @ 71.8 °F room temperature

pH equation $\text{mV} = -43.59(\text{pH}) + 307.52$

The 0.121 M HCl solution was used to titrate 0.0401 g of quartz powder in 40 mL of 0.1 M NaNO_3 solution (made 7/21/92).

Parameters:

1 3 5 0
2 5090 6 30
3 0 7 20
4 10 8 1
9, a, b, c 0

The solution was purged with N_2 gas for 10 min before the titration was started, + was continuously purged during titration.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S.CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|----------------|---------------------|
| .00000 | .00000 | 82.3 | | |
| .02000 | .02000 | 125.8 | 43.5 | 2174.8 |
| .04000 | .02000 | 137.3 | 11.5 | 574.44 |
| .06000 | .02000 | 143.9 | 6.6 | 331.63 |
| .08000 | .02000 | 149.4 | 5.5 | 273.30 |
| .10000 | .02000 | 153.8 | 4.5 | 222.85 |
| .12000 | .02000 | 157.4 | 3.6 | 177.61 |
| .14000 | .02000 | 160.2 | 2.8 | 139.82 |
| .16000 | .02000 | 162.2 | 2.1 | 104.19 |
| .18000 | .02000 | 164.7 | 2.5 | 124.34 |
| .20000 | .02000 | 167.5 | 2.8 | 139.81 |
| .22000 | .02000 | 169.7 | 2.2 | 108.31 |
| .24000 | .02000 | 171.4 | 1.8 | 87.953 |
| .26000 | .02000 | 172.4 | 1.0 | 47.769 |
| .28000 | .02000 | 174.2 | 1.8 | 91.723 |
| .30000 | .02000 | 176.4 | 2.2 | 107.93 |
| .32000 | .02000 | 178.1 | 1.7 | 86.644 |
| .34000 | .02000 | 179.4 | 1.3 | 64.224 |
| .36000 | .02000 | 180.0 | .6 | 29.656 |
| .38000 | .02000 | 181.5 | 1.4 | 72.357 |
| .40000 | .02000 | 183.1 | 1.6 | 81.008 |
| .42000 | .02000 | 184.5 | 1.4 | 71.942 |
| .44000 | .02000 | 185.6 | 1.1 | 53.483 |
| .46000 | .02000 | 186.0 | .4 | 19.524 |
| .48000 | .02000 | 186.9 | 1.0 | 48.454 |
| .50000 | .02000 | 188.4 | 1.4 | 70.911 |
| .52000 | .02000 | 189.3 | 1.0 | 49.294 |
| .54000 | .02000 | 189.9 | .5 | 27.395 |
| .56000 | .02000 | 189.6 | -2 | -12.265 |
| .58000 | .02000 | 190.4 | .8 | 38.616 |
| .60000 | .02000 | 191.8 | 1.4 | 69.452 |
| .62000 | .02000 | 192.7 | .9 | 46.303 |
| .64000 | .02000 | 193.2 | .5 | 25.346 |
| .66000 | .02000 | 193.1 | -2 | -7.8232 |
| .68000 | .02000 | 193.0 | .7 | 33.396 |
| .70000 | .02000 | 195.1 | 1.3 | 65.248 |
| .72000 | .02000 | 195.8 | .8 | 38.915 |
| .74000 | .02000 | 196.0 | .2 | 8.3588 |
| .76000 | .02000 | 195.9 | -1 | -6.7879 |
| .78000 | .02000 | 196.8 | .9 | 46.768 |
| .80000 | .02000 | 198.0 | 1.2 | 60.387 |
| .82000 | .02000 | 198.7 | .7 | 35.042 |
| .84000 | .02000 | 198.8 | .1 | 5.5000 |
| .86000 | .02000 | 198.7 | -1 | -6.0227 |
| .88000 | .02000 | 199.6 | .9 | 44.753 |
| .90000 | .02000 | 200.8 | 1.2 | 62.125 |
| .92000 | .02000 | 201.6 | .7 | 35.548 |
| .94000 | .02000 | 201.5 | -1 | -2.6016 |
| .96000 | .02000 | 201.3 | -2 | -8.6395 |
| .98000 | .02000 | 201.9 | .5 | 27.451 |
| 1.00000 | .02000 | 203.1 | 1.2 | 60.423 |
| 1.02000 | .02000 | 203.9 | .8 | 39.793 |
| 1.04000 | .02000 | 203.9 | .0 | 1.5640 |
| 1.06000 | .02000 | 203.8 | -1 | -3.6583 |
| 1.08000 | .02000 | 204.7 | .9 | 43.189 |
| 1.10000 | .02000 | 205.6 | .9 | 45.843 |
| 1.12000 | .02000 | 206.3 | .7 | 32.744 |
| 1.14000 | .02000 | 206.1 | -1 | -7.3311 |
| 1.16000 | .02000 | 206.3 | .2 | 7.8552 |
| 1.18000 | .02000 | 207.3 | 1.0 | 48.754 |
| 1.20000 | .02000 | 208.4 | 1.2 | 58.467 |
| 1.22000 | .02000 | 209.2 | .7 | 37.090 |
| 1.24000 | .02000 | 209.3 | .1 | 4.4525 |
| 1.26000 | .02000 | 209.4 | .1 | 6.5437 |
| 1.28000 | .02000 | 210.6 | 1.2 | 60.533 |
| 1.30000 | .02000 | 211.8 | 1.2 | 60.045 |
| 1.32000 | .02000 | 212.5 | .7 | 35.665 |
| 1.34000 | .02000 | 212.6 | .1 | 4.2061 |
| 1.36000 | .02000 | 213.0 | .4 | 20.982 |
| 1.38000 | .02000 | 214.0 | 1.0 | 49.876 |
| 1.40000 | .02000 | 214.9 | .9 | 45.964 |
| 1.42000 | .02000 | 215.4 | .5 | 22.588 |
| 1.44000 | .02000 | 215.2 | -2 | -9.1934 |
| 1.46000 | .02000 | 215.7 | .5 | 22.866 |
| 1.48000 | .02000 | 216.7 | 1.0 | 52.155 |
| 1.50000 | .02000 | 217.6 | .9 | 46.459 |
| 1.52000 | .02000 | 218.0 | .4 | 18.636 |
| 1.54000 | .02000 | 217.8 | -2 | -8.3939 |
| 1.56000 | .02000 | 218.1 | .2 | 12.079 |
| 1.58000 | .02000 | 219.1 | 1.0 | 50.430 |

| | | | | |
|--------|--------|-------|-----|---------|
| 1.6000 | .02000 | 220.0 | .9 | 44.920 |
| 1.6200 | .02000 | 220.4 | .4 | 21.563 |
| 1.6400 | .02000 | 220.4 | -.0 | -1.5854 |
| 1.6600 | .02000 | 220.8 | .4 | 19.203 |
| 1.6800 | .02000 | 221.8 | 1.0 | 51.804 |
| 1.7000 | .02000 | 222.8 | 1.0 | 50.787 |
| 1.7200 | .02000 | 223.4 | .6 | 27.899 |
| 1.7400 | .02000 | 223.3 | -1 | -5.2673 |
| 1.7600 | .02000 | 223.6 | .3 | 15.801 |
| 1.7800 | .02000 | 224.6 | 1.0 | 50.186 |
| 1.8000 | .02000 | 225.5 | .9 | 44.439 |
| 1.8200 | .02000 | 225.9 | .4 | 18.676 |

Data was taken at 90s intervals; total titration time using 0.121 M HCl = 136.5 minutes.

