308
Scientific Notebook # 370

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Uranyl adsorption onto clinoptilolite, montmorillonite: XAFS study

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CONTROLLED
COPY 370

Radionuclide Transport KTI (20.01402.871)

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Contents Investigator: Melissa Nugent mNugent
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Initial Entry 10/8/99 by Melissa Nugent

This laboratory notebook chronicles the investigations of radionuclide transport.

Melissa Nugent

This notebook is dedicated to investigating the roles of ion exchange and surface adsorption of the uranyl ion onto clinoptilolite and montmorillonite, for analysis of the solids by XAFS.
Uranyl adsorption onto these minerals is a potential mechanism for retadation of uranium and thus is important in considerations of radionuclide transport. Because the mechanism by which uranium may adsorb onto these minerals is different in different pH solutions, both adsorption mechanisms must be investigated, to understand their effectiveness, and stability. XAFS analysis provides insight into surface complexation and the stability of surface complexes.

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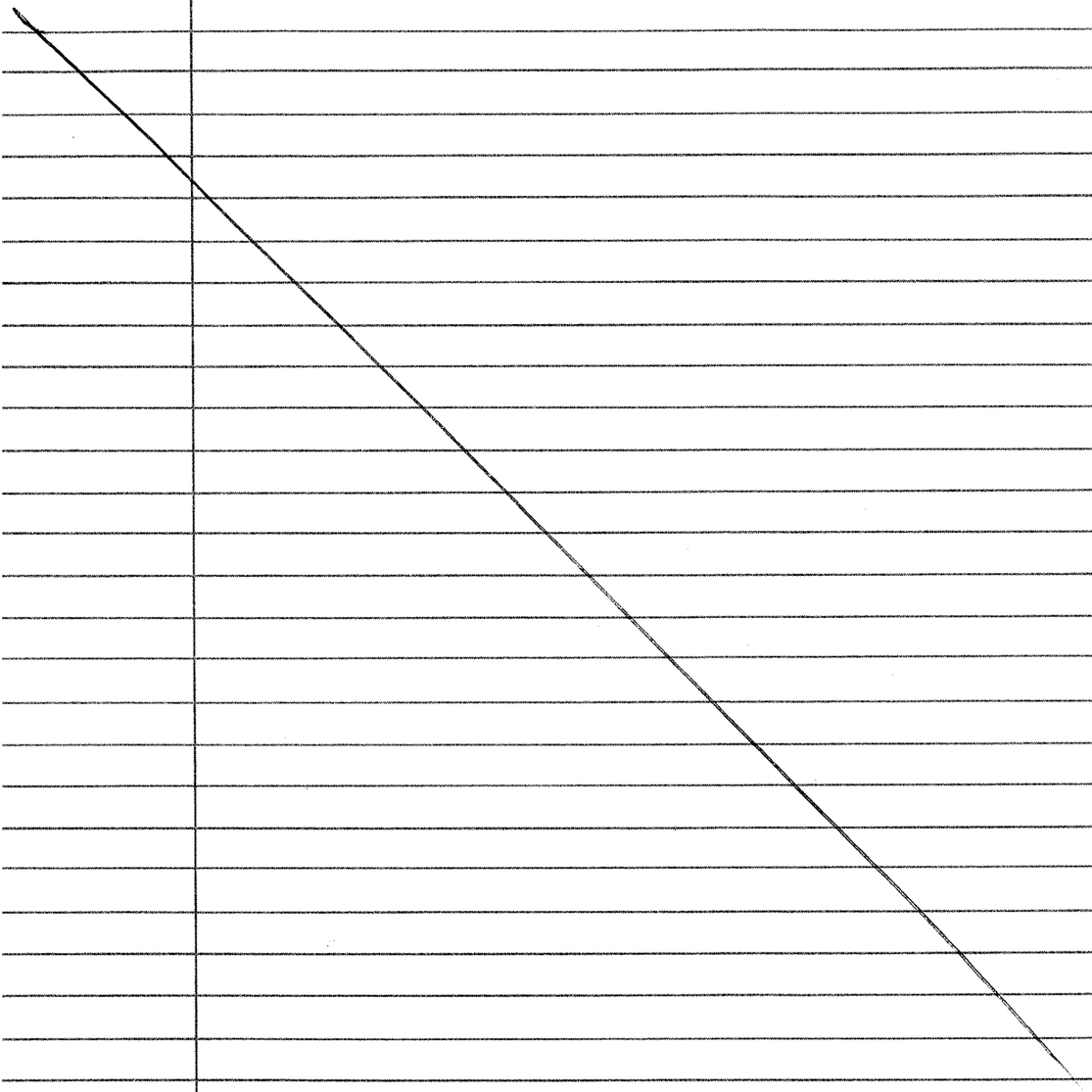
10/8/99



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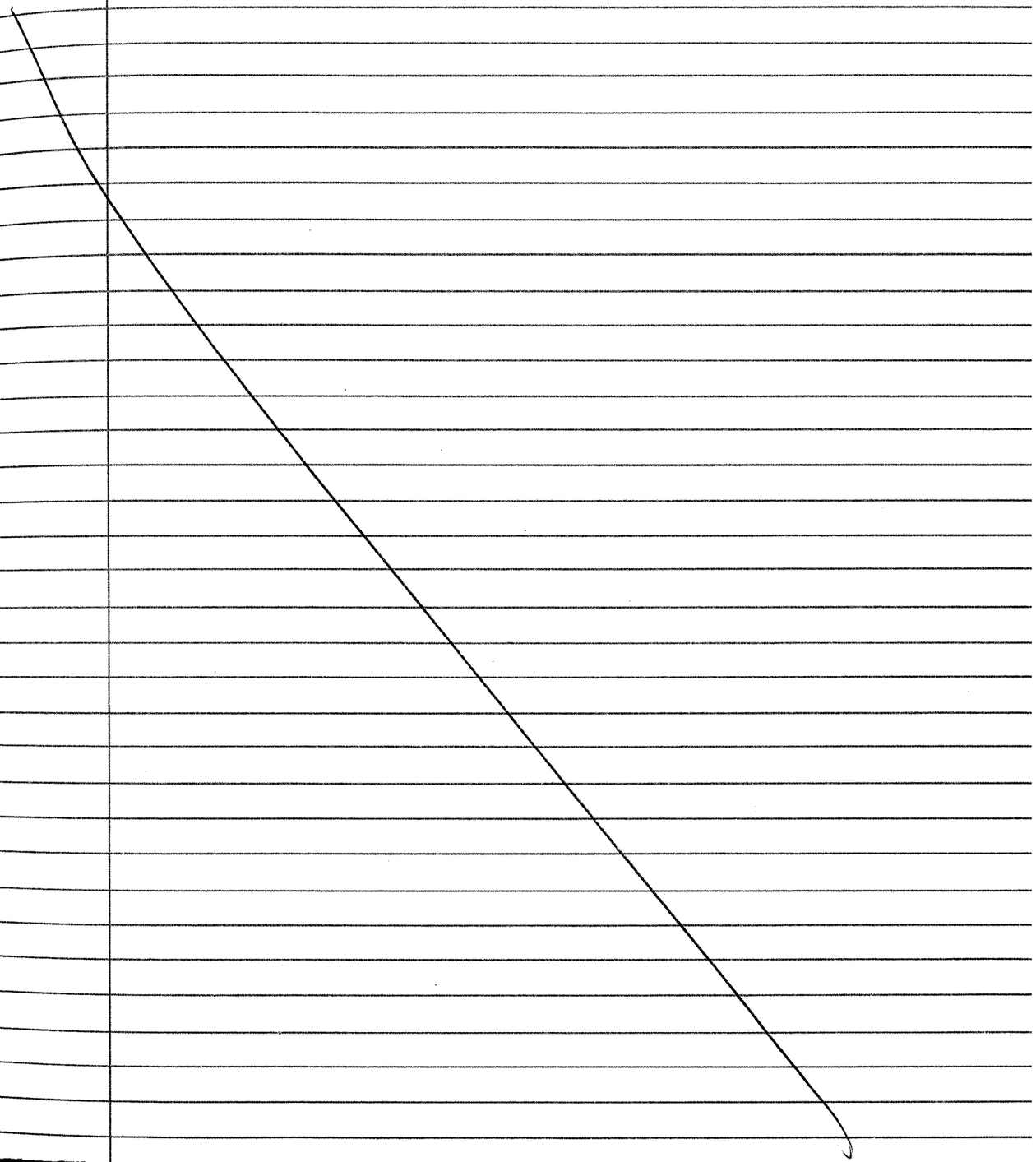

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Oct. 8, 1999 MNugent

Preparation of clinoptilolite for XAFS analysis after U is sorbed to clenoptilolite

Materials & Equipment

1. Clinoptilolite from Death Valley Junction, California
2. Stainless steel sieves: 8 inch diameter, 325 and 425 mesh
3. RO-tap sieve shaker
4. Ultrasonic cleaner FS-28 (Fisher Scientific)
5. Water bath
6. 1N Sodium acetate buffer (pH=5.0]
   preparation: 1.36 g NaOAC to 1 Liter of nanopure H_2O.
   Sodium Acetate: Fisher Lot# 937077
7. 0.3M Na-citrate dihydrate solution
   preparation: 900ml. Na Citrate Dihydrate: solution
   Fisher Scientific, Lot # 940621
8. Na-dithionite ($Na_2S_2O_4$), Fisher purified grade
   Lot #: 912722 ~~Fisher~~ MN 10/8/99
9. Deionized water saturated with NaCl
   NaCl Lot #: 914913, Fisher ACS Grade
10. 1N $NaHW_3$ ~~Soln~~ MN 10/8/99 Solution
    preparation: $NaHW_3$ Manufacturer: Fisher Scientific ACS grade
    prepared 1L. Lot # 986883
11. Nano-pure H_2O = 'deionized H_2O' or d.i. H_2O
12. Graduated beakers - glass (several)
13. Magnetic stirrers
14. Stir/hot plate (Corning)
15. Plastic bottles (acid-washed, to store solutions)
16. Centrifuge tubes (50ml, several)
17. Stirrers
18. Filter paper
19. Spatulas
20. Weighing balance - Mettler PM 4600 Delta Range
21. Acetone [Fisher Scientific, Lot # 992389]

Oct. 8, 1999 M. Nugent

22. Centrifuge - Fisher scientific, marathon 21K
23. Drying oven (Stabil-therm)
24. Ependorf ~~micropet~~ MN 10/8/99 micropipetter
25. Eppendorf micropipettor tips
26. pH Meter Orion 920A
27. pH Electrode - Ross combination
28. NBS Buffers 4, 7, 10.

Continued on next page.

10/8/99 - MNugent

Sieving The Clinoptilolite:

1. Using clinoptilolite which has already been crushed and sieved to ~200 mesh
2. Sieve to 425 using stainless steel sieves, sieve shaker - ~100g starting material, sieved for 45 minutes.
3. 325 - 425 fraction washed with d.i. water several times, until supernatant is clear. Solids are ultra-sonicated each time.

10/11/99 MNugent

Removal of Carbonates

1. Heated water bath to 95°C
2. Placed 5 grams of clinoptilolite in 50ml centrifuge tubes (8 tubes prepared).
3. Added 50ml of 1N NaOAc buffer (@ pH = 5) to each tube, capped, and shook thoroughly.
4. All tubes placed in water bath for digestion. Temp of water bath decreased upon addition of tubes, therefore, the tubes were left in the water bath for 30 minutes after the temperature of the water bath returned to 95°C (45-50 min. total). Each tube was shaken approximately halfway through the digestion.
5. Centrifuge tubes were centrifuged at 6000rpm for 5 minutes, and the supernatant was decanted.
6. The suspension was then washed in deionized water and centrifuged at 6000rpm for five minutes, two times.
7. Clinoptilolite placed in oven at ~60°C to dry.

10/13/99 MNugent

Removal of Iron Oxides:

STOP.

Instead, we will use clinoptilolite that was prepared by B. Pabalan.

This clinoptilolite will NOT be used.

~~MN 10/13/99~~ MN 10/13/99

Note, clinoptilolite prepared on these pages 5 through 8 of CNWRA Scientific notebook controlled copy # 370 will be returned to the storage shelf.

Preparation of Montmorillinite for U adsorption and XAFS analysis — M Nugent 10/15/99

Materials and equipment

1. Montmorillinite - Apache County, Az. [Source: Clay Minerals Repository]
2. all other materials / equipment, see p. 5 of this book (copy # ~~370~~ 370 MN 10/15/99

Montmorillinite was not sieved.

MN 10/15/99
10/~~15~~ 15/99 Removal of Iron Oxides

1. 10 grams of montmorillinite was placed in 50ml centrifuge tubes.
2. 40 ml of 0.3M Na-citrate solution and 5ml of 1M $NaHCO_3$ solution were added to the test tube. The test tubes were closed with caps and shaken well.
3. Tubes were placed in the temperature 75-80°C water bath (bath was preheated and monitored to make sure that the temp did not exceed 80°C)
4. 1g of $Na_2S_2O_4$ was added to each tube, and the mixture was stirred with a stirring rod. The test tube was capped and shaken well. The test tubes were allowed to sit in the water bath for 5 minutes, and each test tube was shaken twice during the 5 minutes.
5. A second 1g of $Na_2S_2O_4$ was then added, stirred with the stirring bar, capped, shaken & digested for 5 minutes (2 shakes during 5 minutes)
6. A third 1g of $Na_2S_2O_4$ was added and stirred with the stirring bar, capped, shaken, and digested for 5 minutes (with 2 shakes during the 5 minutes).
7. The suspensions were removed from the water bath and immediately centrifuged at 6000rpm for 5 minutes. The supernatant was then decanted.

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10/15/99 & 5 mL of NaCl-saturated deionized water was then added to each test tube, the test tubes were shaken, and placed in the hot water bath for 5 minutes, with one shake after approximately TWO minutes. The suspension was then centrifuged at 6000 rpm ~~and~~ MN 10/15/99 for 5 minutes. The supernatant was poured off.

9. The suspension was then rinsed with deionized water, shaken, and centrifuged (6000 rpm x 5 minutes) twice, pouring off the supernatant each time.

10. The montmorillonite was then dried overnight in the oven.

10/16/99 Upon removal, it appeared that there was a sulphur precipitate in the montmorillonite sample. Therefore, the above procedure was repeated with the following modifications: [The numbers below refer to the numbers above, in the 'iron oxides removal' section, pages 9-10 of this notebook - CNWRA controlled copy # 370]:

4,5,6. Test tubes allowed to heat & digest for 10-15 minutes for each digestion.

7. centrifuged at 2000 rpm for 3 minutes.

8. 10 mL of saturated NaCl - deionized water added, allowed to sit in bath for ten minutes, centrifuged 2000 rpm 3 minutes. NaCl procedure repeated.

9. Deionized water washing was repeated 4 times - each time the test tubes were heated just long enough ~~to~~ MN 10/18/99 so the montmorillonite became suspended - test tubes were well shaken. centrifuged at 2000 rpm x 3 min.

10. Montmorillonite was dried in air, with 1 hr in oven at 60°C No sulphur precipitate was seen after drying.

11. Montmorillonite was ground up.

10/18/99 MN Nugent **Removal of Carbonates.**

1. Water bath heated to 95°C

2. Placed 5 grams of mont. in 50 mL test tubes (centrifuge

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test tubes).

MN 10/18/99 10/16/99

3. 50 mL of NaOAc buffer pH=5 added to each ~~cf~~ centrifuge tube. The tubes were capped and shaken vigorously.

4. Centrifuge tubes placed in water bath and digested at 95°C for 30 minutes. Each centrifuge tube was shaken 2 times during digestion.