1140PM Parameter 2 was changed to 6090 (increasing ml increment value of titrant to 0.05) 0.1014 M NaOH was used to titrate the solution back to its beginning pH value.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S.CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|----------------|---------------------|
| .00000 | .00000 | 223.5 | | |
| .05000 | .05000 | 222.8 | -.7 | -14.193 |
| .10000 | .05000 | 221.5 | -1.4 | -27.100 |
| .15000 | .05000 | 220.0 | -1.5 | -29.564 |
| .20000 | .05000 | 218.9 | -1.0 | -20.925 |
| .25000 | .05000 | 218.5 | -.4 | -7.8845 |
| .30000 | .05000 | 217.8 | -.8 | -15.343 |
| .35000 | .05000 | 216.4 | -1.4 | -27.833 |
| .40000 | .05000 | 215.0 | -1.4 | -27.076 |
| .45000 | .05000 | 214.0 | -1.1 | -21.326 |
| .50000 | .05000 | 213.6 | -.3 | -6.4420 |
| .55000 | .05000 | 212.9 | -.7 | -14.243 |
| .60000 | .05000 | 211.7 | -1.2 | -24.926 |
| .65000 | .05000 | 210.5 | -1.2 | -24.549 |
| .70000 | .05000 | 209.8 | -.7 | -13.843 |
| .75000 | .05000 | 209.4 | -.3 | -6.7136 |
| .80000 | .05000 | 208.8 | -.6 | -12.259 |
| .85000 | .05000 | 207.6 | -1.2 | -23.785 |
| .90000 | .05000 | 206.4 | -1.2 | -24.399 |
| .95000 | .05000 | 205.6 | -.8 | -15.982 |
| 1.00000 | .05000 | 205.3 | -.3 | -5.1358 |
| 1.05000 | .05000 | 204.8 | -.5 | -10.584 |
| 1.10000 | .05000 | 203.7 | -1.1 | -22.948 |
| 1.15000 | .05000 | 202.4 | -1.2 | -24.810 |
| 1.20000 | .05000 | 201.6 | -.9 | -17.373 |
| 1.25000 | .05000 | 201.1 | -.5 | -9.5221 |
| 1.30000 | .05000 | 200.6 | -.5 | -9.5166 |
| 1.35000 | .05000 | 199.2 | -1.4 | -27.291 |
| 1.40000 | .05000 | 197.6 | -1.7 | -33.189 |
| 1.45000 | .05000 | 196.1 | -1.4 | -28.966 |
| 1.50000 | .05000 | 195.2 | -.9 | -18.605 |
| 1.55000 | .05000 | 193.9 | -1.3 | -25.491 |
| 1.60000 | .05000 | 192.0 | -2.0 | -39.146 |
| 1.65000 | .05000 | 189.7 | -2.3 | -46.264 |
| 1.70000 | .05000 | 187.4 | -2.3 | -45.291 |
| 1.75000 | .05000 | 185.2 | -2.2 | -43.259 |
| 1.80000 | .05000 | 182.8 | -2.5 | -49.252 |
| 1.85000 | .05000 | 179.3 | -3.4 | -68.675 |
| 1.90000 | .05000 | 174.6 | -4.7 | -94.323 |
| 1.95000 | .05000 | 169.0 | -5.7 | -113.04 |
| 2.00000 | .05000 | 161.1 | -7.8 | -156.39 |
| 2.05000 | .05000 | 147.6 | -13.6 | -271.41 |
| 2.10000 | .05000 | 69.0 | -78.6 | -1571.2 |

Total titration time using 0.1014 M NaOH = 69 minutes.

1401PM 0.0400g of quartz powder was weighed into a titration cup + 40 mL of 0.01 M NaNO_3 solution (made 7/21/92) was added. The solution was purged with N_2 gas for 10 minutes, & then titrated with 0.121 M HCl using the parameters on page 133.

(Note: the meter/electrode was first recalibrated using Fisher pH buffers 1.0, 3.0, 4.0, & 7.0 as before.)

pH equation: $\text{mV} = -45.59(\text{pH}) + 324.1$

| VOLUME mL | INCREMENT mL | SIGNAL mV | S. CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|-----------------|---------------------|
| .00000 | .00000 | 56.3 | | |
| .02000 | .02000 | 57.2 | .8 | 40.818 |
| .04000 | .02000 | 136.9 | 79.8 | 3987.7 |
| .06000 | .02000 | 150.7 | 13.8 | 689.05 |
| .08000 | .02000 | 159.2 | 8.5 | 422.75 |
| .10000 | .02000 | 165.6 | 6.5 | 323.38 |
| .12000 | .02000 | 170.8 | 5.1 | 256.74 |
| .14000 | .02000 | 174.5 | 3.7 | 185.27 |
| .16000 | .02000 | 177.9 | 3.5 | 173.33 |
| .18000 | .02000 | 180.4 | 2.5 | 124.66 |
| .20000 | .02000 | 182.8 | 2.3 | 116.92 |
| .22000 | .02000 | 184.8 | 2.1 | 104.15 |
| .24000 | .02000 | 186.4 | 1.6 | 77.600 |
| .26000 | .02000 | 187.4 | 1.0 | 49.799 |
| .28000 | .02000 | 188.4 | 1.0 | 48.261 |
| .30000 | .02000 | 189.7 | 1.3 | 58.288 |
| .32000 | .02000 | 191.5 | 1.8 | 59.711 |
| .34000 | .02000 | 192.8 | 1.3 | 51.711 |
| .36000 | .02000 | | | |

1430PM The printer paper stuck while it was recording:
7 additions of 0.02 mL titrant were added while the paper was fixed. The titration was stopped for 15 minutes, then restarted.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S. CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|-----------------|---------------------|
| .00000 | .00000 | 196.2 | | |
| .02000 | .02000 | 196.5 | .3 | 12.679 |
| .04000 | .02000 | 197.4 | .9 | 47.497 |
| .06000 | .02000 | 198.7 | 1.3 | 65.311 |
| .08000 | .02000 | 199.6 | .9 | 45.216 |
| .10000 | .02000 | 199.8 | .2 | 9.1324 |
| .12000 | .02000 | 199.8 | .0 | .78583 |
| .14000 | .02000 | 200.5 | .7 | 33.990 |
| .16000 | .02000 | 201.4 | .9 | 43.424 |
| .18000 | .02000 | 202.5 | 1.1 | 54.179 |
| .20000 | .02000 | 202.9 | .5 | 22.508 |
| .22000 | .02000 | 203.1 | .1 | 6.8138 |
| .24000 | .02000 | 203.5 | .4 | 21.217 |
| .26000 | .02000 | 204.3 | .8 | 40.547 |
| .28000 | .02000 | 205.4 | 1.1 | 56.681 |
| .30000 | .02000 | 206.0 | .5 | 26.952 |
| .32000 | .02000 | 206.1 | .1 | 5.5077 |
| .34000 | .02000 | 206.4 | .3 | 15.968 |
| .36000 | .02000 | 207.1 | .7 | 34.292 |
| .38000 | .02000 | 208.1 | 1.0 | 51.863 |
| .40000 | .02000 | 208.8 | .7 | 33.280 |
| .42000 | .02000 | 208.8 | .0 | .78507 |
| .44000 | .02000 | 209.0 | .2 | 7.6004 |
| .46000 | .02000 | 209.5 | .6 | 29.096 |
| .48000 | .02000 | 210.4 | .9 | 44.582 |
| .50000 | .02000 | 211.4 | 1.0 | 48.535 |
| .52000 | .02000 | 211.4 | .0 | .78888 |

| | | | | |
|--------|--------|-------|-----|---------|
| .54000 | .02000 | 211.3 | -.1 | -3.4187 |
| .56000 | .02000 | 211.9 | .6 | 29.657 |
| .58000 | .02000 | 212.7 | .8 | 40.271 |
| .60000 | .02000 | 213.7 | 1.0 | 50.074 |
| .62000 | .02000 | 214.4 | .6 | 31.735 |
| .64000 | .02000 | 214.3 | -.1 | -2.6268 |
| .66000 | .02000 | 214.6 | .3 | 14.431 |
| .68000 | .02000 | 215.3 | .7 | 36.463 |
| .70000 | .02000 | 216.3 | 1.0 | 48.308 |
| .72000 | .02000 | 217.5 | 1.2 | 61.713 |
| .74000 | .02000 | 218.0 | .4 | 20.489 |
| .76000 | .02000 | 217.9 | -.1 | -4.1985 |
| .78000 | .02000 | 218.3 | .5 | 23.119 |

1549PM Another 0.04 mL of titrant was added to the solution while the parameter 2 was changed to 6090 & the titration continued with HCl.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S. CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|-----------------|---------------------|
| .00000 | .00000 | 219.2 | | |
| .05000 | .05000 | 219.9 | .6 | 12.712 |
| .10000 | .05000 | 221.0 | 1.2 | 23.319 |
| .15000 | .05000 | 222.8 | 1.8 | 35.638 |
| .20000 | .05000 | 224.0 | 1.2 | 23.459 |
| .25000 | .05000 | 224.2 | .2 | 4.6298 |
| .30000 | .05000 | 224.6 | .3 | 6.8430 |
| .35000 | .05000 | 225.1 | .5 | 10.104 |
| .40000 | .05000 | 226.4 | 1.3 | 26.015 |
| .45000 | .05000 | 227.6 | 1.2 | 24.423 |
| .50000 | .05000 | 228.1 | .5 | 10.519 |
| .55000 | .05000 | 228.3 | .1 | 2.6318 |
| .60000 | .05000 | 228.9 | .6 | 12.210 |
| .65000 | .05000 | 230.0 | 1.2 | 23.592 |
| .70000 | .05000 | 231.4 | 1.4 | 27.398 |
| .75000 | .05000 | 232.0 | .6 | 11.388 |
| .80000 | .05000 | 232.1 | .1 | 1.8939 |
| .85000 | .05000 | 232.4 | .3 | 5.9061 |
| .90000 | .05000 | 233.4 | 1.0 | 20.254 |
| .95000 | .05000 | 234.6 | 1.3 | 25.226 |
| 1.00000 | .05000 | 235.4 | .7 | 14.349 |
| 1.05000 | .05000 | 235.4 | .0 | .63507 |
| 1.10000 | .05000 | 235.5 | .1 | 1.8976 |
| 1.15000 | .05000 | 236.1 | .6 | 11.701 |
| 1.20000 | .05000 | 237.3 | 1.2 | 23.629 |
| 1.25000 | .05000 | 237.9 | .7 | 13.611 |
| 1.30000 | .05000 | 237.9 | -.0 | -.21088 |
| 1.35000 | .05000 | 238.0 | .1 | 1.4795 |
| 1.40000 | .05000 | 239.0 | 1.0 | 19.637 |
| 1.45000 | .05000 | 240.4 | 1.4 | 28.419 |

Total titration time using 0.121 M HCl: 138 minutes

1653PM 0.1014 M NaOH was used to titrate the solution back to its original pH.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S. CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|-----------------|---------------------|
| .00000 | .00000 | 241.0 | | |
| .05000 | .05000 | 241.6 | .6 | 11.419 |
| .10000 | .05000 | 241.8 | .2 | 3.2764 |
| .15000 | .05000 | 241.2 | -.6 | -11.841 |
| .20000 | .05000 | 240.3 | -.9 | -17.862 |
| .25000 | .05000 | 240.1 | -.2 | -3.9102 |
| .30000 | .05000 | 240.5 | .4 | 8.3490 |
| .35000 | .05000 | 240.6 | .1 | 1.1588 |
| .40000 | .05000 | 239.9 | -.7 | -13.946 |
| .45000 | .05000 | 238.8 | -1.0 | -20.809 |
| .50000 | .05000 | 238.3 | -.5 | -9.3964 |
| .55000 | .05000 | 238.6 | .3 | 5.5936 |
| .60000 | .05000 | 238.7 | .1 | 1.3742 |
| .65000 | .05000 | 238.1 | -.6 | -11.717 |
| .70000 | .05000 | 237.2 | -.9 | -18.678 |
| .75000 | .05000 | 236.8 | -.4 | -8.3399 |
| .80000 | .05000 | 237.1 | .4 | 7.1747 |
| .85000 | .05000 | 237.2 | .0 | .84717 |

| | | | | |
|---------|--------|-------|-------|---------|
| .90000 | .05000 | 236.5 | -.6 | -12.241 |
| .95000 | .05000 | 235.5 | -1.0 | -20.560 |
| 1.00000 | .05000 | 235.1 | -.4 | -7.6965 |
| 1.05000 | .05000 | 235.2 | .1 | 1.4768 |
| 1.10000 | .05000 | 235.2 | .0 | .31677 |
| 1.15000 | .05000 | 234.8 | -.4 | -7.9068 |
| 1.20000 | .05000 | 233.7 | -1.1 | -22.267 |
| 1.25000 | .05000 | 233.0 | -.8 | -15.194 |
| 1.30000 | .05000 | 233.0 | .0 | .63446 |
| 1.35000 | .05000 | 233.0 | .0 | .31769 |
| 1.40000 | .05000 | 232.3 | -.7 | -13.712 |
| 1.45000 | .05000 | 231.3 | -1.1 | -21.301 |
| 1.50000 | .05000 | 230.4 | -.8 | -16.969 |
| 1.55000 | .05000 | 230.5 | .1 | 1.7941 |
| 1.60000 | .05000 | 230.4 | -.1 | -1.7941 |
| 1.65000 | .05000 | 229.8 | -.6 | -12.956 |
| 1.70000 | .05000 | 228.6 | -1.1 | -22.532 |
| 1.75000 | .05000 | 227.7 | -.9 | -17.998 |
| 1.80000 | .05000 | 227.6 | -.1 | -2.5241 |
| 1.85000 | .05000 | 227.5 | -.1 | -1.3690 |
| 1.90000 | .05000 | 226.9 | -.7 | -13.045 |
| 1.95000 | .05000 | 225.5 | -1.3 | -26.741 |
| 2.00000 | .05000 | 224.5 | -1.0 | -20.424 |
| 2.05000 | .05000 | 224.0 | -.5 | -10.632 |
| 2.10000 | .05000 | 223.6 | -.4 | -7.9978 |
| 2.15000 | .05000 | 222.7 | -.9 | -17.043 |
| 2.20000 | .05000 | 221.4 | -1.4 | -27.333 |
| 2.25000 | .05000 | 220.3 | -1.1 | -21.434 |
| 2.30000 | .05000 | 219.7 | -.6 | -11.971 |
| 2.35000 | .05000 | 219.3 | -.4 | -8.6105 |
| 2.40000 | .05000 | 218.2 | -1.1 | -21.307 |
| 2.45000 | .05000 | 216.7 | -1.5 | -30.901 |
| 2.50000 | .05000 | 215.2 | -1.4 | -28.566 |
| 2.55000 | .05000 | 214.2 | -1.1 | -21.517 |
| 2.60000 | .05000 | 213.3 | -.8 | -16.780 |
| 2.65000 | .05000 | 211.9 | -1.4 | -27.890 |
| 2.70000 | .05000 | 209.9 | -2.0 | -40.025 |
| 2.75000 | .05000 | 207.9 | -2.1 | -41.313 |
| 2.80000 | .05000 | 206.1 | -1.8 | -35.302 |
| 2.85000 | .05000 | 204.6 | -1.5 | -30.559 |
| 2.90000 | .05000 | 202.3 | -2.2 | -44.644 |
| 2.95000 | .05000 | 199.4 | -3.0 | -59.125 |
| 3.00000 | .05000 | 196.1 | -3.3 | -66.443 |
| 3.05000 | .05000 | 192.7 | -3.4 | -68.056 |
| 3.10000 | .05000 | 189.0 | -3.7 | -73.023 |
| 3.15000 | .05000 | 183.8 | -5.2 | -103.87 |
| 3.20000 | .05000 | 176.6 | -7.2 | -143.64 |
| 3.25000 | .05000 | 165.7 | -10.9 | -218.05 |
| 3.30000 | .05000 | 143.7 | -22.0 | -440.96 |

1840PM End of NaOH titration. Total NaOH titration time:
99 minutes.

7/23/92 PM

0753 0.0401g of quartz powder was weighed into a titration cup. 40 mL of 0.001 M Na_2CO_3 solution (made 7/21/92) was added, & the mixture purged with N_2 gas for 10 minutes.

0801PM The titrator/electrode was calibrated using the same Fisher pH buffers as were used yesterday.

pH mV reading room temp: 72.3°F
4.0 125.0
7.0 6.3
1.0 250.2
3.0 170.3

pH equation: $\text{mV} = -40.75(\text{pH}) + 290.76$

The solution was titrated using the same parameters used yesterday. The titrant was 0.121 M HCl.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S.CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|----------------|---------------------|
| .00000 | .00000 | 82.4 | | |
| .02000 | .02000 | 129.3 | 46.8 | 2342.3 |
| .04000 | .02000 | 142.9 | 13.6 | 679.09 |
| .06000 | .02000 | 150.8 | 7.9 | 395.72 |
| .08000 | .02000 | 156.2 | 5.4 | 270.41 |
| .10000 | .02000 | 159.9 | 3.7 | 183.90 |
| .12000 | .02000 | 164.0 | 4.2 | 209.53 |
| .14000 | .02000 | 167.6 | 3.6 | 178.82 |
| .16000 | .02000 | 170.9 | 3.3 | 163.71 |
| .18000 | .02000 | 173.4 | 2.5 | 123.16 |
| .20000 | .02000 | 175.2 | 1.8 | 92.129 |
| .22000 | .02000 | 176.8 | 1.6 | 78.465 |
| .24000 | .02000 | 178.8 | 2.0 | 101.67 |
| .26000 | .02000 | 180.8 | 2.0 | 99.960 |
| .28000 | .02000 | 182.6 | 1.8 | 89.804 |
| .30000 | .02000 | 183.5 | .9 | 46.777 |
| .32000 | .02000 | 184.3 | .8 | 38.267 |
| .34000 | .02000 | 185.5 | 1.2 | 59.647 |
| .36000 | .02000 | 187.3 | 1.8 | 92.010 |
| .38000 | .02000 | 188.8 | 1.5 | 73.299 |
| .40000 | .02000 | 189.8 | 1.0 | 50.420 |
| .42000 | .02000 | 190.6 | .8 | 38.352 |
| .44000 | .02000 | 191.9 | 1.3 | 64.007 |
| .46000 | .02000 | 193.5 | 1.6 | 81.154 |
| .48000 | .02000 | 195.1 | 1.7 | 82.523 |
| .50000 | .02000 | 196.2 | 1.0 | 52.023 |
| .52000 | .02000 | 196.8 | .6 | 32.153 |
| .54000 | .02000 | 197.2 | .4 | 18.561 |
| .56000 | .02000 | 198.5 | 1.3 | 64.100 |
| .58000 | .02000 | 199.6 | 1.2 | 58.921 |
| .60000 | .02000 | 200.2 | .6 | 28.751 |
| .62000 | .02000 | 200.2 | -.0 | -1.8288 |
| .64000 | .02000 | 199.9 | -.3 | -15.424 |
| .66000 | .02000 | 200.9 | 1.0 | 49.687 |
| .68000 | .02000 | 202.0 | 1.1 | 54.659 |
| .70000 | .02000 | 202.6 | .6 | 31.403 |
| .72000 | .02000 | 202.9 | .3 | 16.769 |
| .74000 | .02000 | 202.5 | -.4 | -19.646 |
| .76000 | .02000 | 202.9 | .4 | 18.076 |
| .78000 | .02000 | 204.3 | 1.4 | 70.698 |
| .80000 | .02000 | 205.1 | .8 | 40.361 |
| .82000 | .02000 | 205.5 | .4 | 19.644 |
| .84000 | .02000 | 205.4 | -.1 | -5.2338 |
| .86000 | .02000 | 205.4 | .0 | .00000 |
| .88000 | .02000 | 206.3 | .9 | 44.563 |
| .90000 | .02000 | 207.2 | .9 | 43.612 |
| .92000 | .02000 | 207.6 | .5 | 23.818 |
| .94000 | .02000 | 207.5 | -.1 | -7.3288 |
| .96000 | .02000 | 207.6 | .1 | 3.4020 |
| .98000 | .02000 | 208.2 | .7 | 34.058 |
| 1.00000 | .02000 | 209.0 | .7 | 37.202 |
| 1.02000 | .02000 | 209.5 | .5 | 23.853 |
| 1.04000 | .02000 | 209.3 | -.2 | -7.6096 |
| 1.06000 | .02000 | 209.2 | -.1 | -5.7572 |
| 1.08000 | .02000 | 209.5 | .4 | 17.552 |
| 1.10000 | .02000 | 210.5 | 1.0 | 49.816 |
| 1.12000 | .02000 | 211.3 | .8 | 37.779 |
| 1.14000 | .02000 | 211.6 | .3 | 17.057 |
| 1.16000 | .02000 | 211.6 | -.1 | -3.4096 |