5. After 30 minutes, the tubes were removed from the water bath and centrifuged at 2000 rpm for 4 minutes.

6. Each centrifuge tube was decanted and filled with deionized water. Tubes were shaken well, heated briefly (to help clay disperse), and centrifuged at 2000 rpm for 3 minutes. Process was repeated twice (total of three times). - EXCEPT -

7. Tubes were centrifuged at 8000 rpm for 5 minutes after the last washing. The supernatant was collected, and the solids were spread onto watch glasses. The supernatant was then re-centrifuged at 8000 rpm for 6 minutes, and the solid was ~~add~~ MN 10/18/99 smeared onto a watch plate.

8. The montmorillonite was allowed to dry at room temperature, in air.

9. Samples were then ground up.

10/21/99 M. Nugent

Adsorption Experiments for XAFS analysis: UO_2 adsorption onto clinoptilolite, cleaned montmorillonite, and 'uncleaned' montmorillonite. M Nugent 10/21/99

Procedure for preparing uranyl-loaded clinoptilolite and montmorillonite samples for XAFS analysis

Prepared by: Melissa Nugent

Objective:

To prepare samples of uranyl-loaded clinoptilolite and montmorillonite (for each mineral, one set of samples will be prepared via ion exchange and one set of samples via surface adsorption) for XAFS analysis at Brookhaven National Laboratory, to investigate the difference between ion exchange and surface adsorption. Surface adsorbed samples are prepared at pH 6 and ion exchanged samples at pH 3.

Materials:

Please see page 15 of this scientific notebook, CNWRA Controlled Copy # 371 for a list of the materials used in these experiments.
Note, these mineral samples and other materials will be used unless otherwise noted.

Conditions:

Room temperature, 1 g solid per 0.45 L

General Procedure:

1. Prepare a stock solution of uranyl nitrate such that approximately 1ml of stock solution is required to achieve the desired concentration for 0.45ml of experimental solution.
2. Prepare 0.45 L solutions via dilution of the uranyl stock solution:
   A. weigh bottle, add de-ionized water, record total mass.
   B. Add reagants, record total mass.
   C. Weight cap and bubblers separately.
3. Adjust pH of each solution to a pH of 3 for ion exchange with HNO3 or a pH of 6 for surface adsorption using either HNO3 or NaOH. Attach to bubblers to the bottles and place on gyratory shakers.
4. Check pH periodically. If pH is acceptable (3 for ion exchange or 6 for surface adsorption), proceed to step 5. If the pH requires adjustment, perform the adjustment and then return the bottle to the bubblers and shaker. Take two aqueous samples from each bottle:

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A. Weigh sample bottle and cap
B. Remove sample using a plastic syringe. Filter sample into sample bottle using a 0.45 micron gelman-type filter or a syringe tip filter (approximately 0.5 to 1 ml may be lost via filtration). Record mass of sample. Acidify the sample to pH 1.

4. Add solid material. Check pH immediately.
5. Approximately every second day, measure and adjust pH as necessary. Do not sample. Allow experiment to continue for at least 10 days.
6. Collecting the XAFS sample:
   A. Measure pH and take two samples from each bottle, using the procedure in 4. Let bottles sit, without bubbling or shaking, approximately 30 minutes.
   B. Decant as much as possible into a weighed, clear bottle.
   C. Pour the remaining solution and solid into 45 ml centrifuge tubes, taking care to transfer all solid.
   D. Centrifuge at 10,000 to 12,000 g for 25 minutes, pour off liquid, & transfer paste to XAFS holder.

Materials:

- uranyl nitrate: $UO_2(NO_3)_2 \cdot 6H_2O$, Mallinkrodt Lot # 8640 KCAP
- 500ml bottles, acid washed, with a hole in the center of the cap
- clinoptilolite = CDV * 100/200 * UC * WA * bLL * CAT — See below
- montmorillinite - Apache County, Az. [Source, Clay Minerals Repository] — See below
- cleaned montmorillinite: see pages 9-11 of this notebook, CNWRA Controlled Copy 37
- fish tank pumps
- plastic tubing, assorted sizes
- plastic stopcocks
- teflon tape
- bubblers with glassfrits
- NaOH and HNO_3 for base/acid additions, respectively.
- ~5ml sample holders (vials)
- pH meter, 1, 4, 7, 10 buffers, Ross combination electrode.
- pipettors
- pipettor tips
- shaker (gyratory shaker)
- centrifuge
- centrifuge tubes (50ml)
- Sodium Chloride
  Manufacturer Fisher ACS grade
  Lot # 914913

Clinoptilolite:
Source Locality - Death Valley Junction, CA
Minerals Research Co (Clarkson, NY)
JRd 6/14/00 Impurities removed - exchanged to Na-Clinoptilolite Form w/ NaCl
Prepared by R. Pabalan, CNWRA Notebook GC-03 (R. Pabalan) pg. 182

Montmorillonite: The montmorillonite described in this notebook as 'uncleaned' is Apache county, Az montmorillonite that was not treated and was not ~~sieved~~ MN 10/8/99 sieved.

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10/21/99 Preparation of Uranyl-nitrate stock solution:

~~10/21/99 Nugent~~ by M. Nugat, 10/21/99

~~Weight~~ Mass of 500ml bottle: 57.21g

Mass of cap: 9.35g

- Weigh out water:

Mass bottle 57.21

- tare - 0.00

Add deionized water - Mass = 300.03g

- Weigh out uranyl nitrate

weighboat 0.5766g

tare 0.00

U-nitrate 0.2388g

add u-nitrate to 500ml bottle

mass of weighboat 0.0004g

⇒ Mass u-nitrate added:

0.2384 grams.

- Add Uranyl nitrate to D.I. water

Mass D.I. + cap + U-nitrate = 309.58

(after U-nitrate dissolved)

So - calculate concentration of U in solution:

- $0.2384\,g\ \text{U-nitrate} \times \frac{\text{mole}}{502\,g} = 4.2 \times 10^{-4}\ \text{moles}$

- 309.58g

- ~~9.53g~~ -9.35g MNugent 10/21/99

300.23g

- $\frac{4.2 \times 10^{-4}\ \text{moles U}}{0.30023\ L} = 1.399 \times 10^{-4}\ \text{M/L U}$

$= 332.99\ \text{ppm U.}$

Empty space

M. Nugent

10/21/99 2. Weigh out bottles, caps, and bubblers: M. Nugent, 10/21/99

	Cap (g)	Bubbler (g)	Bottle (g)	mineral to be added:
CIE-A	9.2264	19.1994	56.5322	clinopt.
CMSA-A	9.1707	18.9284	57.3730	MNugent 10/21 ~~clinopt.~~ cleaned mont
CMIE-A	9.2928	18.8518	56.5046	cleaned mont mor.
CSA-A	9.2942	MNugent 10/21/99 19.1819	57.3073	clinopt.
UMSA-A	9.2352	19.2320	56.5059	MNugent 10/21/99 ~~clinopt.~~ 'cleaned' mont.
UMIE-A	9.2577	19.2975	56.9495	~~an~~cleaned mont. MNugent 10/21/99

bottles, MN 10/21/99 caps & bubblers weighed after drying in oven & cooling to room T.

3. Dilution of Stock Solution: M. Nugent 10/21/99

for 22 ppm, 29.73 ml of stock solution in 450 ml

for 11 ppm, 14.87 ml of stock solution in 450 ml

CIE-A Mass stock solution: 29.79 g

mass stock solution + D.I. water: 450.04 g

So, calculate Concentration:

$C_1V_1 = C_2V_2$

$332.99\,(29.79) = 450.04 * C_2 \qquad C_2 = 22.042$ ppm

CMSA-A Mass stock slth: 14.86

Mass stock slth + DI water: 450.06

Therefore, 10.995 ppm.

CMIE-A Mass stock slth: 29.75

Mass stock slth + DI Water: 450.08

Therefore, 22.010 ppm

CSA-A Mass stock slth: 14.90

Mass stock slth + DI water: 450.00

Therefore, 11.026 ppm

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UMSA-A Mass stock solution: 14.88

Mass stock slth + DI Water: 450.11

Therefore, 11.008 ppm

UMIE-A Mass stock solution: 29.84

Mass stock slth + DI water: 450.17

therefore, 22.073 ppm.

Solutions are capped and stirred gently.

4. pH measurement and adjustment: M. Nugent, 10/21/99

electrode is cleaned, fresh buffers used.

CMIE-A pH = 4.40

added 13 x 0.5 ml of 0.05 m HNO_3 ⇒ pH = 3.07

CIE-A pH = 4.45

added 3 x 0.1 ml + 12 x 0.5 ml of 0.05 m HNO_3 6.8 ml

MNugent 10/21/99 pH = 3.05

UMIE-A ~~=U~~ MIE-A pH = 4.38

added 12 x 0.5 ml of 0.05 m HNO_3 ⇒ pH = 3.07.

CSA-A pH = 4.55

12 x 0.1 ml of 0.05 m NaOH, pH = 6.37.

CMSA-A pH = 4.52

added 9 x 0.1 ml of 0.05 m NaOH, pH = 6.36

UMSA-A pH = 4.55

added 10 x 0.1 ml of 0.05 m NaOH, pH = 6.39.

Solutions are attached to air bubblers, and shaken on the gyratory shaker overnight.

10/22/99

5. Check pH and add solid to each bottle & take as sample

if the pH has not changed significantly, add clenoptilolite/mont. to the bottle.

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Note: aqueous sample is taken before solid is added.

	(Clinopt.) CIE-A	(Clinopt) CSt-A	uncleaned mont. UMIE-A	uncleaned mont UMSt-A	cleaned mont. CMIE-A	cleaned mont. CMStA
Mass of Mineral	1.0020	1.0002	1.0037	1.0025	1.0048	1.0022
Sample 1 -name	CIEA-1	CStA-1	UMIEA-1	UMStA-1	CMIEA-1	CMSAA1
└ Bottle		3.3254	3.2996	3.3013	3.2896	3.2997
└ Bottle+Sample		7.0216	6.8789	7.0166	7.2745	7.2297
∴ Sample						
Sample 2 -name	CIEA-2	CSAA-2	UMIE A-2	UMStA-2	CMIEA-2	CMStA2
└ Bottle	3.3033	3.4210	3.3373	3.3119	3.2961	3.3970
└ Sample+Bottle	7.1998	7.1678	~~7.6~~ 6.7687 MN 10/29/99	7.2095	7.0521	7.6295
∴ Sample						
pH before mineral	3.03	*7.02?	3.07	6.35	3.07	6.40.
pH after mineral added	3.26	7.01	3.11	6.40	3.25	6.42

note: the amount of time lapsed between mineral addition and pH measurement (for 'pH after mineral added') varied somewhat. Also the mineral dispersal into the solution varied from mineral to mineral. These could explain why the pH changed upon addition for some samples but not for others.
* pH drifting a lot, pH is probably lower?

Bottles are returned to shaker, and are shaken and stirred continuously. Twice a day the bottles are removed and thoroughly mixed so the minerals are suspended.

10/28/99 MNugent 6. pH check & adjustment (next page)

This is empty space

pH check and adjustment. M. Nugent

	CIE-A 10/29/99	CSt-A 10/29/99	UMIE-A 10/29/99	UMSt-A 10/29/99	CMIE-A MN 10/29/99 10/29/99	CMSt-A 10/29/99
pH	4.24					
ml of acid/base	6.7 ml of 0.05m HNO_3	→ 7.31 → 0.1 ml of 0.05m HNO_3	→ 3.84 → 0.5 ml of 0.1m HNO_3	→ 6.95 → 0.3 ml of 0.05m HNO_3	5.41 MN 10/29/99 → ~~3.84~~ 0.5 ml of 0.05m HNO_3	→ 7.43 1.1 ml of 0.05m HNO_3
pH final	3.11	6.28	3.21	6.29	2.98	6.29
	10/30/99 2pm	10/30/99 MN 10/30 7.09	10/30/99	10/30/99	10/30/99	10/30/99
pH	3.93	~~6.88~~	3.19	6.69	3.09	7.14
ml of acid/base added	0.5 ml of 0.1m HNO_3	0.5 ml of 0.05m HNO_3	none added	0.5 ml of 0.05m HNO_3	none added	0.8 ml of 0.05m HNO_3
pH final	3.22	5.98/99	/	5.80	/	6.33
	11/1/99 ~2pm					
pH	3.89	~~6.11~~ MN 11/1/99 7.16	3.34	6.26	3.22	7.00 filter slightly clogged
ml of HNO_3	0.5 ml of 0.1m HNO_3	0.5 ml of 0.05m HNO_3	0.1 ml of 0.1m HNO_3	none added	none added	0.5 ml of 0.05m HNO_3
pH final	3.29	~~5.73~~ 6.31 MN 11/1/99	3.28		filter greenish	6.33/34
	11/2/99 ~1130 AM					
pH	3.55	6.71	3.30	6.29	3.27	6.70
ml of HNO_3 added	0.5 ml of 0.1m HNO_3	0.3 ml of 0.05m HNO_3	0.1 ml of 0.1m HNO_3	none added	none added	0.2 ml of 0.05m HNO_3
pH final	3.16	6.29	3.22		filter greenish	6.35
	11/3/99 - Last day of experiment! ~5 pm.					
pH	3.40	6.72	3.24	6.33	3.30	6.54

✓ Date: 11/3/99 ~7pm

7. Final sampling - take an aqueous sample @ end of experiment

Mass of:	CIEA	CStA	UMIE A	UMStA	CMIE A	CmStA
Bottle+cap	7.3132	7.2396	7.2907	7.3161	7.2194	7.2667
~~Bottle+cap+acid~~	MN 11/3/99					
Bottle+cap+sample	9.3114	9.0918	9.1006	9.3862	8.2124	7.9352
Bottle+cap+acid+sample	9.7001	9.5367	9.5525	9.8087	8.7059	8.3353
Name:	~~CIBA-3~~ CIEA-3 MN 11/3/99	CSt A 3	UMIEA 3	UMStA 3	CMIE A-3	CMStA 3
Bottle+cap	7.2777	7.3534	7.2881	7.3303	7.7139	
Bottle+cap+sample	9.4896	9.2626	10.1663	10.0467	7.8710	no sample.
Bottle+cap+acid+sample	9.9744	9.7378		10.4130	8.0357	
Name	CIEA-4	CSAA4	UMIEA4	UMStA4	UMIEA-4	

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11/3/99

Take down: 11/3/99

Bottles were removed from the bubblers, and centrifuged in 45ml centrifuge tubes. Centrifuging was performed at 10,000 rpm until solids had collected on the bottom of the test tubes. Solutions were discarded. Wet solids were mounted in XAFS sample holders and sealed tightly.

Samples were Then brought to Brookhaven National Laboratory for analysis by XAFS.

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11/3/99

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10/22/99 MNugat

U Adsorption Experiments

10/22/99
M. Nugent

Objective: to perform adsorption experiments with uranium (UO_2) and montmorillonite (cleaned and uncleaned) and clinoptilolite. These experiments will be replicates of the U-adsorption experiments which are described on pages 13 through 23 of this notebook (CNWRA Controlled Copy # 370), except they will be scaled down to 100ml, and spiked with 5ppb of 233U.

Procedure: see pages 13 - 14 of this notebook (CNWRA Controlled copy # 370).

Modification:

- 10 ml of 50ppb 233U will be added to each bottle (for 5ppb final U concent.)
- Total volume will be 100ml (mineral: 0.22g to keep M/V the same as in previous adsorption experiments)
- Solutions will be sampled and samples will be analyzed by LSA.