| | | | | |
|--------|--------|-------|-----|----------|
| 1.1800 | .02000 | 211.9 | .4 | 18.114 |
| 1.2000 | .02000 | 213.1 | 1.1 | 57.220 |
| 1.2200 | .02000 | 213.8 | .7 | 33.626 |
| 1.2400 | .02000 | 213.9 | .1 | 5.6114 |
| 1.2600 | .02000 | 213.8 | -.0 | -1.4214 |
| 1.2800 | .02000 | 214.4 | .5 | 26.007 |
| 1.3000 | .02000 | 215.4 | 1.1 | 53.159 |
| 1.3200 | .02000 | 216.2 | .8 | 41.461 |
| 1.3400 | .02000 | 216.7 | .5 | 23.105 |
| 1.3600 | .02000 | 216.7 | -.0 | -1.78430 |
| 1.3800 | .02000 | 217.0 | .4 | 17.590 |
| 1.4000 | .02000 | 218.1 | 1.1 | 54.640 |
| 1.4200 | .02000 | 219.0 | .8 | 41.780 |
| 1.4400 | .02000 | 219.3 | .4 | 18.136 |
| 1.4600 | .02000 | 219.2 | -.2 | -7.6225 |
| 1.4800 | .02000 | 219.6 | .4 | 18.926 |
| 1.5000 | .02000 | 220.3 | .7 | 36.559 |
| 1.5200 | .02000 | 221.2 | .9 | 43.152 |
| 1.5400 | .02000 | 221.6 | .4 | 22.306 |
| 1.5600 | .02000 | 221.5 | -.1 | -5.7388 |
| 1.5800 | .02000 | 221.8 | .3 | 14.743 |
| 1.6000 | .02000 | 222.7 | 1.0 | 48.056 |
| 1.6200 | .02000 | 223.5 | .8 | 37.849 |
| 1.6400 | .02000 | 224.0 | .5 | 25.768 |
| 1.6600 | .02000 | 223.8 | -.2 | -8.9363 |
| 1.6800 | .02000 | 223.9 | .1 | 3.6835 |
| 1.7000 | .02000 | 224.8 | .9 | 44.193 |

1046PM The titration was temporarily halted and a 0.02 ml addition was made of the titrant. Parameter 2 was changed to 6090 and the titration continued.

| | | | | |
|--------|--------|-------|-----|---------|
| .00000 | .00000 | 223.6 | | |
| .05000 | .05000 | 223.4 | -.1 | -2.9486 |
| .10000 | .05000 | 223.7 | .3 | 6.6281 |
| .15000 | .05000 | 224.4 | .6 | 12.627 |
| .20000 | .05000 | 224.6 | .3 | 5.2615 |
| .25000 | .05000 | 224.3 | -.4 | -7.5769 |
| .30000 | .05000 | 223.8 | -.4 | -8.5196 |
| .35000 | .05000 | 224.4 | .5 | 10.731 |
| .40000 | .05000 | 225.0 | .6 | 12.942 |
| .45000 | .05000 | 225.6 | .6 | 11.162 |
| .50000 | .05000 | 225.4 | -.1 | -2.6334 |

1105PM The HCl titration was finished.
Titration time: 144 minutes.

The titrant was changed to 0.101 M NaOH.

| VOLUME | INCREMENT | SIGNAL | S.CHANGE | DERIVATIVE |
|--------|-----------|--------|----------|------------|
| mL | mL | mV | mV | mV/mL |
| .00000 | .00000 | 229.5 | | |
| .05000 | .05000 | 229.5 | -.0 | -4.2236 |
| .10000 | .05000 | 228.9 | -.6 | -11.685 |
| .15000 | .05000 | 228.3 | -.7 | -13.261 |
| .20000 | .05000 | 227.9 | -.4 | -7.8925 |
| .25000 | .05000 | 227.8 | -.0 | -8.9539 |
| .30000 | .05000 | 227.5 | -.3 | -6.4664 |
| .35000 | .05000 | 226.5 | -1.0 | -19.072 |
| .40000 | .05000 | 225.6 | -1.0 | -19.589 |

1139PM Parameter 2 was changed to 5090 and the titration continued.

| mL | mL | mV | mV | mV/mL |
|---------|--------|-------|-----|---------|
| .00000 | .00000 | 225.5 | | |
| .02000 | .02000 | 225.6 | .1 | 6.0417 |
| .04000 | .02000 | 225.1 | -.5 | -25.251 |
| .06000 | .02000 | 224.7 | -.4 | -18.953 |
| .08000 | .02000 | 224.7 | -.0 | -1.8471 |
| .10000 | .02000 | 225.4 | .7 | 35.534 |
| .12000 | .02000 | 225.8 | .4 | 18.239 |
| .14000 | .02000 | 225.6 | -.2 | -8.5068 |
| .16000 | .02000 | 225.1 | -.5 | -25.790 |
| .18000 | .02000 | 224.7 | -.3 | -17.103 |
| .20000 | .02000 | 224.9 | .2 | 9.4643 |
| .22000 | .02000 | 225.1 | .2 | 10.797 |
| .24000 | .02000 | 224.9 | -.2 | -10.001 |
| .26000 | .02000 | 224.3 | -.6 | -31.307 |
| .28000 | .02000 | 224.2 | -.1 | -6.8398 |
| .30000 | .02000 | 224.7 | .5 | 23.935 |
| .32000 | .02000 | 225.3 | .6 | 29.738 |
| .34000 | .02000 | 225.2 | -.1 | -2.6360 |
| .36000 | .02000 | 224.7 | -.5 | -23.943 |
| .38000 | .02000 | 224.6 | -.1 | -4.4792 |
| .40000 | .02000 | 224.8 | .1 | 6.3240 |
| .42000 | .02000 | 225.1 | .3 | 16.581 |
| .44000 | .02000 | 225.0 | -.1 | -6.0577 |
| .46000 | .02000 | 224.3 | -.6 | -31.570 |
| .48000 | .02000 | 224.0 | -.3 | -16.577 |
| .50000 | .02000 | 224.0 | -.0 | -2.3651 |
| .52000 | .02000 | 224.5 | .6 | 29.202 |
| .54000 | .02000 | 224.6 | .1 | 4.2015 |
| .56000 | .02000 | 224.1 | -.5 | -26.037 |
| .58000 | .02000 | 223.8 | -.3 | -16.841 |
| .60000 | .02000 | 223.9 | .2 | 7.6347 |
| .62000 | .02000 | 224.2 | .3 | 14.985 |
| .64000 | .02000 | 224.3 | .1 | 5.7976 |
| .66000 | .02000 | 223.8 | -.5 | -25.784 |
| .68000 | .02000 | 223.4 | -.4 | -18.935 |
| .70000 | .02000 | 223.3 | -.2 | -8.1429 |
| .72000 | .02000 | 223.7 | .4 | 21.290 |
| .74000 | .02000 | 223.9 | .2 | 9.9938 |
| .76000 | .02000 | 223.4 | -.5 | -23.663 |
| .78000 | .02000 | 223.1 | -.3 | -16.828 |
| .80000 | .02000 | 223.0 | -.1 | -6.3110 |
| .82000 | .02000 | 223.3 | .3 | 16.564 |
| .84000 | .02000 | 223.5 | .2 | 9.2072 |
| .86000 | .02000 | 223.2 | -.3 | -14.204 |
| .88000 | .02000 | 222.7 | -.6 | -27.601 |
| .90000 | .02000 | 222.5 | -.2 | -7.6279 |
| .92000 | .02000 | 222.8 | .3 | 13.936 |
| .94000 | .02000 | 223.0 | .2 | 8.9417 |
| .96000 | .02000 | 222.7 | -.3 | -15.250 |
| .98000 | .02000 | 222.1 | -.6 | -27.853 |
| 1.00000 | .02000 | 221.7 | -.4 | -21.811 |
| 1.02000 | .02000 | 221.4 | -.3 | -14.978 |
| 1.04000 | .02000 | 221.0 | -.4 | -19.430 |

1300PM Parameter 2 was changed to 6090 and the titration continued.

| VOLUME | INCREMENT | SIGNAL | S.CHANGE | DERIVATIVE |
|--------|-----------|--------|----------|------------|
| mL | mL | mV | mV | mV/mL |
| .00000 | .00000 | 218.3 | | |
| .05000 | .05000 | 216.8 | -1.5 | -29.956 |
| .10000 | .05000 | 215.9 | -1.0 | -19.217 |
| .15000 | .05000 | 215.2 | -.6 | -12.916 |
| .20000 | .05000 | 214.1 | -1.1 | -22.668 |
| .25000 | .05000 | 212.4 | -1.7 | -33.037 |
| .30000 | .05000 | 210.8 | -1.6 | -32.153 |
| .35000 | .05000 | 209.7 | -1.1 | -22.032 |
| .40000 | .05000 | 208.8 | -.9 | -18.455 |
| .45000 | .05000 | 207.6 | -1.2 | -23.273 |
| .50000 | .05000 | 205.8 | -1.9 | -37.388 |
| .55000 | .05000 | 203.8 | -2.0 | -39.554 |
| .60000 | .05000 | 202.2 | -1.6 | -32.519 |
| .65000 | .05000 | 201.0 | -1.1 | -22.503 |
| .70000 | .05000 | 199.3 | -1.7 | -33.997 |
| .75000 | .05000 | 196.9 | -2.4 | -47.750 |
| .80000 | .05000 | 194.3 | -2.6 | -52.808 |
| .85000 | .05000 | 191.8 | -2.5 | -50.541 |

1330PM Parameter 2 was changed to 5090 & the titration continued.

| VOLUME mL | INCREMENT mL | SIGNAL mV | S. CHANGE mV | DERIVATIVE mV/mL |
|--------------|-----------------|--------------|-----------------|---------------------|
| .00000 | .00000 | 189.3 | | |
| .02000 | .02000 | 188.4 | -0.9 | -45.083 |
| .04000 | .02000 | 186.7 | -1.7 | -86.329 |
| .06000 | .02000 | 185.2 | -1.5 | -76.154 |
| .08000 | .02000 | 183.9 | -1.3 | -66.428 |
| .10000 | .02000 | 182.6 | -1.3 | -64.300 |
| .12000 | .02000 | 180.9 | -1.7 | -84.535 |
| .14000 | .02000 | 178.6 | -2.3 | -115.59 |
| .16000 | .02000 | 176.1 | -2.4 | -120.94 |
| .18000 | .02000 | 173.5 | -2.6 | -130.64 |
| .20000 | .02000 | 170.9 | -2.7 | -134.03 |
| .22000 | .02000 | 167.3 | -3.5 | -176.99 |
| .24000 | .02000 | 162.4 | -4.9 | -246.23 |
| .26000 | .02000 | 156.2 | -6.2 | -308.47 |
| .28000 | .02000 | 147.8 | -8.4 | -420.11 |
| .30000 | .02000 | 133.9 | -13.9 | -693.78 |

1355PM NaOH titration ended. Actual titration time: 140 minutes.

7/27/92 PM Taking CO₂ out of solution

Boiling doesn't seem to be removing enough CO₂ from water used in solutions which should be CO₂-free, & N₂ purge also gives uneven results.

An acrylic column previously used for Drierite (anhydrous CaSO₄) was filled with Drierite (II) lot # 0187, and this was connected between a N₂-gas tank & a glass tube with frit.

1230PM A 1x10⁻³M NaCl solution was made by weighing out 0.0051g NaCl (lot # 9/27/63) & dissolving it with ultrapure water in a 100 mL volumetric flask.

An Orion 290 pH meter & glass electrode were used to measure the solution pH. The meter/electrode were calibrated using Fisher pH buffers, pH = 4.0, 5.0, & 7.0.

The solution was stirred at all times.

1303PM Starting pH: 5.35 @ 20.4 °C
N₂ purge was then started.

| Time | pH | Temp °C |
|------|------|---------|
| 1307 | 5.51 | 20.5 |
| 1312 | 5.71 | " |
| 1318 | 6.00 | 21.1 |
| 1323 | 6.23 | 20.6 |
| 1328 | 6.43 | 20.7 |
| 1333 | 6.81 | " |
| 1338 | 6.97 | " |
| 1343 | 7.07 | 20.8 |
| 1353 | 7.20 | " |
| 1403 | 7.07 | 20.9 |
| 1414 | 7.05 | " |

7/28/92 PM more taking CO₂ out of solution

A glass tower with 0.5M NaOH (10g NaOH pellets, lot # in 500mL ultrapure water) was attached via tetlar tubing to another glass tower with boiled ultrapure water. Another 100 mL solution of 1x10⁻³M NaCl solution was purged as yesterday, with the two glass towers substituted for the acrylic column.

1326PM Starting pH: 5.63 @ 20.8 °C (N₂ purge started)

| Time | pH | Temp °C |
|------|------|---------|
| 1343 | 6.01 | 20.7 |
| 1354 | 6.30 | " |
| 1411 | 6.44 | " |
| 1422 | 6.65 | " |
| 1432 | 6.83 | 20.6 |
| 1437 | 7.01 | 20.7 |
| 1442 | 6.89 | " |
| 1447 | 6.88 | " |

7/30/92 TD

TESTED THE ^{TD} STABILITY OF THE NEW
^{1/20} ~~DATA~~ ^{MICRO} PH ELECTRODE BY RECORDING ITS
 READING OF A pH 5.00 BUFFER FOR
 4.5 HOURS.

CALIBRATION

CH1-PH
 PH = 4.00
 168.9mV 20.10
 P2 PH = 7.00
 -3.9mV 20.10
 SLP = 99.8%
 ISO = 7.000
 09:29 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.10
 09:30 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.10
 09:32 07-30-92

CH1-PH
 PH = 5.00
 111.4mV 20.20
 09:42 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.20
 09:52 07-30-92

CH1-PH
 PH = 5.00
 111.4mV 20.30
 10:02 07-30-92

CH1-PH
 PH = 5.00
 111.3mV 20.30
 10:12 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.40
 10:22 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.40
 10:32 07-30-92

CH1-PH
 PH = 5.00
 111.5mV 20.50
 10:42 07-30-92

CH1-PH
 PH = 5.00
 111.7mV 20.50
 10:52 07-30-92

CH1-PH
 PH = 5.00
 111.7mV 20.60
 11:02 07-30-92

CH1-PH
 PH = 5.00
 111.8mV 20.70
 11:12 07-30-92

CH1-PH
 PH = 4.99
 111.9mV 20.70
 11:22 07-30-92

CH1-PH
 PH = 4.99
 112.0mV 20.80
 11:32 07-30-92

CH1-PH
 PH = 4.99
 112.1mV 20.90
 11:42 07-30-92

CH1-PH
 PH = 4.99
 112.1mV 20.90
 11:52 07-30-92

CH1-PH
 PH = 4.99
 112.4mV 21.00
 12:02 07-30-92

CH1-PH
 PH = 4.99
 112.4mV 21.10
 12:12 07-30-92

CH1-PH
 PH = 4.99
 112.5mV 21.10
 12:22 07-30-92

CH1-PH
 PH = 4.99
 112.6mV 21.20
 12:32 07-30-92

CH1-PH
 PH = 4.99
 112.6mV 21.20
 12:42 07-30-92

CH1-PH
 PH = 4.98
 112.8mV 21.30
 12:52 07-30-92

CH1-PH
 PH = 4.98
 112.9mV 21.40
 13:02 07-30-92

CH1-PH
 PH = 4.98
 113.0mV 21.40
 13:12 07-30-92

CH1-PH
 PH = 4.98
 113.1mV 21.50
 13:21 07-30-92

CH1-PH
 PH = 4.98
 113.3mV 21.60
 13:31 07-30-92

CH1-PH
 PH = 4.97
 113.4mV 21.80
 13:41 07-30-92

CH1-PH
 PH = 4.97
 113.6mV 21.70
 13:51 07-30-92

CH1-PH
 PH = 4.97
 113.7mV 21.70
 14:01 07-30-92

CH1-PH
 PH = 4.97
 114.0mV 21.80
 14:11 07-30-92

CH1-PH
 PH = 4.96
 114.2mV 21.80
 14:21 07-30-92

CH1-PH
 PH = 4.96
 114.4mV 21.90
 14:31 07-30-92

CH1-PH
 PH = 4.96
 114.6mV 21.90
 14:41 07-30-92

CH1-PH
 PH = 4.95
 114.7mV 21.90
 14:51 07-30-92

CH1-PH
 PH = 4.95
 115.0mV 22.00
 15:01 07-30-92

4 AUG 1992 TD

Prepared 5L of 0.1M NaNO₃ by dissolving 8.499gm NaNO₃
 in 1000g of H₂O; To make 5 L, this was repeated 5 times in
 5 different 1000 mL PP bottles. The bottles had been previously
 acid cleaned and dried and labeled 0.1M NaNO₃
 TD 4 AUG 1992.