Materials: See page 15 of this notebook (CNWRA controlled copy #370)

Modification: Add the following materials:

233U spike (described below)
pipettor for 10ml.
pipette tips for 10ml

The 233 uranium spike used was ~~#46~~ MN 10/22/99 #47.
This uranium spike is described on pages 293 and 294 of CNWRA Scientific notebook Controlled copy # 031.
U-233 spike: Nist Certificate, Catalog # 7223, Source # 678-32-3, Reference Date 1-Sept-99.

10/22/99 M. Nugent

1. Weigh out bottles, caps, bubbling tubes, 50ppb stock solution, U-nitrate, deionized water. M. Nugent 10/22/99

10/22/99

(Mineral to be added)	CIE-B (clinopt.)	CSA-B (clinopt.)	CMIE-B (cleaned mont.)	CMSA-B (cleaned mont.)	UMIE-B (uncleaned mont.)	UMSA-B (uncleaned mont.)
Mass-Bottle	16.5412	16.6160	16.3006	16.5782	16.3919	16.3957
-Cap	2.4745	2.6598	2.6794	2.7014	2.4469	2.6836
-2 Bubble Tubes	0.3020	0.3204	0.3331	0.2979	0.3176	0.4194
Mass 50ppb spike of 233U	10.0114	10.0680	10.0954	10.0155	10.1673	10.1106 MN 10/22/99
Mass of 332.99ppm U stock sltn *	6.6621	3.3150	6.6354	3.3310	6.6576	3.3658
Mass D.I. water	84.5772	88.4560	85.0296	86.1218	86.1202	86.5576

* Calculated concentrations *

$C_1V_1 = C_2V_2$

ex: 50ppb dilution for CIE B:

$50ppb \times 10.0114 = (84.5772 + 6.6621 + 10.0114) * C_2$

$C_2 = \frac{50 \times 10.0114}{101.2507} = 4.944 ppb.$

	CIE B	CSA-B	CMIE B	CMSA-B	UMIE-B	UMSA-B
Total Mass	101.2507	101.839	101.7604	99.4683	102.9457	100.0034
233U (ppb)	4.944	4.943	4.960	5.035	4.624	5.055
U * (ppm)	21.910	10.839	21.713	11.151	21.535	11.207

* note stock solution is The 332.99ppm U-nitrate solution described on page 17 of this notebook (CNWRA #370)

10/22/99 M. Nugent

2. pH measurement and adjustment 10/22/99

10/22/99	CIE B	CSA B (MN 10/22/99)	CMIE B	CMSA-B	UMIE B	UMSA B
pH	4.38	4.41	4.36	4.36	4.42	4.33
Acid/Base Addition	2x0.5ml 0.05m HNO_3 + 4x0.2ml 0.05m HNO_3	2x0.2ml 0.005m NaOH (approx-air bubble)	8x0.2ml of 0.05m HNO_3	0.4ml of 0.005M NaOH	8x0.2ml of 0.05m HNO_3	~0.35ml NaOH (0.005M) Air bubble!
pH	3.1	6.21	3.07	6.57	3.09	6.34

Solutions placed on gyratory shaker, attached to air bubblers, and sat over night.

10/23/99 3. Check pH, take a sample (for LSA), and add mineral 10/22/99

	CIE-B	CSA-B	CMIE B	CMSA B	UMIE B	UMSA B
pH	3.09	~~6.42~~ * 6.42 MN 10/22/99	3.07	6.55	3.09	6.39
Sample 1 name	CIE B-1	CSA B-1	CMIE B-1	CMSA B-1	UMIE B-1	UMSA B-1
Mass Bottle	3.2970	3.3297	3.3177	3.4005	3.3252	3.3231
Mass Sample + Bottle	4.8316	4.7384	4.3932	5.2002	5.1397	5.0158
∴ Mass of Sample	calculated in spreadsheet					
Sample 2 name	CIE B-2	CSA B-2	CMIE B-2	CMSA B-2	UMIE B-2	UMSA B-2
bottle	3.2891 ~~3.3297~~ 10/23 MN Nugent	3.3119	3.4094	3.3233	3.3162	3.3271
bottle + sample	4.5191	4.4668	4.9929	4.3291	4.4396	4.4046
∴ Mass of sample						
Mass of Mineral	0.2228	0.2230	0.2235	0.2226	0.2216	0.2221
pH after	3.62	6.52	3.49	6.66	3.52	6.73

Solutions were shaken well and placed on gyratory shaker to shake and bubble.

* pH drifting.

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10/27/99

Check pH, take a sample

	CIEB	CSA B	CMIE B	CMSA B	UMIE B	UMSA B
	MN 10/27/99 5.14			MN 10/27/99 7.43		
pH	~~5.20~~	7.08	4.70	~~7.45~~	3.67	6.96
Sample 3 Name						
Mass Bottle+Cap						
Mass Bottle+Cap +Sample						
Sample 4 Name						
Mass Bottle+Cap						
Mass Bottle+Cap +Sample						

Samples were recorded on page 30 of this notebook, CNWRA Controlled Copy # 370, instead of here.

pH checks and adjustments:

	CIE-B	CSA-B	CMIE-B	CMSA-B	UMIE-B	UMSA-B
Date ~3:30pm	10/29/99	10/29/99	10/29/99	10/29/99	10/29/99	10/29/99
pH	5.16	6.89	4.73	7.40	3.75	6.89
ml of acid/base added to sltn.	1.5ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3	1ml of 0.05m HNO_3	< 0.5ml of 0.05m HNO_3	0.5ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3
pH final	3.31	6.33	3.36	6.23	3.32	6.20/6.21
~2pm	10/30/99	10/30/99	10/30/99	10/30/99	10/30/99	10/30/99
	4.16	6.88	3.73	6.99	3.43	6.81
	1ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3	0.5ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3	MN 10/30/99 0.3ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3
	3.29	5.98	3.28	6.09	3.23	5.99
	11/1/99 ~2pm					
pH	3.79	6.71	3.43	6.33	3.27	6.26
ml of HNO_3 added	0.7ml of 0.05 HNO_3	0.1ml of 0.05m HNO_3	0.3ml of 0.05m HNO_3	none added	none added	none added
pH final	3.25	5.73	3.25			
	11/2/99 ~11:30a.m.			switch! MN 11/2/99		
pH	3.55	6.36	3.34	3.33	6.40	6.38
ml of HNO_3	0.5ml of 0.05m HNO_3	none added	0.2ml of 0.05m HNO_3	0.2ml of 0.05m HNO_3	0.1ml of 0.05m HNO_3	3 drops of 0.05m HNO_3
pH final	3.24		3.23	3.21	5.20 oops!	5.98

pH check con't

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11/3/99

	CIEB	CSA B	CMIE B	CMSA B	UMIE B	UMSA B
11/3/99 – Last day of experiment ~5pm						
pH	3.48	6.42	3.26	6.17	3.22	5.70

~~Take down~~ MN 11/3/99

Please see next page of this notebook, CNWRA controlled copy # 370, for sampling and page 31 of this notebook, CNWRA controlled copy 370, for take down.

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10/29/99

Note: acid is 0.02m HNO_3 (unless otherwise noted) for any acid added on this page.

Sampling

MN 10/29/99
~~UMIE-B~~

	CIE-B	CSA-B	CMIE-B	CMSA-B	UMIE-B	UMSA-B
Date taken:	10/29/99					
Sample Name:	CIEB-3	CSAB-3	CMIEB-3	CMSAB-3	UMIEB-3	UMSAB-3
Mass: Bottle+Cap	7.3100	7.2485	7.2517	7.2824	7.3175	7.3725
Mass: Bottle+cap+sample	8.5041	8.7678	8.5441	7.8553	8.4551	8.5132
Date Taken:	10/29/99					
Sample Name	CIEB-4	CSAB-4	CMIEB-4	CMSAB-4	UMIEB-4	UMSAB-4
Mass: Bottle+Cap	7.3138	MN 7.2709	7.2409	7.2943	7.2835	7.2802
Mass: Bottle+Cap+sample	9.0184	8.6275	8.6118	7.7560	8.3915	8.3014
Mass Sample #-3:	1.1941	1.5193	1.2924	0.5729	1.1376	1.1407
Mass Sample -4.	1.7046	1.3566	1.3709	0.4617	1.108	1.0212

note: Samples had to be filtered - therefore a few drops (estimated 0.5ml) of sample were lost to rinsing the filter out.

Date taken:	11/1/99 ~ 4.30 pm					
Mass Bottle+Cap	7.2816	7.3163	7.2635	7.3184	7.3158	7.2275
Mass Bottle+Cap+Sample	8.2677	8.6350	7.9798	7.9219	8.7265	8.9185
Mass Sample+Bottle+Cap	8.7622	9.1284	8.4757	8.4178	9.2053	9.4120
Name: MN 11/1/99	CIEB-5	CSAB5	CMIEB5	CMSAB5	UMIEB5	UMSAB5
Date taken:	11/1/99 ~ 4.30 pm					
Name:	CIEB-6	CSAB6	CMIEB6	CMSAB6	UMIEB6	UMSAB6
Mass Bottle+Cap	7.3050	7.3195	7.3063	7.3100	7.2353	7.3264
Mass Bottle+cap+sample	8.4786	8.2407	8.1012	7.9299	8.5037	8.1256
Mass Bottle+cap+sample+acid	8.9743	8.7369	8.5994	8.4241	8.9979	8.6191
Date Taken:	11/3/99 ~7pm Last day of experiment					
Mass Bottle+Cap	7.2950	7.2945	7.2728	7.2618	7.2729	7.2798
Mass Bottle+cap+acid	7.7873	7.7863	7.7526	7.7421	7.7727	7.7500
Mass Bottle+Cap+acid+sample	8.9465	8.6001	8.5027	8.4230	8.8945	9.1395
Name	CIEB-7	CSAB7	CMIEB7	CMSAB7	UMIEB7	UMSAB7
Date	11/3/99 ~7pm Last Day of experiment					
Name	CIEB-8	CSAB8	CMIEB8	CMSAB8	UMIEB8	UMSAB8
Mass Bottle+Cap	7.2469	7.2695	7.2418	7.2751	7.3128	7.3108
Mass Bottle+cap+acid	7.7391	7.7611	7.7342	7.7653	7.8064	7.8015
Mass Bottle+cap+acid+sample	9.2728	9.0668	8.5640	8.3987	9.1201	9.3366

11/3/99

Take down of experiment: by M. Nugent 11/3/99 M. Nugent

Solids and solutions were not separated.

The shaker was stopped, the bubblers were stopped and removed, and the solutions and solids were collected, without separation. There will be no analysis of the solid material.

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11/3/99

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11/3/99

10/26/99

10/26/99
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Uranyl ion-adsorption onto clinoptilolite, cleaned montmorillonite and 'un-cleaned' montmorillonite @ high UO_2 concentration

Objective: to repeat the preparation (found on p13-23 and 25-33 of CNWRA #370 MNugent 10/26/99) of uranyl-adsorbed minerals for XAFS analysis - except, for these experiments the uranyl ion will be 100ppm.

A set of 450 ml and 100 ml experiments - for XAFS analysis and monitoring, respectively, will be prepared - each sample with:

- 100 ppm U
- M/V = 1/450 = 0.222/100 = 2.2 g/L
- pH ~3 for ion exchange

Then, for each mineral, a 450 ml sample and a 100 ml sample (the 100 ml sample spiked with 5ppb ^{233}U) will be prepared.

Procedure: The 450 ml samples will be prepared according to the method described on p. 13-19 of this notebook (CNWRA controlled copy #370) and the 100 ml sample will be prepared by the procedure described on p. 25 of this notebook (CNWRA Controlled Copy #370).

Materials see p. 15 and 25 of this notebook (CNWRA Controlled Copy # 370).

10/26/99 1. Prepare a 330 ppm Uranyl stock solution from uranyl nitrate

		MN 10/26/99	Unitrate:	
Mass of Bottle	~~57.21~~ g	56.43	mass weighboat	0.5234
mass of cap	~~9.35g~~	9.39	tare	0.0000
			mass unitrate	0.3501
Mass Bottle	~~57.21~~	56.43	mass weighboat after unitrate addition	0.0000
tare		0.00		
Add unitrate +	Add D.I.	501.70	Mass unitrate added:	0.3501

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Therefore, actual concentration of U in stock solution:

$\frac{0.3501\,g}{0.5017\,L} \times \frac{mol}{502\,g} = 0.00139\,M/L = 330.883\,ppm.$

2. Weigh out bottles, caps, bubblers & dilute stocksolution dilutions:

for 450mL of 100ppm: $C_1V_1 = C_2V_2$

$450ml \times 100ppm = 330.883ppm \times V_2$

$V_2 = 135.9997 = 136mL$

for 100 ml 100ppm:

$100 \times 100 = 330.883 \times V_2$

$V_2 = 30.222\,ml.$

10/26/99

CIE-C (100ml)	UMIE-C (100)	CMIE-C (100)	CIE-D (450ml)	UMIE-D (450ml)	CMIE-D (450ml)
U mineral to be added	(clinopt.)	(uncleaned mont.)	(Cleaned mont.)	(clinopt.)	(uncleaned mont.)	(Cleaned mont.)
Bottle	16.7236	32.5845	32.3653	57.2510	56.5401	56.7383
Cap	2.6537	5.8620	5.8299	9.3525	9.2837	9.3061
Bubblers	0.5016	0.5022 MN	0.4913	17.54	24.15	19.25
		31.3654				
U-nitrate Stock Sltn	30.2231	~~30.2514~~ MN 10/26	31.3192	136.00	136.03	136.07
U nitrate stock solution + D.I. water	100.1163	120.1083	125.6781	455.14	451.27	450.94
pH	4.07	3.92	3.92	3.85	3.89	3.84

* calculated concentrations * (ppm)

	CIE-C	UMIE-C	CMIE-C	CIE-D	UMIE-D	CMIE-D
Total mass 233U (ppb)	~~99.88~~ MN 10/26/99	—	—	—	—	—
U(u-nitrate) (ppm)	99.8869	~~89.4566~~ MN 10/26/99 86.4407	~~98.8708~~ MN 10/26/99 82.4566	98.8708	99.7408	99.8431

* Addition of HNO_3 - 0.05m HNO_3

	CIE-C	UMIE-C	CMIE-C	CIE-D	UMIE-D	CMIE-D
ml of 0.05m HNO_3	3x0.5ml	3x0.5ml	3x0.5ml	4x0.5ml	3x0.5ml	3x0.5ml
pH	3.08	3.02	3.04	3.01	3.08	3.08

Set to bubble; shake overnight.

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10/27/99 M Nugent 3. Forgot to add 233U to the 100ml solutions. Therefore, will add the appropriate amt of 233U and adjust everything else to keep M/V, etc, the same.

1. 233U: $C_1V_1 = C_2V_2$

~~100ml x 5ppb~~ MN 10/27/99

$(100ml + x\,ml) \times 5ppb = 50ppb \times X \Rightarrow (100 \times 5) + 5x = 50x$

$500 = 45x, \quad x = 11.11\,ml$

⇒ Add 11.11 ml 233U to solutions.

2. amount of U stock solution for 100ppm ~~100~~ MN 10/27 111 ml solution:

$330.883ppm \times V_1 = 111ml \times 100ppm$

$V_1 = 33.5466\,ml$

Therefore add to the solution the difference between 33.5466ml and the amount actually added on 10/26:

Ex: CIE-C $33.5466 - 30.2231 = 3.3235$

Effect on 233U:

$(111.11 + 3.3235)ml * C_2 = 5ppb \times 111.11$

$C_2 = (5 \times 111.11) / 114.4335 \sim 4.855$, ok.

∴ will not add any additional 233U.

3. Amount of solid to add:

Want M/V = 2.2 $V \tilde{=} 114$ (will calculate for each case)

$M = \frac{22 \times 114.4335}{1000}$ for CIE-D

∴ 0.25175 grams solid.

CIE-C

ml of 233U: $(100.1163 * 5)/4.5 = 111.24\,ml$ | ml of U-nitrate: 2.2538 ml

g of clinoptilolite: $2.2 * (\frac{100.1163 * 2.2538}{1000} + 11.124) = ~~0.2447~~$ MN 10/27/99 $= 2.2 \times \frac{113.4941}{1000}$

$= 0.249.68702$

UMIE-C $= 0.2497$ grams.

ml 233U: $120.1083 \times 5/45 = 13.3454\,ml$

ml U nitrate: $\frac{100}{330.883} * (120.1083 + 13.3454) = 40.3326$

$40.3326 - 31.3654 = 8.9672\,ml.$

g uncleaned mont: $2.2 \times (8.9672 + 120.1083 + 13.3454)/1000$

$= 0.3133$ grams.

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CMIE-C: ml ^{233}U: 5 * 125.6781 / 45 = 13.9642 ml

ml U-nitrate: 100/330.883 * (13.9642 + 125.6781) = 42.2029 ml

42.2029 - 31.3192 = 10.8837 ml to add

g cleaned mont: 2.2 * (125.6781 + 10.8837 + 13.9642)/1000
= 0.3312 grams.

10/27/99 Actually Added:

	CIE-C	UMIE-C	CMIE-C
ml of ^{233}U	11.1646	13.5230	14.0718
ml of U-nitrate	2.2812	8.9875	10.9178
pH	3.16	3.24	3.26
~~g solid added~~ MN 10/27/99	Addition of Sample:		
mass weighboat	0.5370 tare	0.5234 tare	0.5421 tare
g of Sample	0.2503	0.3132	0.3337
Mass (after sample added)	0.0002	0.0004	~~0.0011~~ MN 10/27/99 0.0011
: Mass Sample Added	~~0.2501~~ MN 10/27/99 0.2501	0.3128	0.3326
pH after	3.87	3.39	4.13

10/27/99 4. Sampling: note: samples taken before solid added.

	CIE-C	UMIE-C	CMIE-C
Sample 1			
Name:	CIE C-1	UMIE-C-1	CMIE-C-1
Mass Bottle + cap	7.3556	7.3056	7.2928
Mass Bottle + cap + Sample:	7.8895	7.8494	7.8116
Sample 2			
Name	CIEC-2	UMIEC-2	CMIE-C-2
Mass Bottle + Cap	7.3148	7.2577	7.3256
Mass Bottle + Cap + Sample	7.8410	~~7.7741~~ MN 10/27/99 7.7741	7.8606

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5. Addition of solid to the XAFS samples, & sampling
10/27/99

	CIE-D	UMIE-D	CMIE-D
pH	3.30	3.28	3.21
Sample Name	CIED-1	UMIE-D-1	CMIED-1
Mass Bottle+Cap	7.2903	7.3109	7.2930
Mass Bottle+Cap+Sample	7.8080	7.8179	7.8316
Sample 2	CIED-2	UMIED-2	CMIED-2
Mass Bottle+Cap	7.2541	7.2759	7.2732
Mass Bottle+Cap+Sample	7.7874	7.8114	7.8175
Add the Solid			
Mass Weighboat	0.5190	0.5479	0.5209
Mass solid	1.0012	1.0014	1.0023
	0.0004	0.0010	0.0009
pH final	3.63	3.32	3.32

All solutions were attached to bubblers and placed on the gyratory shaker. Caps were secured, bubblers were started, and the gyratory shaker was started.

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10/29/99

pH checks and adjustments

	CIE-C	UMIE-C	CMIE-C	CIE-D	UMIE-D	CMIE-D
Date:	10/29/99	10/29/99	10/29/99	10/29/99	10/29/99	10/29/99
pH	4.71	4.72	4.69	5.02	3.77	5.22
# of acid/Base added to solution	0.5ml of 0.1m HNO_3	1ml of 0.05m HNO_3	0.5ml of 0.1m HNO_3	1.5ml of 0.1m HNO_3 AND 1.5ml of 0.05 m HNO_3	0.1ml of 0.1 m HNO_3	0.2ml of 0.1m HNO_3
pH final	3.21	3.29	3.02	3.25	3.22	3.25
	10/30/99 ~2pm for all			MN 10/30/99		
pH	3.79	3.38	3.21	4.55	3.31	3.73
# of acid added	0.8ml of 0.05m HNO_3	0.2ml of 0.05m HNO_3	none added	0.8ml of ~~0.05~~ 0.1 m HNO_3 MN 10/30/99	0.1ml of ~~0.5~~ 0.05m HNO_3 MN 10/30/99	0.5ml of 0.1m HNO_3
pH final	3.27	3.29	/	3.26	3.21	3.21
	11/1/99 ~2pm				No entry	
pH	3.61	3.36	3.30	3.90	~~3.32~~ MN 10/1/99	3.44
# of HNO_3 added	0.6ml of 0.05m HNO_3	0.0ml of 0.05m HNO_3	none added	0.6ml of 0.05m HNO_3	0.1ml of ~~0.05~~ m HNO_3	0.2ml of 0.05m HNO_3
pH final	3.28	3.30	/	3.28	~~3.30~~	3.25
	11/2/99 ~1:30 a.m.				MN 11/2/99	
pH	3.47	3.38	3.35	3.55	~~3.38~~ 3.46	3.33
# of HNO_3 added	0.5ml of 0.05m HNO_3	0.3ml of 0.05m HNO_3	0.2ml of 0.05 m HNO_3	0.5ml of 0.1m HNO_3	0.3ml of ~~0.05~~ 0.1m HNO_3 0.4ml of 0.1m HNO_3	0.2ml of 0.1m HNO_3
pH final	3.23	3.26	3.28	3.17	3.26 3.23	3.20
	11/3/99 ~7pm Last day of experiment!					
pH	3.39	3.30	3.23	3.42	3.27	3.25

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10/29/99

Sampling

note: acid is 0.02 m HNO_3 unless otherwise noted.

	CIE-C	CMIE-C	UMIE-C	CIE-D	CMIE-D	UMIE-D
Date:	10/29/99	10/29/98	10/29/99			
Sample Name:	CIE-C 3	CMIEC-3	UMIEC3			
Mass Bottle+Cap:	7.3563	7.2865	7.2862			
Mass Bottle+cap+Sample	8.7539	8.5490	8.4191			
Mass Sample:	1.3976	1.2625	1.1329			
Date	10/29/99	10/29/99	10/29/99			
Sample Name	CIEC-4 MN 10/29/99	CMIEC-3 MN 10/29/99	UMIEC-4 MN 10/29/99			
Mass Bottle+Cap	7.2824	7.3053	7.2842			
Mass Bottle+cap+Sample	8.5272	8.7276	8.1113			
Mass Sample:	1.2448	1.4223	0.8271			
Date	11/1/99 ~4:30pm		MN 11/1/99			
Sample Name	CIEC5	~~7.3024~~ CMIEC5	UMIEC5	CIED3	CMIED3	UMIED3
Mass Bottle+cap	7.2803	7.3024	7.2232	4.0689	4.0646	4.0345
Mass Bottle+cap+sample	8.4260	8.5989	8.4676	6.0843	4.9494	5.7270
Mass Bottle+cap+sample+acid	8.9233	9.0781	8.9586	6.5378	5.4227	6.2089
Date	11/1/99 ~4:30pm					
Sample Name	CIEC6	CMIEC6	UMIEC6	CIED4	CMIED4	UMIED4
Mass Bottle+Cap	7.2913	7.2879	7.2830	4.0524	4.0702	4.0606
Mass Bottle+cap+sample	8.5731	8.4896	8.5307	5.0905	4.9813	5.0853
Mass Bottle+cap+sample+acid	9.0476	8.9851	9.0262	5.5787	5.4574	5.5644
Date	11/3/99 ~7pm Last Day of Experiment					
Name	CIEC7	CMIEC7	UMIEC7	CIED5	CMIED5	UMIED5
Mass Bottle+cap	7.3592	7.2838	7.2746	7.2793	7.2647	7.2396
Mass Bottle+cap+acid	7.8533	7.7731	7.7599	Bottle+cap MN 11/3/99 9.2169	9.5478	9.3449
Mass Bottle+cap+acid+Sample	9.0401	8.8552	8.9941	Bottle+cap+acid+sample 9.6567	9.9205	9.8449
Date	11/3/99 ~7pm Last Day of Experiment					
Name	CIEC8	CMIEC8	UMIEC8	CIED6	CMIED6	UMIED6
Mass Bottle+cap	7.2284	7.2615	7.2773	Bottle+cap 7.2138	7.2991	7.4030
Mass Bottle+cap+acid	7.7811	7.7230	7.7667	Bottle+cap+sample 9.4617	9.7583	9.7356
Mass Bottle+cap+acid+sample	9.1422	9.2728	9.1425	Bottle+cap+sample+acid 9.8191	10.2191	10.1178

11/3/99
M. Nugent

Take-down of experiment : 11/3/99

Stopped bubbling, waited for minerals to settle, removed bubblers & rinsed them.
~~###~~ MN 11/3/99 Ultracentrifuged solutions in 50ml increments to seperate solids from liquids.
Note, the 'D' samples were not separated.

M. Nugent
11/3/99

M. Nugent 11/17/99

Surface Area Analysis for Cleaned Montmorillonite

M. Nugent 11/17/99

These surface area analyses are for the montmorillonite cleaned according to pages 9-11 of this scientific notebook, CNWRA Controlled copy #371.

Sample Name:

Sample 1 = CMont1 = CM1
2 = CMont2 = CM2
3 = CMont3 = CM3

Mass weighboat: 0.5206 g
tare to 0.0000 g
Mass of sample: 0.2391 g

This mass was not used for surface area analyses. The masses used are recorded in the pages pasted in on pages 45-49 of this notebook, CNWRA Scientific Notebook controlled copy MW 11/17/99 Copy #370.

MW 11/17/99

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-1	Start Date	07/23/89
Customer	CNWRA	Start Time	00:24:53
Operator	NUGENT	Elapsed Time	39 min
Sample Wt	0.1784 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Summary

Surface Area Report

BET Surface area	107.13 sq.m/g
Correlation Coefficient	0.99982

Note, surface area analyses set (CMont 1, CMont 2, and CMont 3) were analyzed by B. Cherrington in the Geochemistry Lab, Division 20, CNWRA/SWRI. A Coulter SA 3100 surface area analyzer and the appropriate glassware were used.

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

M. Nugent
11/17/99

Serial No.	w46020	Software Version	2.11
Sample ID	CM-1	Start Date	07/23/89
Customer	CMWRA	Start Time	00:24:53
Operator	NUGENT	Elapsed Time	39 min
Sample Wt	0.1784 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Surface Area Report

BET Surface area 107.13 sq.m/g

Slope	0.040655
Intercept	-0.000026
C_value	-1541.785
Monolayer Volume	24.6129 cc/g (STP)
Correlation Coefficient	0.99982

One Point BET Surface Area (Ps/Po=0.3) 107.29 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0508	0.002087	25.629
0.0590	0.002403	26.072
0.0647	0.002622	26.360
0.0709	0.002862	26.660
0.0796	0.003198	27.036
0.1000	0.004000	27.776
0.1193	0.004768	28.403
0.1376	0.005506	28.971
0.1601	0.006436	29.609
0.1807	0.007314	30.153
0.1993	0.008128	30.627
0.2023	0.008257	30.722

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.002006	26.232
0.0800	0.003226	26.954
0.1200	0.004852	28.103
0.1600	0.006479	29.401
0.2000	0.008105	30.846

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

M. Nugent
11/17/99

Serial No.	w46020	Software Version	2.11
Sample ID	CM-1	Start Date	07/23/89
Customer	CMWRA	Start Time	00:24:53
Operator	NUGENT	Elapsed Time	39 min
Sample Wt	0.1784 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Isotherm Data

Freespace Calculation

Slope	0.03582
Intercept	-0.006211
Correlation Coefficient	1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	757.61
0.0000	2.418	0.002	757.53
0.0000	4.837	0.024	757.71
0.0001	7.656	0.077	757.71
0.0003	10.786	0.215	757.76
0.0007	14.210	0.524	757.90
0.0019	17.865	1.431	757.84
0.0053	20.524	4.019	757.92
0.0130	22.434	9.833	757.78
0.0238	23.733	18.022	757.66
0.0354	24.660	26.835	757.72
0.0426	25.136	32.281	757.76
0.0508	25.629	38.459	757.63
0.0590	26.072	44.685	757.82
0.0647	26.360	48.989	757.67
0.0709	26.660	53.720	757.71
0.0796	27.036	60.297	757.66
0.1000	27.776	75.754	757.55
0.1193	28.403	90.353	757.47
0.1376	28.971	104.199	757.44
0.1601	29.609	121.232	757.35
0.1807	30.153	136.874	757.48
0.1993	30.627	150.993	757.52
0.2023	30.722	153.253	757.38

M. Nugent
11/17/99

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-2	Start Date	07/23/89
Customer	CNWRA	Start Time	04:44:37
Operator	NUGENT	Elapsed Time	33 min
Sample Wt	0.2139 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Summary

Surface Area Report

BET Surface area 108.97 sq.m/g
Correlation Coefficient 0.99981

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-2	Start Date	07/23/89
Customer	CNWRA	Start Time	04:44:37
Operator	NUGENT	Elapsed Time	33 min
Sample Wt	0.2139 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Surface Area Report

BET Surface area 108.97 sq.m/g

Slope 0.039969
Intercept -0.000028
C_value -1415.723
Monolayer Volume 25.0368 cc/g (STP)
Correlation Coefficient 0.99981

One Point BET Surface Area (Ps/Po=0.3) 109.15 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0485	0.001962	25.988
0.0563	0.002259	26.431
0.0643	0.002561	26.844
0.0699	0.002769	27.119
0.0794	0.003132	27.532
0.1012	0.003974	28.324
0.1184	0.004650	28.894
0.1376	0.005412	29.485
0.1604	0.006339	30.143
0.1805	0.007181	30.678
0.1997	0.008007	31.163
0.2025	0.008122	31.256

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.001970	26.715
0.0800	0.003169	27.437
0.1200	0.004768	28.599
0.1600	0.006367	29.917
0.2000	0.007966	31.385

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

MN
11/17/2000

Serial No.	w46020	Software Version	2.11
Sample ID	CM-2	Start Date	07/23/89
Customer	CNWRA	Start Time	04:44:37
Operator	NUGENT	Elapsed Time	33 min
Sample Wt	0.2139 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Isotherm Data

Freespace Calculation

Slope 0.03634
Intercept -0.005921
Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	755.79
0.0000	2.013	0.001	755.73
0.0000	4.018	0.011	755.83
0.0000	6.372	0.034	755.90
0.0001	8.998	0.097	755.89
0.0003	11.904	0.252	755.89
0.0008	15.107	0.574	755.81
0.0021	18.532	1.561	755.75
0.0054	20.945	4.091	755.71
0.0126	22.799	9.500	755.79
0.0232	24.124	17.520	755.77
0.0339	25.018	25.656	756.04
0.0407	25.502	30.809	756.18
0.0485	25.988	36.692	756.21
0.0563	26.431	42.606	756.16
0.0643	26.844	48.642	756.15
0.0699	27.119	52.819	756.10
0.0794	27.532	60.017	756.03
0.1012	28.324	76.500	756.14
0.1184	28.894	89.538	755.95
0.1376	29.485	104.043	756.03
0.1604	30.143	121.281	756.04
0.1805	30.678	136.473	755.96
0.1997	31.163	150.983	756.04
0.2025	31.256	153.062	755.96

M. Nugent
11/17/99

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-3	Start Date	07/23/89
Customer	CNWRA	Start Time	05:43:09
Operator	NUGENT	Elapsed Time	37 min
Sample Wt	0.2583 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Summary