The lot # of the NaNO₃ is 7808 KIDE.

| WT. OF NaNO ₃ USED (gm) | WT. WATER USED (gm) |
|------------------------------------|---------------------|
| bottle #1 8.4994 | 1000.0 |
| bottle #2 8.4990 | 1000.0 |
| bottle #3 8.4995 | 1000.0 |
| bottle #4 8.4988 | 1000.0 |
| bottle #5 8.4989 | 1000.0 |

8/4/92 POT More CO₂-removal Procedures

If the N₂ tank is used to simultaneously purge
 a NaOH/H₂O system + an Ascarite system, the
 gas pressure is not sufficient to remove the CO₂
 efficiently; when this was tried yesterday for 2 1/2
 hours, the results were

1x10⁻³ NaCl solution attached to NaOH (0.5M) +
 deionized, N₂ purged H₂O towers,

pH_{init} = 5.47 @ 10:42, 27.3°C
 Final = 5.79 12:50 28.5°C

1x10⁻³ NaCl solution attached to Ascarite II column

pH_{init} = 5.40 @ 10:42, 20.1°C
 5.40 @ 12:52, 20.7°C

0726PM 500 mL of ultrapure water previously equilibrated with atmospheric CO_2 was degassed by boiling on a hot plate for 30 minutes⁺. The boiled water was allowed to cool for ~1 minute to reduce steam, then was poured into a glass tower + purged with N_2 gas.

200 mL of $1 \times 10^{-3} \text{ M NaCl}$ solution was prepared by weighing out 0.0117 g NaCl (wt used:

0.0124 g, lot # 912763)

+ dissolving it with pre-equilibrated ultrapure water in a 200 mL volumetric flask. The solution was brought to volume with pre-equilibrated ultrapure water.

The solution was stirred to mix + divided evenly between two ^{PM 8/11/92} 250 mL PP bottles labeled " $1 \times 10^{-3} \text{ M NaCl}$ ", + stir bars put in the bottles.

NaOH solution (0.5M) prepared 7/28/92 was poured into a second glass tower, this tower connected to the N_2 gas tank, the exit tube of the tower connected to the water tower, a glass tube with frit attached to the water tower.

0922PM The 920 Orion pH meter / glass electrode was calibrated using Fisher pH buffers 5.0, 7.0, + 9.0, lot #5 913284-24, 910943-24, + 910944-24.

0932PM The pH of one of the $1 \times 10^{-3} \text{ M NaCl}$ solutions was measured:

$\text{pH}_{\text{init}} = 5.54 @ 20.0^\circ \text{C}$

The tube with frit was inserted into the solution bottle, the N_2 purge started w gauge reading ~3.8 psi, + the pH monitored while the solution was purged + stirred.

| time | pH | temp $^\circ \text{C}$ |
|------|------|------------------------|
| 0937 | 5.65 | 20.0 |
| 0939 | 5.75 | " |
| 0940 | 5.85 | 20.1 |
| 0943 | 5.96 | " |
| 0950 | 6.06 | " |
| 1000 | " | " |
| 1010 | 6.05 | 20.2 |
| 1030 | 6.03 | " |
| 1100 | 6.00 | 20.3 |
| 1110 | " | " |

The purge was stopped + the frit repeatedly rinsed with ultrapure water.

1155PM ~100 mL of pre-equilibrated ultrapure water was poured into a 125 mL PP bottle + labeled, a stir bar added, + the initial pH of the water measured:

$\text{pH}_{\text{init}} = 5.63 @ 20.4^\circ \text{C}$

The water was purged + monitored in the same manner as the $1 \times 10^{-3} \text{ M NaCl}$ solution, with the gauge reading ~3.8 psi.

| time | pH | temp °C |
|------|------|---------|
| 1121 | 5.77 | 20.4 |
| 1130 | 6.32 | " |
| 1140 | 6.38 | 20.5 |
| 1150 | 6.32 | " |
| 1200 | 6.39 | " |
| 1210 | 6.31 | 20.6 |
| 1250 | 6.30 | 20.7 |
| 1300 | 6.30 | 20.8 |
| 1320 | 6.29 | 20.9 |

1340 PM The pH meter / electrode was recalibrated. The unpurged 1×10^{-3} NaCl solution's pH was measured.

1405 PM pH init: 5.55 @ 21.0 °C

The NaOH tower was disconnected from the N_2 tank, + the Ascarite column connected in its place. A glass tube with frit was connected to the column outlet, + the tube + frit placed in the unpurged solution bottle. The N_2 purge was started with the gauge on the N_2 tank not even moved.

| time | pH | temp °C |
|------|------|---------|
| 1407 | 5.67 | 21.0 |
| " | 5.79 | " |
| 1408 | 5.91 | " |
| 1409 | 6.01 | 21.1 |
| 1410 | 6.06 | " |
| 1420 | 6.03 | " |
| 1440 | 5.98 | 21.2 |
| 1500 | 5.96 | " |
| 1520 | 5.93 | 21.3 |
| 1540 | 5.91 | " |
| 1550 | 5.89 | 21.4 |

8/5/92 TD

Measured the Na^+ conc in the solutions (0.1M $NaNO_3$) prepared yesterday.

| | Na^+ conc (ppm) |
|----------|-------------------|
| bottle 1 | 219 |
| 2 | 220 |
| 3 | 222 |
| 4 | 223 |
| 5 | 222 |

there is a 1:10 dilution factor.

TD 0330

Prepared 1000 ml 1.0M HNO_3 by Diluting 63ml concentrated HNO_3 (lot # 910692) to 1000 ml with Ultrapure H_2O in a Volumetric flask. The solution was placed in a clean, dry 1000ml PP bottle and labeled.

TD 0400

Prepared 1000ml 0.1M HNO_3 by using a DILUT-IT concentrate (lot # 244099) according to directions on the package. This was stored in a clean, dry 1000ml PP bottle labeled with the contents, date prepared and initials of preparer.

TD 6 AUG 1992

Prepared 2000ml 0.02M HNO_3 by diluting 40ml 1.0M HNO_3 (prepared above) to 2000 ml in a Volumetric flask using Ultrapure H_2O . Prepared 500ml 0.01M HNO_3 by diluting 50ml 0.1M HNO_3 (prepared above) to 500ml in a volumetric flask using Ultrapure H_2O .

All the HNO_3 solutions are for the next set of U Kinetics and Sorption experiments.

TO slr/az
21 AUG 1992 0900 HRS

PREPARED 6 L OF 0.1 M NaNO₃ by dissolving
8.499 g NaNO₃ in 1000 kg of H₂O 6 times in
different 1000 ml PP bottles.

| Bottle # | Amt. USED (g) |
|----------|---------------|
| 1 | 8.4987 |
| 2 | 8.4987 |
| 3 | 8.4990 |
| 4 | 8.4988 |
| 5 | 8.4991 |
| 6 | 8.4995 |

~~0930 16 Sept 1992 TD~~

KINETICS EXPERIMENT K3-1

OBJECTIVE

To determine the time it takes to reach sorption equilibrium
between uranium (²³³U) solutions and clinoptilolite.

16 Sept

16 Sept 1992 Todd Dietrich 0930

KINETICS EXPERIMENT K3

(Initial pH=6.0, 7.0; 0.1 M NaNO₃; equil. with atm. CO₂)

WRITTEN BY: R. T. PABALAN

DATE WRITTEN: July 30, 1992

REVISION NO.:

REVISION DATE:

OBJECTIVE:

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

NOTES:

Previous experiment A-II-3 (initial ΣU=476 ppb) indicated that maximum sorption of uranium on clinoptilolite is obtained at equilibrium pH=6.5. A-II-3 solutions which had initial pH values of 4.0 to 6.0 ended up with equilibrium pH in the vicinity of 6.5. However, later experiments B-IV (in 0.1 M NaNO₃) and B-V (no NaNO₃) indicated that the pH change upon addition of clinoptilolite to the uranium solution is mainly due to ion exchange between Na⁺ initially in the solid phase and H⁺ in solution. The B-IV experiments demonstrated that this ion exchange process is smaller where there is a relatively high concentration of Na⁺ initially in solution (e.g., 0.1 M NaNO₃) due to mass action constraints. Thus, in contrast to the kinetic experiments K1 and K2, this set of kinetic runs will be constrained to have initial pH of 6.0 or 7.0. The ionic strength will be approximately constant at 0.1 M NaNO₃. The initial pH will be adjusted from that of the 50 ppb stock solution by addition of NaHCO₃, the amount of which is estimated using EQ3 calculations.