Surface Area Report

BET Surface area 109.91 sq.m/g
Correlation Coefficient 0.99980

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-3	Start Date	07/23/89
Customer	CNWRA	Start Time	05:43:09
Operator	NUGENT	Elapsed Time	37 min
Sample Wt	0.2583 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Surface Area Report

BET Surface area 109.91 sq.m/g

Slope 0.039637
Intercept -0.000036
C_value -1085.826
Monolayer Volume 25.2524 cc/g (STP)
Correlation Coefficient 0.99980

One Point BET Surface Area (Ps/Po=0.3) 110.15 sq.m/g

Analysis Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0527	0.002101	26.455
0.0603	0.002388	26.866
0.0656	0.002586	27.143
0.0694	0.002728	27.339
0.0799	0.003124	27.789
0.1027	0.003997	28.620
0.1161	0.004516	29.074
0.1190	0.004624	29.195
0.1400	0.005456	29.830
0.1658	0.006503	30.562
0.1784	0.007029	30.903
0.1989	0.007901	31.414
0.2017	0.008020	31.510

Interpolated Data

Ps/Po	BET Function	Vads cc/g(STP)
0.0500	0.001945	27.055
0.0800	0.003134	27.742
0.1200	0.004720	28.891
0.1600	0.006305	30.208
0.2000	0.007891	31.682

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11/17/99

Coulter SA 3100 Surface Area and Pore Size Analyzer
Analysis Report

Serial No.	w46020	Software Version	2.11
Sample ID	CM-3	Start Date	07/23/89
Customer	CNWRA	Start Time	05:43:09
Operator	NUGENT	Elapsed Time	37 min
Sample Wt	0.2583 g	Outgas Time	720 min
Profile	BET5	Outgas Temperature	100 C

Isotherm Data

Freespace Calculation

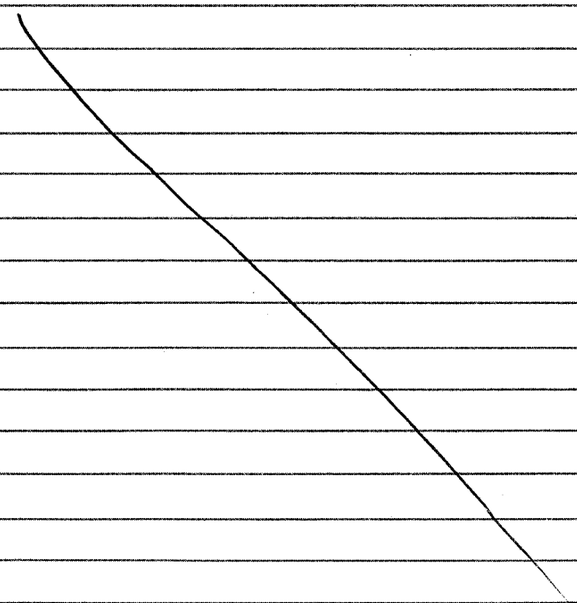
Slope 0.03611
Intercept -0.006347
Correlation Coefficient 1.00000

Isotherm Data Table

Ps/Po	Vads cc/g(STP)	Ps mmHg	Po mmHg
0.0000	0.000	0.001	754.49
0.0000	1.666	0.004	754.45
0.0000	3.329	0.015	754.56
0.0000	5.273	0.031	754.46
0.0001	7.452	0.078	754.66
0.0002	9.877	0.179	754.64
0.0004	12.567	0.329	754.57
0.0009	15.525	0.649	754.60
0.0021	18.715	1.558	754.67
0.0049	20.953	3.700	754.57
0.0113	22.787	8.537	754.44
0.0214	24.165	16.137	754.62
0.0314	25.054	23.665	754.60
0.0379	25.533	28.601	754.56
0.0452	26.014	34.124	754.51
0.0527	26.455	39.733	754.43
0.0603	26.866	45.485	754.41
0.0656	27.143	49.483	754.40
0.0694	27.339	52.362	754.50
0.0799	27.789	60.278	754.52
0.1027	28.620	77.457	754.55
0.1161	29.074	87.575	754.57
0.1190	29.195	89.775	754.73
0.1400	29.830	105.607	754.54
0.1658	30.562	125.087	754.52
0.1784	30.903	134.654	754.61
0.1989	31.414	150.046	754.53
0.2017	31.510	152.196	754.45

Average The 3 results:
107.13
108.97
109.91

Average = 108.67 m^2/grams



M. Nugent
11/30/99

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 02-Nov-1999 11:13
C14 Eff (0-156 keV) = 96.92 %
C14 CHI SQUARE IPA DATA PROCESSED - 02-Nov-1999 11:23
C14 Chi Square = 28.78
H3 IPA DATA PROCESSED - 02-Nov-1999 11:25
H3 Eff (0-18.6 keV) = 65.11 %

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

BKG IPA DATA PROCESSED - 02-Nov-1999 12:35
Bkg (0-18.6 keV) = 16.67 cpm
Bkg (0-156 keV) = 24.80 cpm
C14 E^2/B (1-156 keV) = 492.81
H3 E^2/B (1-18.6 keV) = 256.37

LSA analyses

11/30/99
M. Nugent

These LSA analyses were performed on samples collected in the experiments recorded in this notebook #BTECNWRA.

03 Nov 1999 05:28 — ALPHA/BETA - 1.09 — Page #1
Protocol #:23 — U-233 3% 2 Sigma — User : Alka Jain

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

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	100	0	0.3	16.99
Region B:	100 -	350	0	3.0	3.01
Region C:	0.0 -	2000	0	0.1	25.34

Quench Indicator: SIS
Alpha CPM U-233 1st Vial Bkgnd.
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Count Data Filename: c:\JimP\SDATA23.DAT

DKW 8/14/00

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	FLAG	
1	999.99	16.99	1.53	3.01	3.64	25.34	1.26	145.00	B	Blank
2	24.88	18.26	13.11	175.61	3.05	195.20	3.05	494.66		c1eb3
3	17.84	26.68	11.77	246.09	3.04	273.99	2.99	467.20		c1eb4
4	70.67	27.15	5.90	59.87	3.16	91.95	2.82	412.85		cmieb 3
5	67.03	29.69	5.69	63.30	3.15	99.03	2.77	401.14		cmieb 4
6	42.90	14.64	11.86	100.58	3.09	116.36	3.14	484.34		umieb3
7	42.23	15.78	11.28	102.27	3.09	120.19	3.10	486.24		umieb 4
8	38.96	20.72	9.58	111.15	3.08	133.85	3.03	466.14		cmsab3
9	46.36	19.06	9.35	92.87	3.10	113.12	3.07	471.86		cmsab4
10	267.13	7.86	8.44	13.63	3.75	22.69	3.99	415.81		umsab3
11	247.78	6.81	9.87	14.92	3.68	23.30	4.04	444.48		umsab4
12	89.53	14.73	8.27	46.62	3.20	62.34	3.22	450.06		umseb 3
13	96.46	14.03	8.29	43.06	3.22	57.58	3.27	449.45		umsab4
14	35.38	32.31	7.35	122.60	3.08	166.60	2.80	478.42		c1ec1
15	36.71	30.14	7.57	118.07	3.08	159.39	2.82	479.77		c1ec2
16	16.19	28.35	11.84	271.48	3.03	309.74	2.94	476.39		c1ec3
17	14.45	30.21	12.00	304.88	3.03	344.14	2.94	468.26		c1ec4
18	37.24	35.64	6.71	116.38	3.08	163.43	2.76	457.63		cmiec1
19	36.37	36.60	6.67	119.20	3.08	167.32	2.76	449.01		cmiec2
20	37.89	59.26	4.81	114.27	3.08	192.68	2.50	385.42		cmiec3
21	42.32	51.84	4.95	102.00	3.09	171.85	2.52	395.21		cmiec4
22	36.90	35.37	6.78	117.42	3.08	165.06	2.76	461.45		umiec2
23	35.24	37.75	6.64	123.09	3.07	173.38	2.75	454.21		umiec1
24	28.56	15.50	13.86	152.66	3.06	172.56	3.06	467.67		umiec3
25	40.61	9.93	16.61	106.42	3.09	119.84	3.17	484.68		umiec4
26	35.51	16.33	11.97	122.14	3.08	141.65	3.07	539.38		um8ab1
27	54.97	10.34	13.87	77.83	3.12	89.98	3.24	563.41		umsab2
28	17.56	31.53	10.58	250.12	3.04	287.24	2.94	470.95		umieb1
29	27.74	19.02	12.06	157.30	3.06	179.34	3.03	509.99		umieb2
30	19.73	26.14	11.35	222.28	3.04	253.22	2.97	484.80		Cmsab1
31	36.48	16.43	11.76	118.89	3.08	137.43	3.08	523.41		Cmsab2
32	22.30	26.73	10.52	196.32	3.05	227.71	2.96	500.52		Cmieb1
33	20.39	27.89	10.68	215.09	3.04	247.73	2.96	486.50		Cmieb2
34	25.01	23.24	10.97	174.88	3.05	201.81	2.99	504.32		c1eb1
35	25.40	22.30	11.21	171.95	3.05	197.45	3.00	500.36		c1eb2 umiecsab1
36	29.91	17.15	12.55	145.63	3.06	166.07	3.05	519.32		Csab2
37	23.35	11.15	19.83	187.31	3.05	202.82	3.09	505.46		umiecs

38	23.82	10.22	21.08	183.64	3.05	197.96	3.10	501.72	umiec5
39	29.93	68.71	4.94	148.47	3.06	238.61	2.49	408.25	cmiec6
40	28.83	71.25	4.42	151.20	3.06	247.60	2.49	405.33	cmiec5

04 Nov 1999 13:23 ALPHA/BETA - 1.09 Page #2
Protocol #:23 U-233 3% 2 Sigma User : Alka Jain

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	FLAG
41	14.72	15.76	19.00	298.89	3.03	319.02	3.03	497.53	c1ec6 ← insert c1ec5 as #42
42	16.17	13.69	20.22	271.94	3.03	290.80	3.04	505.34	umieb6 → 43
43	31.77	18.39	11.56	136.87	3.07	157.53	3.05	492.03	umieb5 → 44
44	29.35	20.12	11.25	148.44	3.06	170.57	3.04	487.06	cmieb6 → 45
45	80.26	9.82	12.06	52.36	3.18	63.36	3.36	501.62	cmieb5 → 46
46	91.02	9.29	11.90	45.82	3.21	56.40	3.41	500.88	c1eb6 → 47
47	21.92	21.06	12.57	199.82	3.05	222.47	3.03	506.45	c1eb5 → 48
48	26.23	17.51	13.18	166.49	3.05	185.87	3.06	520.16	umsab6 → 49
49	125.24	12.16	8.22	32.47	3.30	45.30	3.39	457.28	*
50	63.25	25.54	6.50	67.28	3.14	94.09	2.94	410.53	umsab5
51	204.27	9.42	8.12	18.74	3.53	29.43	3.68	439.28	cmsab6
52	211.29	9.11	8.23	18.03	3.55	28.60	3.70	435.92	cmsab5
53	62.74	12.51	11.16	67.82	3.14	80.89	3.24	503.13	c1eb6
54	40.07	18.85	10.13	107.92	3.09	128.86	3.05	485.67	c1eb5

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 05-Nov-1999 06:31
C14 Eff (0-156 keV) = 96.62 %
C14 CHI SQUARE IPA DATA PROCESSED - 05-Nov-1999 06:41
C14 Chi Square = 18.51
H3 IPA DATA PROCESSED - 05-Nov-1999 06:43
H3 Eff (0-18.6 keV) = 65.18 %
H3 CHI SQUARE IPA DATA PROCESSED - 05-Nov-1999 06:53
H3 Chi Square = 17.47
BKG IPA DATA PROCESSED - 05-Nov-1999 07:54
Bkg (0-18.6 keV) = 18.18 cpm
Bkg (0-156 keV) = 25.70 cpm
C14 E^2/B (1-156 keV) = 477.70
H3 E^2/B (1-18.6 keV) = 231.87

MNugent
11/30/99

M. Nugent ~~to~~ mw 11/30/99 11/30/99

3/17/2000
mNugent

Preparation of Uranyl-Loaded Clinoptilolite and Montmorillonite for XAFS analysis and investigate effect of Ionic strength on UO_2 adsorption onto clinoptilolite

Objective: to repeat selected adsorption exps for XAFS analysis (ExpE) and to investigate the effect of ionic strength on adsorption of U onto clinoptilolite during ion-exchange adsorption (Exp f)

All exps will be 450 mL, 1 gram of solid, pH ≅ 3 for ion exchange and pH ≅ 6 for surface adsorption.

Procedure: See page 13-14 of this notebook [CNWRA controlled copy #370], with modifications as noted on page 58 of this notebook CNWRA Controlled Copy #370.

Materials: see pg 15 of this notebook [CNWRA controlled copy #370]

Note, only uncleaned (original) montmorillonite used here.

3/17/2000 1. Prepare a U-nitrate stock solution

Uranyl Nitrate:

Mass - Bottle 56.67 g
- cap 9.41 g
tare

Mass - Nanopure water: 300.04 g

Uranyl Nitrate:
Mass weighboat - 0.5309 g
tare
mass U-nitrate 0.2416 g
Mass-weighboat after U-nitrate added to nanopure 0.0000 g.

Bottle + cap + nanopure + uranyl nitrate = 309.64 g, after unitrate dissolved.

Concentration:

$$\frac{0.2416\ g\ U\text{-nitrate}}{502\ g/mole} \times \frac{1}{0.3004\ L} = 1.604 \times 10^{-3}\ mol/L \rightarrow 381\ ppm\ U.$$

$C_1V_1 = C_2V_2$

for 11ppm → 12.99 mL stock for 450mL sltn.
22ppm → 25.98 mL stock sltn for 450mL.

for 18ppm → 21.259 mL
36ppm → 42.5197 mL

choose higher concentration b/c of potential Rb in samples. Rb interferes w/ U XAFS.

M Nugent 3/17/2000

3/17/2000 M. Nugent

Modifications to Experiments from the procedure described on pages 13-14 of this notebook, CNWRA controlled copy #370.

The samples prepared to investigate the effect of ionic strength on uranyl adsorption for clinoptilolite will be ~~performed~~ performed MN 3/17/2000 at different ionic strengths. There will be a total of Three samples prepared, with 0m NaCl, 0.01, and 0.1m NaCl.

All the reagants and equipment will be The same, and the experiments will be sampled but the solids will not ~~be~~ MN 3/17/2000 be collected.

M Nugent
3/17/2000

Mass measurements: XAFS samples (E)

	cie-E	CSa-E	umie-E	umsa-E	umic-E2
Date	3/17/2000	3/17/2000	3/17/2000	3/17/2000	3/20/2000
Bottle	56.72	56.50	57.23	57.40	56.46
Cap	9.17	9.30	9.30	9.23	9.26
U-nitrates+H	42.54	21.29 ~~452.18~~	42.50 ~~449.97~~	21.27 ~~450.18~~	42.97
U-Nthanopure	453.82	~~449.97~~ MN	~~452.63~~ MN	~~450.63~~	450.72
		DRW 8/14/00	DRW 8/14/00	DRW 8/14/00	

Adding the solid:

Time	6pm	6pm	6pm	6pm	330pm
Date	3/17/2000	3/17/2000	3/17/2000	3/17/2000	3/20/2000
Mass-Weigh Bt.	0.5229	0.0005	0.0006	0.0000	0.0000
Sample, g	1.0111	1.0023	1.0016	1.0037	1.0001
Weight boat, after solid addtn	0.0005	0.0006	0.0007	0.0001	0.0011
∴ Mass of Sample Added grams	1.006	1.0017	1.0009	1.0036	0.9990

note: added clinoptilolite, instead of montmorillonite! Will finish exp. anyway, but redid mont montlonite exp with umie-E2

DRV 8/14/00 ~~Samy~~ Mass measurements - Ionic strength exps (F)

	CieF1	CieF2	CieF3
Date	3/17/2000	3/17/2000	3/17/2000
Bottle	56.91	56.88	57.33
Cap	9.24	9.23	9.29
U-Nitrate s/tn	30.04	30.07	30.10
U-N s/tn + nanopure	450.56	451.64	450.26
NaCl (g)	0.0	0.2394	2.6348

Adding The solid:

Date	3/17/2000	3/17/2000	3/17/2000
Time	6pm	6pm	6pm
Mass-Weighboat	0.0000	0.0012	0.0010
Mass-Sample	1.0003	1.0032	0.8696
Mass-Weighboat after solid addtn.	0.0012	0.0010	0.0003

MNV gent 8/17/2000

MNugent
3/17/2000

Sampling

	cieE	csaE	umieE	umsaE	umieE2
Sample Name ~~Date~~ MN 3/17/2000	cieE1	csaE1	umieE1	umsaE1	umieE2-1
Time	~545pm	545pm	545pm	5:45pm	~~545pm~~ MN 330pm
Mass: Bottle + Cap	4.0479	4.0413	4.0478	4.0401	4.0747
Mass: Bottle + Cap + Sample	8.3060	8.0622	8.8977	8.7200	8.0711
Mass: B+C+S+acid	8.2768→8.6844	8.0471→8.4524	9.2857	9.1176	8.4723 MN
~~Sample Name~~ ~~MN~~ Date MN 3/17/2000	3/17/2000	3/17/2000	3/17/2000	3/17/2000	3/29/2000 MN 3/20/2000

SampleName	cieE2	csaE2	umieE2	umsaE2
Date	3/20/2000	3/20/2000	3/20/2000	3/20/2000
Time	11am	11am	11am	11am
Mass: Bottle+Cap	4.0629	4.0453	4.0419	4.0409
Mass: B+C+Sample	8.0366	7.7607	7.5000	7.6149
Mass: B+C+S+Acid	8.4419	7.7583-8.1638	7.4894-7.8922	8.0173

SampleName	cieE3	csaE3	umieE3	umsaE3	umie-E2-2
Date	3/27/2000	3/27/2000	3/27/2000	3/27/2000	3/27/2000
Time	845am	845am	845am	845am	845am
Mass: Bottle+Cap	4.0359	4.0629	4.0328	4.0551	4.0395
Mass: B+C+Sample	8.0039	8.5454	8.2849	8.4010	8.4714
Mass: B+C+S+Acid	8.4081	8.9490	8.6868	8.8046	8.8736

Sampling (cont')

MNugent
3/17/2000

	cieF1	cieF2	cieF3
	cieF1-1	cieF2-1	cieF3-1
	545pm	545pm	545pm
	4.1620	4.0554	4.0401
	8.5040	8.9487	8.5623
	8.9041	9.3477	8.5491→8.9626
	3/17/2000	3/17/2000	3/17/2000

	cieF1-2	cieF2-2	cieF3-2
	3/20/2000	3/20/2000	3/20/2000
	11am	11am	11am
	4.0290	4.0815	4.0374
	7.7368	7.5770	7.4829
	8.1352	7.9731	7.8850

	cieF1-3	cieF2-3	cieF3-3
	3/27/2000	3/27/2000	3/27/2000
	845am	845am	845am
	4.0387 MN	4.0140	~~4.0616~~ MN 3/28/2000 4.1616
	8.6239	8.0719	8.0239
	MN 9.0282	8.4757	8.4268

M Nugent
3/17/2000

pH measurements

see next page (#65)

	cie-E	csa-E	umie-E	cumsa-E	umie-E2
Date	3/17/2000	3/17/2000	3/17/2000	3/17/2000	~~3/17/2000~~ 3/20/2000
Time	5pm	5pm	5pm	5pm	~~5pm~~ 3pm
pH	4.39	4.57	4.40	4.57	4.34
ml acid added	0.8	0.4	1.0	0.2m	
Type of acid	0.1m HNO_3	0.2m ~~HNO_3~~ MN 3/17/2000 NaOH	0.1m HNO_3	0.2m NaOH	
pH after acid	3.19	7.35	3.10	6.34	
Date	3/20/2000	3/20/2000	3/20/2000	3/20/2000	
Time	noon	noon	noon	noon.	
pH	5.66	7.93	5.64	6.91	
ml of acid added	1.2ml	~~1.2ml~~ MN 0.2ml	1.2ml	0.2ml 0.1m HNO_3 → 5.12	
Type of acid	0.1m HNO_3	0.1m HNO_3	0.1m HNO_3	0.15ml 0.2m NaOH → 5.39	
pH after	3.29	5.83	3.33	~~5.39~~	
Date	3/22/2000	3/22/2000	3/22/2000	3/22/2000	
Time	245pm	245pm	245pm	245pm	
pH	4.44	7.79	4.20	3.78	
ml of acid/Base	0.6ml	0.2ml	0.6ml	0.4ml	
Type acid/Base	0.1m HNO_3	0.1m HNO_3	0.1m HNO_3	0.1m HNO_3	
pH after	3.27	5.76	3.43	3.33	
Date	3/27/2000	3/27/2000	3/27/2000	3/27/2000	
Time	9am	9am	9am	9am	
pH	3.46	5.97	3.46	5.61	
ml acid/Base					
Type acid/base					
pH after					

(pH measurements, cont)

M Nugent
3/17/2000

	umie-E2	cie F1	cie F2	cie F3
	3/20/2000	3/17/2000 ~~3/20/2000~~ MN 3/20/2000	3/17/2000	3/17/2000
	~3pm	~~3pm~~ 5pm	5pm	5pm
	4.34	4.52	4.42	4.46
	0.8ml	1.0	1.0	1.0
	0.1m HNO_3	0.1 ml HNO_3	0.1m HNO_3	0.1 m HNO_3
	3.16	3.12	3.11	3.12
	3/20/2000	3/20/2000	3/20/2000	3/20/2000
belongs here.		noon	noon	noon
		5.87	4.83	4.16
		1.5	1.0	0.7
MN 3/20/00		0.1m HNO_3	0.1m HNO_3	0.1m HNO_3
		3.29	3.30	3.28
Belongs here	3/22/2000	3/22/2000	3/22/2000	3/22/2000 MN 3/22/2000
	245pm	245pm	245pm	~~245pm~~ 245pm
	4.11	4.11	3.73	
MN 3/20/00	0.6ml	0.6ml	0.4ml	
	0.1m HNO_3	0.1m HNO_3	0.1m HNO_3	
	3.28	3.32	3.30	
	3/27/2000			
	9am	3/27/2000	3/27/2000	3/27/2000
	3.24	9am	9am	9am
		3.49	3.60	3.56

M Nugent
3/27/2000

Experiments taken down 3/27/2000. Centrifuged, solution decanted into original bottles, and solids sent to Rich Reeder, SUNY Stony Brook, for XAFS analysis.

Preparation of Samples for ICP analysis

M. Nugent
4/4/2000

The aqueous samples collected in the experiments in this notebook, CNWRA Controlled Copy #371, will be analyzed for uranium by ICP in ~~Division~~ MN 4/4/2000 Division 1. The samples to be analyzed are from the experiments on pages 13-22, 25-31, 35-43, and 57-66 of this notebook (controlled copy #371, CNWRA).

1. Prepare standards to send as "unknowns" to Division 1.
   * prepare 100 ml of ~~100ppm~~ MN 4/4/2000 200ppm uranium solution:

-Mass, Bottle + cap: 24.5644 grams.
-Mass, uranyl nitrate: 0.0525 grams
Uranyl Nitrate: Manufacturer:
Lot #:
-Mass, nanopure water: 100.2071 g
-Add uranyl nitrate.
-Mass, Bottle + cap + nanopure + uranyl nitrate: 124.8257 g

* Prepare standards at 3ppm, 5, 7, 10, 12 ppm.

Standard: ppm U	~~mls of 200ppm~~ Stock slth to add:	Mass, Bottle + cap (g)	Mass of 200ppm Stock slth actually added:	Mass, Stock slth + nanopure	Calc. ppm U
3	0.45	7.9784	0.4461	29.1302	3.8008
5	0.75	8.0388	0.7432	29.8232	6.185
7	1.05	8.0202	1.0045	29.1061	8.5655
10	1.5	8.0285	MN 4/4/2000 ~~1.5~~ 1.4872	~~37.8074~~ MN 4/4/2000 29.7789	12.395
12	1.8	7.9922	1.7850	31.6906	13.98

M. Nugent
4/4/2000

2. Dilute The samples: Dilutions performed 4/6/2000 by M. Nugent

Prepare ~3 ml for analysis. Dilute as necessary. performed by Melissa Nugent on 4/6/2000
should be ~ 10 ppm????

page 1/2

Sample Name	U sample #	Mass Bottle+cap	Mass B+C+ sample	Mass B+C+S+ dilution
cieA2	u2	4.143 4.1457 (MN 4/6/2000)	0.9893	4.5667
cieA3	u3	4.0611	5.0526	9.2159
cieA4	u4	4.0423	5.4713	9.5099
csaA1	u5	4.1513	5.8806	9.6194
csaA2	u6	4.0534	6.5371	9.3934
csaA3	u7	4.0625	6.2740	8.8989
csaA4	u8	4.1389	6.4446	8.1800
cmieA1	u10	4.0745	5.6983	9.6924
cmieA2	u11	4.0724	5.7802	9.2419
cmieA3	u14	4.0526	5.3420	8.3308
cmieA4	u15	4.0412	4.6452	8.3897
cmsaA1	u16	4.0601	6.0194	9.2283
cmsaA2	u17	4.0448	6.7939	9.6676
cmsaA3	u18	4.0504	4.9772	8.9820
umsaA3	u19	4.0303	6.3157	8.4963
umsaA4	u20	4.0546	6.9269	9.0721
umieA1	u21	4.0335	5.7643	9.2446
umieA2	u23	4.0453	6.0385	8.8588
umieA3	u24	4.0639	6.0720	9.2302
umieA4	u25	4.0351	6.6660	9.2221
cied3	u26	4.0399	5.0411	8.7472
cied4	u27	4.0540	5.0495	9.1818
cied5	u28	4.0575	5.6697	9.2733
cied6	u29	4.0393	6.1559	8.5719
cmied3	u30	4.0653	5.0408	7.9646
cmied4	u31	4.0284	5.3088	8.6859
cmied5	u32	4.0670	5.6244	9.2521
cmied6	u33	4.0422	5.7 (MN 4/6/2000) 6.7569	9.2921
umied3	u34	4.0275	5.0233	8.1973
umied5	u37	4.0455	5.0532	8.2518
umied6	u38	4.0382	6.6339	8.7781
cieE1	u39	4.0429	5.7942	9.0252
cieE2	u40	4.0468	6.0521	9.0592
cieE3	u41	4.0633	7.0734	9.1533
csaE1	u42	4.1445	7.1564	9.1597
csaE2	u43	4.1486	8.1089	9.0840

Dilutions, continued

4/4/2000
M. Nugent

performed by M. Nugent, 4/6/2000

page 2/2

Sample Name	U sample #	Mass Bottle+cap	Mass B+C+ sample	Mass B+C+S+ dilution
cieE3	u44	4.0354	8.9031	← Non added
umieE1	u46	4.0536	6.0599	8.9058
umieE2	u47	4.0735	7.8754	9.2631
umieE3	u48	4.0629	8.2250	10.4668
umsaE1	u50	4.0778	6.9316	8.6111
umsaE2	u51	4.0480	7.9830	9.3077
umsaE3	u52	4.0544	8.7847	← non added
cieF11	u53	4.0711	5.5332	9.0904
umieE22	u55	4.0340	6.9858	8.8336
umieE21	u56	4.0441	6.0310	8.9148
cieF12	u58	4.1476	6.1456	8.4136
cieF13	u59	4.0428	6.9603	8.6971
cieF21	u60	4.0593	5.8097	8.8538
cieF22	u61	4.0891	6.1041	8.6317
cieF23	u62	4.1455	7.1656	8.9804
cieF31	u64	4.0678	6.0858	8.6826
cieF32	u65	4.0825	6.1022	8.3627
cieF33	u66	4.0328	7.0401	8.7192

3. Prepare the standards by transferring aliquots of each one into labelled bottles.

ICP Sample #	U, ppm, of standard (approx.)	Mass, Bottle + Cap (g)	Mass, Bottle + Cap + Standard (g)
U9	5	4.0311	5.4879 = mass standard only.
U12	10	4.0266	9.6031
U13	3	4.0615	9.6620
U22	7	4.0369	9.3117
U35	12	4.0420	9.4078
U36	10	4.0421	9.3415
U45	3	4.0412	9.0015
U49	7	4.0441	9.0142
U54	12	4.0488	9.5582
U57	5	~~4.04~~ 4.1420 MN 4/6/2000	9.4131
U63	7	4.0643	9.3116
U67	3	4.0295	9.0092
U68	10	4.0358	9.9968
U69	5	4.0614	9.4947
U70	3	4.0631	9.0364

4. Samples sent to Division 1 on 4/7/2000 for ICP analysis. Results received 4/19/2000. M. Nugent 4/7/2000

SOUTHWEST RESEARCH INSTITUTE

SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute — Client: Division 20

Lab Code: SwRI — Date Received: 04/07/00

Matrix: Water — Project No.: 20.01402.871

Work Order: 17668

Sample ID	Lab System ID	Silicon Result (mg/L)	Silicon RL (mg/L)	Uranium Result (mg/L)	Uranium RL (mg/L)
U1	141584	<0.02	0.02	<0.08	0.08
U2	141595	<0.02	0.02	4.89	0.08
U3	141606	2.82	0.05	4.02	0.2
U4	141617	3.43	0.05	5.24	0.2
U5	141628	<0.05	0.05	4.40	0.2
U6	141639	<0.05	0.05	5.17	0.2
U7	141650	4.67	0.05	0.280	0.2
U8	141652	5.24	0.05	0.363	0.2
U9	141653	<0.05	0.05	5.98	0.2
U10	141585	<0.05	0.05	7.26	0.2
U11	141586	<0.05	0.05	8.06	0.2
U12	141587	<0.1	0.1	12.5	0.4
Duplicate result	141587	<0.1	0.1	12.7	0.4
RPD	141587	0.00%	----	1.79%	----
Spike result	141587	19.9	----	32.1	----
Spike added	141587	20.0	----	20.0	----
Recovery	141587	99.7%	----	97.8%	----
U13	141588	<0.05	0.05	3.76	0.2
U14	141589	6.59	0.05	0.871	0.2
U15	141590	3.60	0.05	0.458	0.2
U16	141591	<0.05	0.05	4.78	0.2
U17	141592	<0.05	0.05	5.82	0.2
U18	141593	3.78	0.05	0.201	0.2
U19	141594	4.61	0.05	1.01	0.2
U20	141596	4.24	0.05	1.15	0.2

RL = Reporting Limit.