K3-1: Solution K3-1 (2 bottles)

- Initial ΣU = 50 ppb
- Initial ΣNa = 0.1+x (where 0.1 is from NaNO₃ and x is from NaHCO₃)
- Initial pH = 6.0
- Initial solution mass = 1000 g
- Wt. zeolite used = 2.0 g of CDV*100/200*UC*WA*HL*CPT*Naf
- Equilibrium with atmospheric CO₂(g) (pCO₂ = 10^{-3.5} bar)

K3-1-A: In a pre-cleaned 4 liter plastic bottle, prepare 3100 g of 50 ppb U solution by diluting 310 g of a 500 ppb ²³³U stock solution (in 0.1 M NaNO₃ matrix; prepared previously from commercial 50 ppm ²³³U spike) to a total of 3100 g by carefully taring 0.1 M NaNO₃ solution into the plastic bottle on a Mettler 4600 balance.

K3-1-B: Transfer three 1000 g aliquots of the 50 ppb solution into three 1000-ml polypropylene (PP) bottles labeled K3-1*A, K3-1*B, and K3-1*C. Transfer the remaining 50 ppb solution into a 125-ml PP bottle labeled K3-1*IU.

Take 2 10-ml aliquots of solution K3-1*IU using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-labeled [e.g., K3-1*IU*a (or b)] and pre-weighed 50-ml centrifuge tubes. Reweigh each tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 µl of 1.0 M HNO₃ solution for later analysis with alpha-spectrometry.

K3-1-C: Measure and record the initial pH of solutions K3-1*A, K3-1*B and K3-1*C (-4.2 calculated using EQ3) using a combination glass Microelectrode. The automatic temperature compensator (ATC) probe should be immersed in water in a separate container. Carefully add -0.0072 g of NaHCO₃ to each 1000 ml ²³³U solution. Cover the bottle with a porous material (e.g., kimwipe) and keep the solution agitated on a New Brunswick gyratory shaker to equilibrate it with atmospheric CO₂(g).

Measure the pH of the solutions periodically until a constant value is reached. This step may take at least 10 days. Then proceed to the next step.

K3-1-D: Take 2 10-ml aliquots of K3-1*A, K3-1*B and K3-1*C using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-labeled [e.g., K3-1*A*11 (or 12)] and pre-weighed 50-ml centrifuge tubes. Reweigh each tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO₃ solution for later analysis with alpha-spectrometry.

Then into bottles K3-1*A and K3-1*B (not to K3-1*C), add 2.0 g of Na₂ clinoptilolite which was previously tared (quickly) onto weighing paper. Record the time and room temperature. Solution K3-1*C is the control experiment to determine uranium losses to the container walls. Keep the solutions agitated on a gyratory shaker (note the approximate rpm of the shaker on the notebook).

K3-1-E: At Δ time approximating the values listed below, take one 10-ml sample from K3-1*A, K3-1*B, and K3-1*C with an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-weighed 50-ml centrifuge tubes. Reweigh each centrifuge tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO₃ solution for later analysis with alpha-spectrometry. Label the samples [e.g., K3-1*A**i*a, where *i* is the sampling time number (1,2,3,...)].

Take two 10-ml samples from K3-1*A, *B, *C every third sampling time. Label the second sample [e.g., K3-1*A**i*b].

In addition, measure the pH of each solution. Record the pH and solution temperature.

Use the following Δ time (hours): 2.4; 22; 46; 70; 94; 142; 190; 238; 310; 382; 454. These may be changed as deemed necessary.

K3-1-F: After the last samples have been taken, take 2 10-ml aliquots of solution K3-1*IU using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-weighed 50-ml centrifuge tubes. Reweigh the tubes after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO₃ for later analysis using alpha-spectrometry. Label the bottles K3-1*FU*a (or b).

K3-1-G: Measure the U concentrations using alpha-spectrometry.

K3-1-A

AMT SOLIDS USED 310.01 g
FINAL SOLUTION WT 310.0 g

~~*C 0.0015 g~~ ID 10/500⁺ 1992

K3-1-B

WT. OF SOLUTION IN K3-1*A 1000.0 g
* B 1000.0 g
* C 1000.0 g

WT OF SAMPLE K3-1*IU*a 10.0635 g
* b 10.0812 g

K3-1-C

pH of SOLUTION K3-1*A 4.12
* B 4.12
* C 4.12

AMT NaHCO₃ ADDED TO K3-1*A 0.0073 g Lot # 897186A
* B 0.0072 g
* C 0.0075 g

The NaNO₃ solution used for the dilution was prepared 21 Aug 1992
p. AC-07-150.

The dilution of the ²³³U spike can be found in notebook
031 (Radiochemistry/Bret Leslie) on pages 111-112. The dilution
was done by Dr. Bret Leslie.

The HNO₃ was prepared on 5 Aug 1992, see pg GC-07-149.

16 Sept 1992 TO 1200

K3-2: Solution K3-2 (2 bottles)

- Initial $\Sigma U = 50$ ppb
- Initial $\Sigma Na = 0.1+x$ (where 0.1 is from $NaNO_3$ and x is from $NaHCO_3$)
- Initial pH = 7.0
- Initial solution mass = 1000 g
- Wt. zeolite used = 2.0 g of CDV*100/200*UC*WA*HL*CPT*Naf
- Equilibrium with atmospheric $CO_2(g)$ ($pCO_2 = 10^{-3.5}$ bar)

THE PROCEDURES ARE THE SAME AS IN K3-1, ONLY A DIFFERENT AMOUNT OF $NaHCO_3$ IS ADDED TO ADJUST THE PH TO 7.0.

K3-2-A: In a pre-cleaned 4 liter plastic bottle, prepare 3100 g of 50 ppb U solution by diluting 310 g of a 500 ppb ^{233}U stock solution (in 0.1 M $NaNO_3$ matrix; prepared previously from commercial 50 ppm ^{233}U spike) to a total of 3100 g by carefully taring 0.1 M $NaNO_3$ solution into the plastic bottle on a Mettler 4600 balance.

K3-2-B: Transfer three 1000 g aliquots of the 50 ppb solution into three 1000-ml polypropylene (PP) bottles labeled K3-2*A, K3-2*B, and K3-2*C. Transfer the remaining 50 ppb solution into a 125-ml PP bottle labeled K3-2*IU.

Take 2 10-ml aliquots of solution K3-2*IU using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-labeled [e.g., K3-2*IU*a (or b)] and pre-weighed 50-ml centrifuge tubes. Reweigh each tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO_3 solution for later analysis with alpha-spectrometry.

K3-2-C: Measure and record the initial pH of solutions K3-2*A, K3-2*B and K3-2*C (~4.2 calculated using EQ3) using a combination glass Microelectrode. The automatic temperature compensator (ATC) probe should be immersed in water in a separate container. Carefully add ~0.0127 g of $NaHCO_3$ to each 1000 ml ^{233}U solution. Cover the bottle with a porous material (e.g., kimwipe) and keep the solution agitated on a New Brunswick gyratory shaker to equilibrate it with atmospheric $CO_2(g)$.

Measure the pH of the solutions periodically until a constant value is reached. This step may take at least 10 days. Then procede to the next step.

K3-2-D: Take 2 10-ml aliquots of K3-2*A, K3-2*B and K3-2*C using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-labeled [e.g., K3-2*A*I1 (or I2)] and pre-weighed 50-ml centrifuge tubes. Reweigh each tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO_3 solution for later analysis with alpha-spectrometry.

Then into bottles K3-2*A and K3-2*B (not to K3-2*C), add 2.0 g of Na-clinoptilolite which was previously tared (quickly) onto weighing paper. Record the time

and room temperature. Solution K3-2*C is the control experiment to determine uranium losses to the container walls. Keep the solutions agitated on a gyratory shaker (note the approximate rpm of the shaker on the notebook).

K3-2-E: At Δ time approximating the values listed below, take one 10-ml sample from K3-2*A, K3-2*B, and K3-2*C with an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-weighed 50-ml centrifuge tubes. Reweigh each centrifuge tube after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO_3 solution for later analysis with alpha-spectrometry. Label the samples [e.g., K3-2*A*ia, where i is the sampling time number (1,2,3...)].

Take two 10-ml samples from K3-2*A, *B, *C every third sampling time. Label the second sample [e.g., K3-2*A*ib].

In addition, measure the pH of each solution. Record the pH and solution temperature.

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

K3-2-F: After the last samples have been taken, take 2 10-ml aliquots of solution K3-2*IU using an Eppendorf Maxipettor (pre-rinsed with the solution being sampled), transfer into pre-weighed 50-ml centrifuge tubes. Reweigh the tubes after addition of the sample. Add to each sample using an Eppendorf micropipet 500 μ l of 1.0 M HNO_3 for later analysis using alpha-spectrometry. Label the bottles K3-2*FU*a (or b).

K3-2-G: Measure the U concentrations using alpha-spectrometry.

K3-2-A

WT 500ppb Used 310.02g

FINAL SOLUTION WT 3100.0g

K3-2-B

WT OF SOLUTION IN K3-2*A 1000.0g

*B 1000.0g

*C 1000.0g

WT OF SAMPLE K3-2*IU*a 10.0887g

*b 10.0751g

K3-2-C

pH OF SOLUTION K3-2*A 4.13

*B 4.14

*C 4.14

WT OF $NaHCO_3$ ADDED TO K3-2*A 0.0128g

*B 0.0130g

*C 0.0129g

Lot # 897186A

The spike, NaNO_3 solution, and HNO_3 solution are the same as that used in K3-1 ^{9/16/92} and (see p. 153).

The bottles K3-1*A, B, and C as well as K3-2*A, B, C were placed on a New Brunswick gyratory shaker set to ~ 125 rpm

9/17/92 1000 MTP

Prepared 5 batches of 1-Liter 0.1 M NaNO_3 solution. Used NaNO_3 reagent Lot # 78 0860

| | |
|------------|---------------------------|
| bottle # 1 | 8.499 gms NaNO_3 |
| 2 | 8.500 gms |
| 3 | 8.5018 gms |
| 4 | 8.488 gms |
| 5 | 8.4994 gms |

MTP

9/17/92 MTP
1300 hrs

URANIUM SORPTION EXPERIMENT B-I:
Kd vs pH; Equilibrium with atmospheric pCO_2 ; Initial $\Sigma\text{U}=50$ ppb

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

DATE WRITTEN: August 3, 1992
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/ $^{\circ}\text{C}$ 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-I*IU)
 - 39 125-ml PP bottles (to contain experimental mixtures and control solutions)
 - 78 50-ml centrifuge tubes (to contain samples for U analysis)
 - 2 100-ml volumetric pipet (1 to prepare 50 ppb U solution and 1 to pipet 50 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 50 ppb U solution)
 - 3 glass droppers (for adjusting pH by addition of HNO_3)
 - weighing paper
 - 1 Eppendorf pipet (for taking 5-ml samples)
 - Na^+ -clinoptilolite (CDV*100/200*UC*WA*HL*CPT*Naf)
 - reagent grade NaHCO_3
 - 500 ppb U stock solution prepared from 50 ppm ^{233}U commercial spike
 - 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
 - ultrapure water

PROCEDURE:

Solution B-I (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 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) In a pre-cleaned 4 liter plastic bottle, prepare 4000 g of 50 ppb U solution by diluting 400 g of a 500 ppb stock solution (in 0.1 M NaNO_3 matrix; prepared previously from commercial 50 ppm ^{235}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-I* $p\text{Hi}$ [where i is the approximate initial pH of the solution (see below)], tare 100 g of the 50 ppb uranium solution.

Into each of 10 125-ml PP bottle labeled B-I-C* $p\text{Hi}$ [where i is 2, 4, 6, 8, or 9.5, representing the approximate initial pH of the solution], tare 100 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 125-ml PP bottle labeled B-I*IU. Take two 5-ml samples from B-I*IU with an Eppendorf pipet, transfer into pre-labeled [e.g., B-I-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-I* $p\text{Hi}$ and B-I-C* $p\text{Hi}$, where $i \leq 4.2$:

Measure and record the initial pH (~4.2, 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-I-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-I* $p\text{Hi}$ and B-I-C* $p\text{Hi}$, where $i > 4.2$:

Measure and record the initial pH (~4.2, based on EQ3 calculation). Tare onto weighing paper reagent grade NaHCO_3 in the amounts listed in Table B-I-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-I* $p\text{Hi}$ (not the B-I-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-I* $p\text{Hi}$ and B-I-C* $p\text{Hi}$ with an Eppendorf pipet, transfer into pre-labeled [e.g., B-I- $p\text{Hi}$ *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-I* $p\text{Hi}$ and B-I-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-I*IU)
- 39 125-ml PP bottles (to contain experimental mixtures and control solutions)
- 78 50-ml centrifuge tubes (to contain samples for U analysis)
- 2 100-ml volumetric pipet (1 to prepare 50 ppb U solution and 1 to pipet 50 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 50 ppb U solution)
- 3 glass droppers (for adjusting pH by addition of HNO_3)

2. Prepare:

- 500 ppb U stock solution prepared from 50 ppm ^{235}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

Table B-I-1. Amount of reagent grade HNO_3 or NaHCO_3 to add to 100 ml 0.1 M NaNO_3 solution containing 50 ppb U to result in pH values given in column-1. The amount of reagent to be added was estimated using EQ3 calculations and 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 | 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 (0.22 mL) | 0.1 |
| 4.00 | 3 (0.12 mL) | 0.1 |
| Solution pH | Amount of NaHCO_3 Needed, Grams | |
| 4.25 | 0.000101 | |
| 4.50 | 0.000351 | |
| 4.75 | 0.000492 | |
| 5.00 | 0.000574 | |
| 5.25 | 0.000624 | |
| 5.50 | 0.000658 | |
| 5.75 | 0.000687 | |
| 6.00 | 0.000721 | |
| 6.25 | 0.000772 | |
| 6.50 | 0.000858 | |
| 6.75 | 0.001006 | |
| 7.00 | 0.001270 | |
| 7.25 | 0.001738 | |
| 7.50 | 0.002573 | |
| 7.75 | 0.004072 | |

| Solution pH | Amount of NaHCO_3 Needed, Grams | |
|-------------|--|--|
| 8.00 | 0.006773 | |
| 8.25 | 0.011692 | |
| 8.50 | 0.020818 | |
| 8.75 | 0.038270 | |
| 9.00 | 0.073327 | |
| [9.25] | [0.149388] | |
| [9.50] | [0.335545] | |

9/18/92 MPM EXPT. B-I
 1500 hrs - Prepared 4000 g 65 ppm U solution as in step B-I(a).
 However, after almost taring ~3300 gms, overrange error occurred on Mettler 4600 balance. Thus, tared another bottle on Sartorius 3808 balance, and transferred all the solution from the first bottle. Made up to 4000 gms with 0.1M NaNO_3 . Used bottle #5 217, 486 prepared on 9/17/92 (AC-07-156).

9/18/92 TD 0800

EXPERIMENT K3-1

Measured pH in the ^{TD} ~~the~~ of the three solutions in this experiment using the microelectrode and the Orion 920A benchtop ^{TD} benchtop meter.

TD 9/18/92
 K3-1

| | pH |
|---------|------|
| K3-1 *A | 5.29 |
| *B | 6.03 |
| *C | 5.20 |

?? MP 9/18/92

0830 TD

EXPERIMENT K3-2

Remeasured the pH in the K3-2 solutions in the same manner as those above

| | pH |
|---------|------|
| K3-2 *A | 6.81 |
| *B | 6.72 |
| *C | 6.84 |

0900 RD

EXPERIMENT B-I

B-I-b

(39) 100 g aliquots were taken as described in the procedure. 2-5 ml samples of B-I-1H were taken and weighed.

| WT | SAMPLE | TP | g/g/g SAMPLE | ACID (0.02M HNO ₃) |
|----|-----------|----|--------------|--------------------------------|
| | B-I-1H *a | | 5.0455g | 5.0532g |
| | *b | | 5.0550g | 5.0207g |

B-I-c(1)

Calibrated droppers to be used in pH adjustments by counting drops to fill a 5 ml volumetric to the mark.

1.0M HNO₃

$$\frac{128 \text{ drops}}{5 \text{ ml}} = 0.0391 \frac{\text{ml}}{\text{drop}}$$

0.1M HNO₃

$$\frac{5 \text{ ml}}{125 \text{ drops}} = 0.04 \frac{\text{ml}}{\text{drop}}$$

B-I-C(2)

Began adding NaHCO₃ in the weight in table B-I-1 to the highest pH (most basic) solns. These will take the longest to equilibrate.

| | Amt NaHCO ₃ added (g) |
|-------------------|----------------------------------|
| B-I-C * pH 9.5 *a | 0.3356 |
| *b | 0.3360 |
| B-I * pH 9.00 | 0.0740 |
| B-I * pH 8.75 | 0.0384 |
| B-I * pH 8.50 | 0.0209 |
| B-I * pH 8.25 | 0.0125 |
| B-I * pH 8.00 | 0.0071 |
| B-I * pH 8.00 *a | 0.0068 |
| *b | 0.0069 |

9/18/92 R.T.P.
Procedure for Expt. B-II

URANIUM SORPTION EXPERIMENT B-II:

K_d vs pH; Equilibrium with atmospheric pCO₂; Initial ΣU=500 ppb

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

DATE WRITTEN: August 4, 1992
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)
- 34 125-ml PP bottles (to contain experimental mixtures and control solutions)
 - 70 50-ml centrifuge tubes (to contain samples for U analysis)
 - 1 100-ml volumetric pipet (to pipet 500 ppb U solution into PP bottles)
 - 1 5-ml volumetric pipet (to pipet 0.02 M HNO₃ into 50-ml centrifuge tubes)
 - 1 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*Na)
 - reagent grade NaHCO₃
 - 500 ppb U stock solution prepared from 50 ppm ²³³U commercial spike
 - 1000 ml stock solution of 1.0 M HNO₃
 - 1000 ml stock solution of 0.02 M HNO₃
 - ultrapure water