SOUTHWEST RESEARCH INSTITUTE

M. Nugent
4/19/2000

SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 04/07/00

Matrix: Water
Project No.: 20.01402.871

Work Order: 17668

Sample ID	Lab System ID	Silicon Result (mg/L)	Silicon RL (mg/L)	Uranium Result (mg/L)	Uranium RL (mg/L)
U21	141597	<0.05	0.05	7.77	0.2
U22	141598	<0.05	0.05	8.54	0.2
U23	141599	<0.05	0.05	9.87	0.2
U24	141600	5.44	0.05	4.39	0.2
U25	141601	6.49	0.05	6.98	0.2
U26	141602	1.28	0.05	17.8	0.2
U27	141603	0.913	0.05	12.9	0.2
U28	141604	3.80	0.05	25.0	0.2
U29	141605	5.40	0.05	39.1	0.2
U30	141607	3.23	0.05	4.49	0.2
U31	141608	3.09	0.05	4.41	0.2
U32	141609	9.32	0.05	12.5	0.2
U33	141610	9.72	0.05	12.9	0.2
U34	141611	1.54	0.05	13.4	0.2
U35	141612	<0.1	0.1	13.8	0.4
Duplicate result	141612	<0.1	0.1	13.8	0.4
RPD	141612	0.00%	----	0.37%	----
Spike result	141612	19.9	----	33.2	----
Spike added	141612	20.0	----	20.0	----
Recovery	141612	99.7%	----	97.0%	----
U36	141613	<0.05	0.05	12.0	0.2
U37	141614	5.26	0.05	22.3	0.2
U38	141615	5.95	0.05	26.9	0.2
U39	141616	<0.05	0.05	11.4	0.2
U40	141618	1.75	0.05	8.07	0.2

RL = Reporting Limit.

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

M. Nugent
4/19/2000

Lab Name: Southwest Research Institute
Client: Division 20

Lab Code: SwRI
Date Received: 04/07/00

Matrix: Water
Project No.: 20.01402.871

Work Order: 17668

Sample ID	Lab System ID	Silicon Result (mg/L)	Silicon RL (mg/L)	Uranium Result (mg/L)	Uranium RL (mg/L)
U41	141619	5.83	0.05	19.6	0.2
U40	141620	<0.05	0.05	9.68	0.2
U43	141621	1.14	0.05	2.14	0.2
U44	141622	5.08	0.05	24.6	0.2
U45	141623	<0.05	0.05	3.78	0.2
U46	141624	<0.1	0.1	13.5	0.4
Duplicate result	141624	<0.1	0.1	13.4	0.4
RPD	141624	0.00%	----	0.57%	----
Spike result	141624	19.2	----	33.2	----
Spike added	141624	20.0	----	20.0	----
Recovery	141624	96.2%	----	98.2%	----
U47	141625	3.78	0.05	14.8	0.2
U48	141626	6.38	0.1	21.3	0.4
U49	141627	<0.05	0.05	8.29	0.2
U50	141629	<0.1	0.1	10.5	0.4
U51	141630	1.40	0.05	3.57	0.2
U52	141631	4.28	0.1	3.75	0.4
U53	141632	<0.05	0.05	6.67	0.2
U54	141633	<0.1	0.1	14.1	0.4
U55	141634	4.58	0.1	10.8	0.4
U56	141635	<0.1	0.1	13.6	0.4
U57	141636	<0.05	0.05	5.95	0.2
U58	141637	2.54	0.1	6.28	0.4
U59	141638	5.93	0.1	15.6	0.4
U60	141640	<0.1	0.1	8.34	0.4

RL = Reporting Limit.

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute
Client: Division 20

M. Nugent
4/19/2000

Lab Code: SwRI

Matrix: Water

Work Order: 17668

Date Received: 04/07/00

Project No.: 20.01402.871

Sample ID	Lab System ID	Silicon Result (mg/L)	Silicon RL (mg/L)	Uranium Result (mg/L)	Uranium RL (mg/L)
U61	141641	1.40	0.1	8.03	0.4
U62	141642	4.56	0.1	14.2	0.4
U63	141643	<0.05	0.05	8.36	0.2
U64	141644	<0.1	0.1	9.23	0.4
U65	141645	0.681	0.1	10.2	0.4
U66	141646	3.96	0.1	1.42	0.4
U67	141647	<0.05	0.05	3.69	0.2
U68	141648	<0.1	0.1	12.4	0.4
Duplicate result	141648	<0.1	0.1	12.4	0.4
RPD	141648	0.00%	----	0.19%	----
Spike result	141648	19.8	----	31.5	----
Spike added	141648	20.0	----	20.0	----
Recovery	141648	99.1%	----	95.7%	----
U69	141649	<0.05	0.05	5.99	0.2
U70	141651	<0.05	0.05	3.66	0.2
LCSW-D12AT1	----	2.00	----	2.06	----
True Value	----	2.00	----	2.00	----
Recovery	----	100.0%	----	103.0%	----
LCSW-D12M2	----	2.11	----	2.08	----
True Value	----	2.00	----	2.00	----
Recovery	----	105.5%	----	104.0%	----
LCSW-D13AT1	----	2.04	----	2.02	----
True Value	----	2.00	----	2.00	----
Recovery	----	102.0%	----	101.0%	----
LCSW-D13AT2	----	2.05	----	2.04	----
True Value	----	2.00	----	2.00	----
Recovery	----	102.5%	----	102.0%	----
PBW-D12AT1	----	<0.01	0.01	<0.04	0.04
PBW-D12M2	----	<0.01	0.01	<0.04	0.04
PBW-D13AT1	----	<0.01	0.01	<0.04	0.04
PBW-D13AT2	----	<0.01	0.01	<0.04	0.04

RL = Reporting Limit.

M. Nugent
4/19/2000

Sample list/chain of custody for the samples sent for ICP.

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
☐ 1 Week
☒ 2 Weeks (Normal)
☐ 3 Weeks
☐ Other: ____

SwRI Contact: Melissa Nugent 6525

Analyses Requested

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)
pH<3 HNO_3

Project is nuclear safety related
10CFR50 Part 21
Appendix B.
Questions, POC Melissa Nugent x6525

ICP U, Si

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers
U1			W	DM	1
U2			W	DM	1
U3			W	DM	1
U4			W	DM	1
U5			W	DM	1
U6			W	DM	1
U7			W	DM	1
U8			W	DM	1
U9			W	DM	1
U10			W	DM	1

Client Name/Address: Melissa Nugent CNWRA/B90 Building 57

Client Purchase Order/Other ID#: Site/Zone ID:

Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water

Sample Types: DM - Dissolved Metals; ER - Equipment Rinsate; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; EB - Environmental Samples; FD - Field Duplicate

Relinquished by (Signature):

Received by (Signature)

Relinquished by (Signature):

Relinquished by Sampler (Signature):

Comments:

Received by (Signature):

Div 01 COC Form 01-01-001, Rev 1/97

Page 1 of 1

MNugent 4/19/2007

Client Name/Address: Melissa Nugent, CNWRA/D20, Building 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: ____

Client Purchase Order/Other ID #: ____ Site/Zone ID: ____

Analyses Requested

SwRI Contact: Melissa Nugent

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U11			W	DM	1	X
U12			W	DM	1	X
U13			W	DM	1	X
U14			W	DM	1	X
U15			W	DM	1	X
U16			W	DM	1	X
U17			W	DM	1	X
U18			W	DM	1	X
U19			W	DM	1	X
U20			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH~3 (HNO_3)

Project is nuclear safety related
10CFR50 Part 21
Appendix B

Questions, POC
Melissa Nugent
x6566

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): [redacted]

Received by (Signature): ____

Relinquished by (Signature): [redacted]

Received by (Signature): [redacted]

Relinquished by (Signature): [redacted]

Comments: ____

Div 01 COC Form 01-01-001, Rev 1/97

Page 2 of 7

MNugent

Client Name/Address: Melissa Nugent, CNWRA/D20, Building 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: ____

Client Purchase Order/Other ID #: ____ Site/Zone ID: ____

Analyses Requested

SwRI Contact: ____

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U21			W	DM	1	X
U22			W	DM	1	X
U23			W	DM	1	X
U24			W	DM	1	X
U25			W	DM	1	X
U26			W	DM	1	X
U27			W	DM	1	X
U28			W	DM	1	X
U29			W	DM	1	X
U30			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH~3 (HNO_3)

Project is nuclear safety related
10CFR50 Part 21
Appendix B

Questions, POC
Melissa Nugent
x6565

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): [redacted]

Received by (Signature): ____

Relinquished by (Signature): [redacted]

Received by (Signature): [redacted]

Relinquished by (Signature): [redacted]

Comments: ____

Div 01 COC Form 01-01-001, Rev 1/97

Page 3 of 7

M. Nugent 4/19/2007

M Nugent 4/19/2000

Client Name/Address: Melissa Nugent SWRI/CNWRA/D20 Building 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: _____

Client Purchase Order/Other ID * | Site/Zone ID | Analyses Requested | SwRI Contact: Melissa Nugent 6885

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U31			W	DM	1	X
U32			W	DM	1	X
U33			W	DM	1	X
U34			W	DM	1	X
U35			W	DM	1	X
U36			W	DM	1	X
U37			W	DM	1	X
U38			W	DM	1	X
U39			W	DM	1	X
U40			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH ~ 3 HNO_3

Project is nuclear safety related. 10CFR50 Pt 21 Appendix B

Questions, POC Melissa Nugent 6885

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): [signature]

Received by (Signature):

Relinquished by (Signature):

Received by (Signature)

Relinquished by (Signature)

Comments:

Div 01 COC Form 01-01-001, Rev 1/97

Page 4 of 7

M Nugent

Client Name/Address: Melissa Nugent CNWRA/D20 Building 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: _____

Client Purchase Order/Other ID * | Site/Zone ID | Analyses Requested | SwRI Contact:

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U41			W	DM	1	X
U42			W	DM	1	X
U43			W	DM	1	X
U44			W	DM	1	X
U45			W	DM	1	X
U46			W	DM	1	X
U47			W	DM	1	X
U48			W	DM	1	X
U49			W	DM	1	X
U50			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH ~ 3 HNO_3

Project is nuclear safety related 10CFR50 Pt 21 Appendix B

Questions, POC Melissa Nugent 6885

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): [signature]

Received by (Signature):

Relinquished by (Signature):

Received by (Signature)

Relinquished by (Signature)

Comments:

Div 01 COC Form 01-01-001, Rev 1/97

Page 5 of 7

M. Nugent 4/19/2000

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M. Nugent 4/20/2010

M Nugent

Client Name/Address: Melissa Nugent CNWRA/DZO Bldng 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: _____

Client Purchase Order/Other ID *: _____ Site/Zone ID: _____

Analyses Requested

SwRI Contact: _____

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U51			W	DM	1	X
U52			W	DM	1	X
U53			W	DM	1	X
U54			W	DM	1	X
U55			W	DM	1	X
U56			W	DM	1	X
U57			W	DM	1	X
U58			W	DM	1	X
U59			W	DM	1	X
U60			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH<3 HNO_3

Project is nuclear safety related
10CFR50 Part 21
Appendix B

Questions POC
Melissa Nugent
6565

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): _____

Received by (Signature): _____

Relinquished by (Signature): _____

Received by (Signature) _____

Relinquished by (Signature) _____

Comments: _____

Div 01 COC Form 01-01-001, Rev 1/97

Page 6 of 7

M Nugent

Client Name/Address: Melissa Nugent CNWRA/DZO Bldng 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute
Chemistry and Chemical Engineering Division
6220 Culebra Road
San Antonio, Texas 78238-5166

Requested Turnaround:
- ☐ 1 Week
- ☒ 2 Weeks (Normal)
- ☐ 3 Weeks
- ☐ Other: _____

Client Purchase Order/Other ID *: _____ Site/Zone ID: _____

Analyses Requested

SwRI Contact: _____

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP
U61			W	DM	1	X
U62			W	DM	1	X
U63			W	DM	1	X
U64			W	DM	1	X
U65			W	DM	1	X
U66			W	DM	1	X
U67			W	DM	1	X
U68			W	DM	1	X
U69			W	DM	1	X
U70			W	DM	1	X

REMARKS

Preservation
a = HCl to pH <2
b = HNO_3 to pH <2
c = H_2SO_4 to pH <2
d = NaOH to pH >12
e = Other (Specify)

pH<3 HNO_3

Project is nuclear safety related
10CFR50 Part 21
Appendix B

Questions POC
Melissa Nugent
6565

Matrix Types: **A** - Air; **P** - Product; **S** - Soil; **T** - Tissue; **W** - Water

Sample Types: **DM** - Dissolved Metals; **ER** - Equipment Rinsate; **FB** - Field Blank; **MSD** - Matrix Spike Duplicate; **MS** - Matrix Spike; **TB** - Trip Blank; **TM** - Total Metals; **ES** - Environmental Samples; **FD** - Field Duplicate

Relinquished by Sampler (Signature): _____

Received by (Signature): _____

Relinquished by (Signature): _____

Received by (Signature) _____

Relinquished by (Signature) _____

Comments: _____

Div 01 COC Form 01-01-001, Rev 1/97

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4/20/2010 M. Nugent

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M.Nugent
6/12/2000

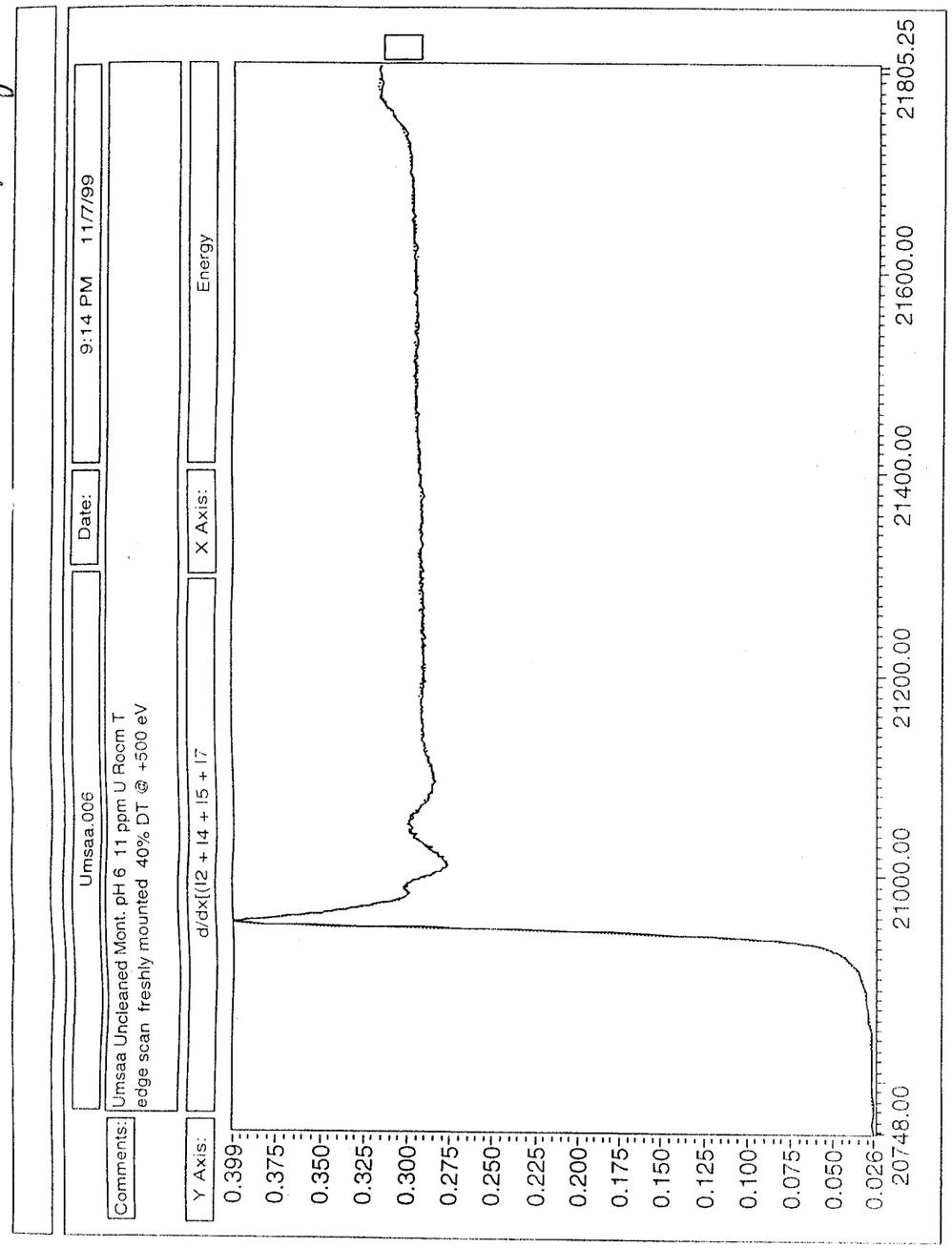
Representative Scans from
XAFS Analysis
at Brookhaven National Lab

Date of Data Collection: 11/7/99
Collected by: MNugent and R. Reeder.

Contact info:
Richard Reeder
Dept. Geosciences
SUNY Stony Brook
Stony Brook NY 11790-2100
631-632-8208

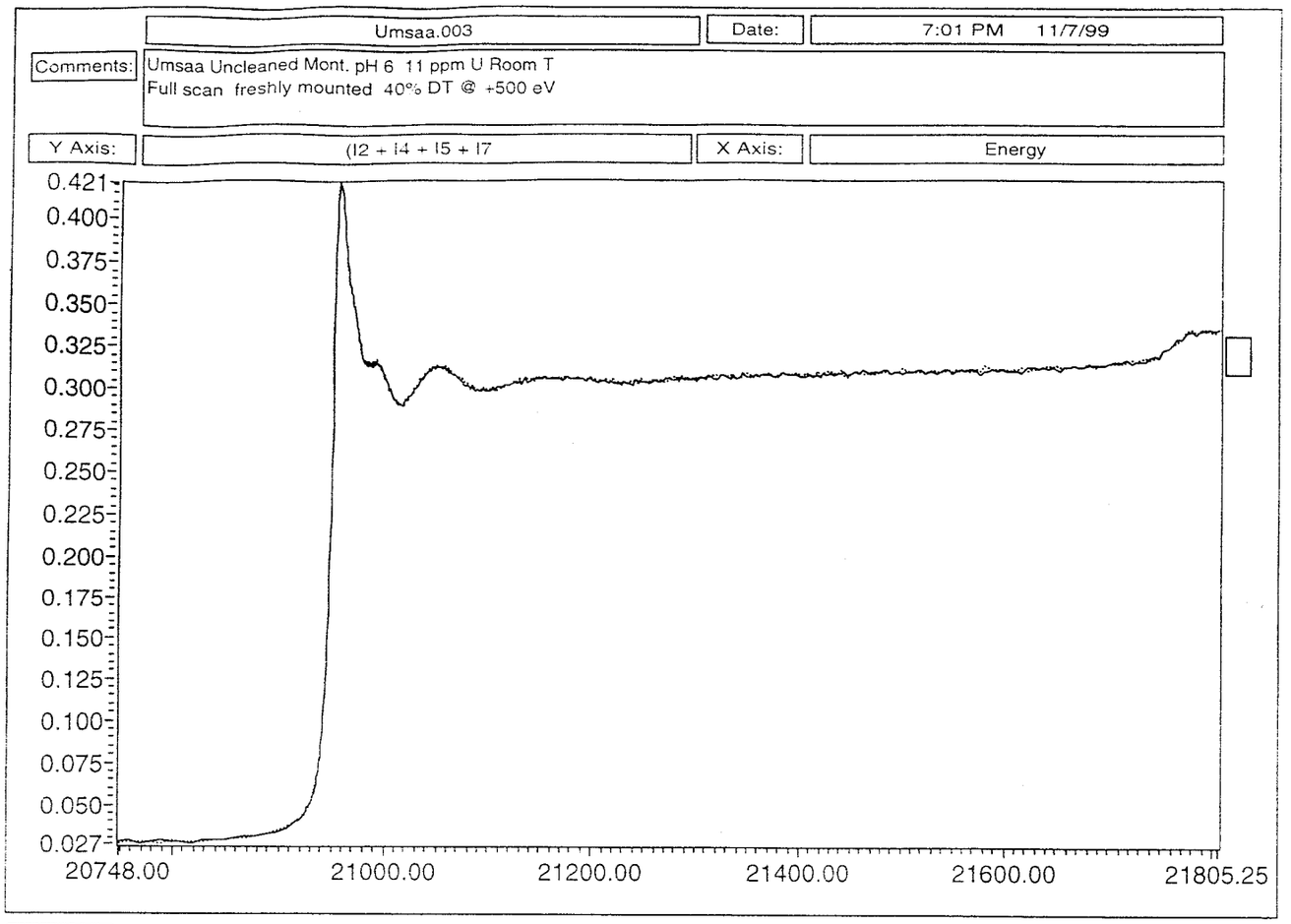
M.Nugent 6/12/2000

M Nugent



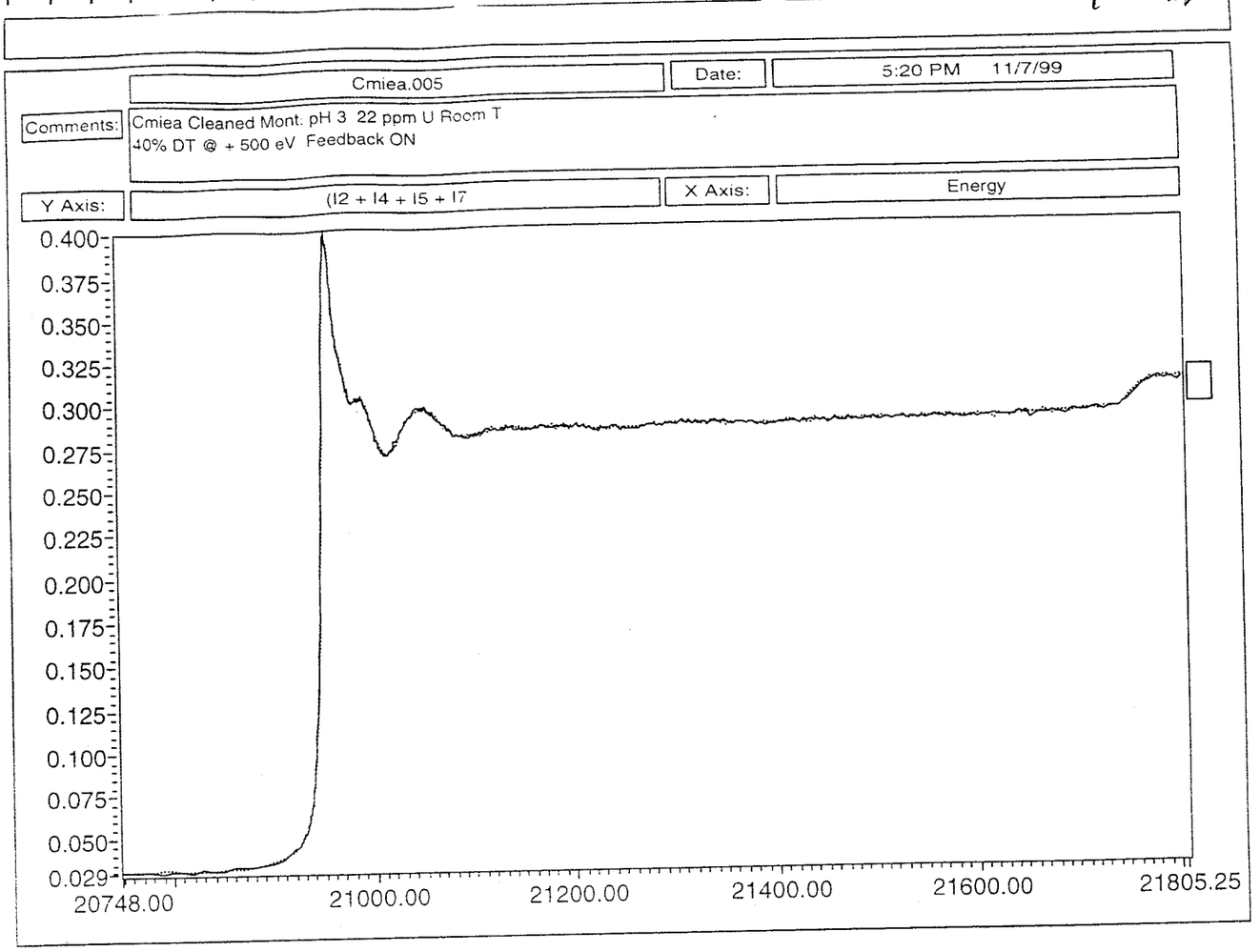
Notes:

M Nygent 6/12/2000



Notes:

M Nygent 6/12/2000



Notes:

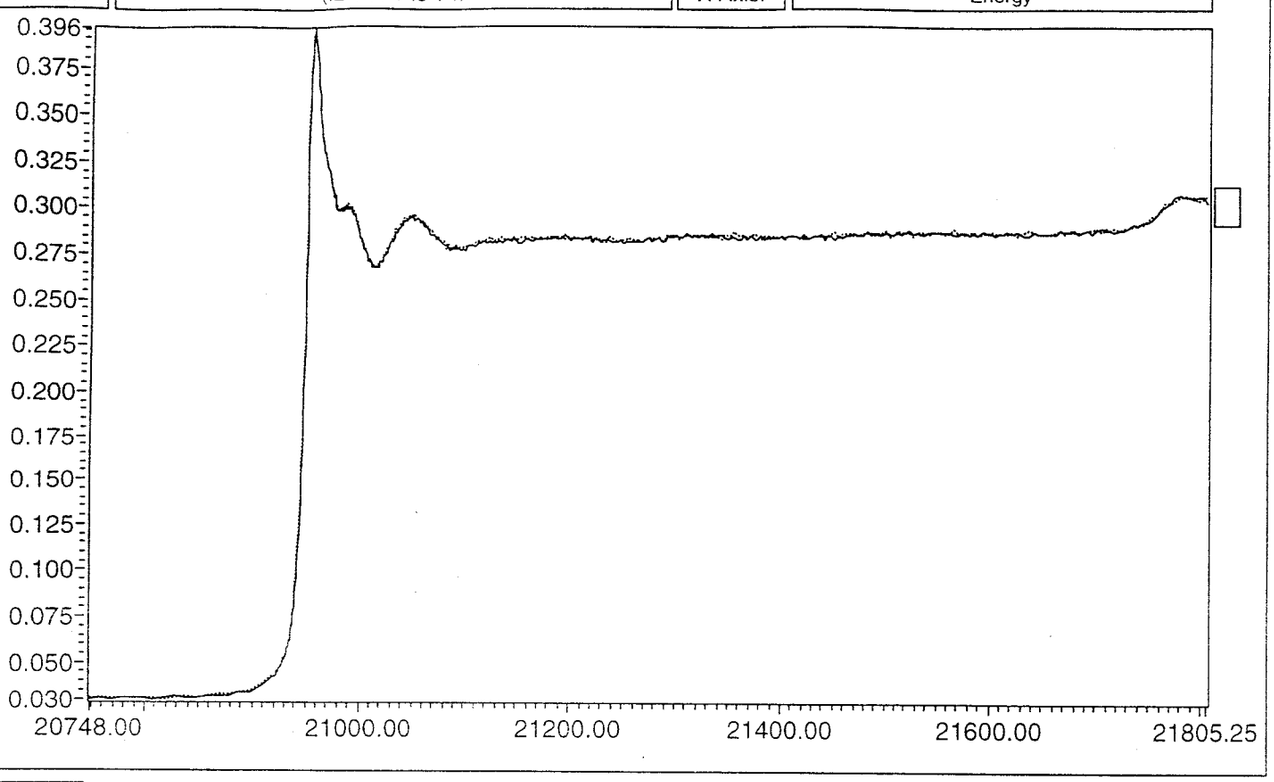
M.Nugent 6/12/2000

MNugent

Cmiea.003 | Date: 4:18 PM 11/7/99

Comments: Cmiea Cleaned Mont. pH 3 22 ppm U Room T
40% DT @ + 500 eV Feedback ON

Y Axis: (I2 + I4 + I5 + I7 | X Axis: Energy



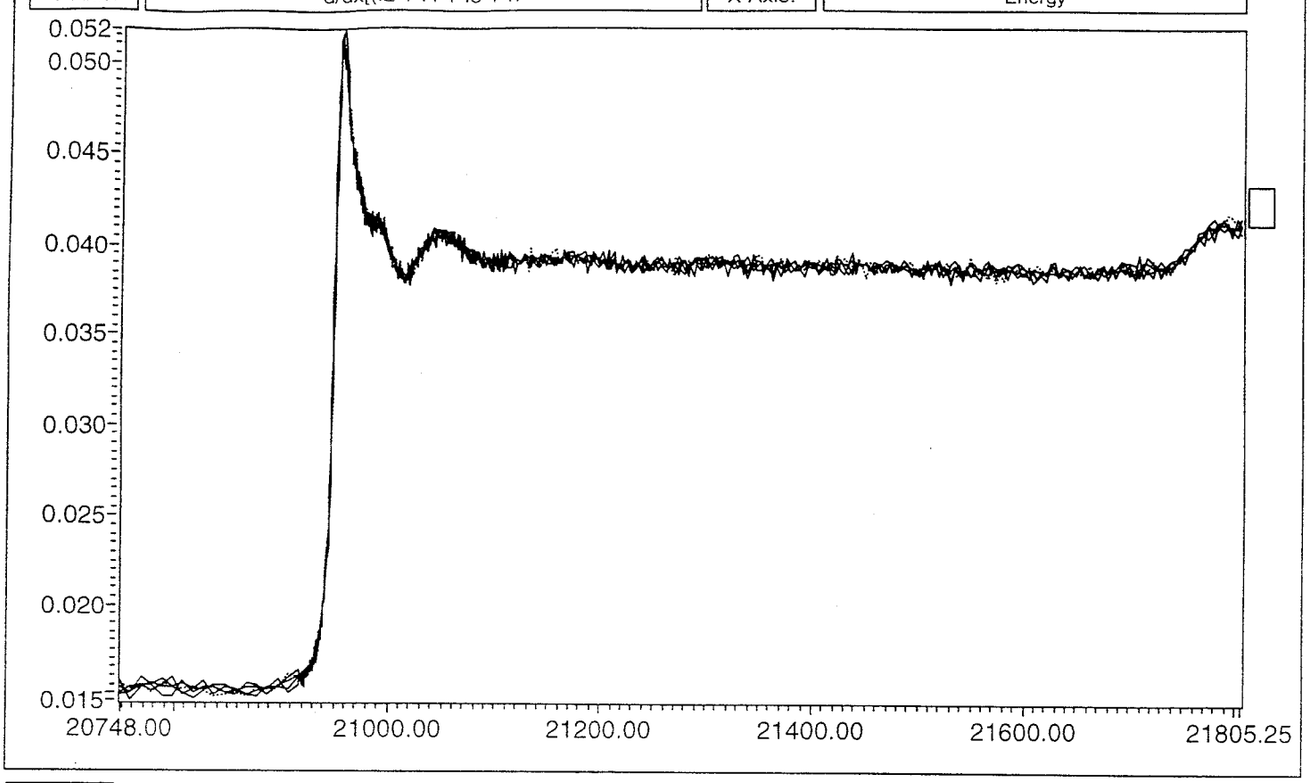
Notes:

MNugent

Ciea.010 | Date: 6:19 AM 11/7/99

Comments: CIEA Clinop. pH 3 22 ppm U Moist Full scan Rm T

Y Axis: d/dx[(I2 + I4 + I5 + I7 | X Axis: Energy



Notes:

M.Nugent 6/12/2000

MNugent

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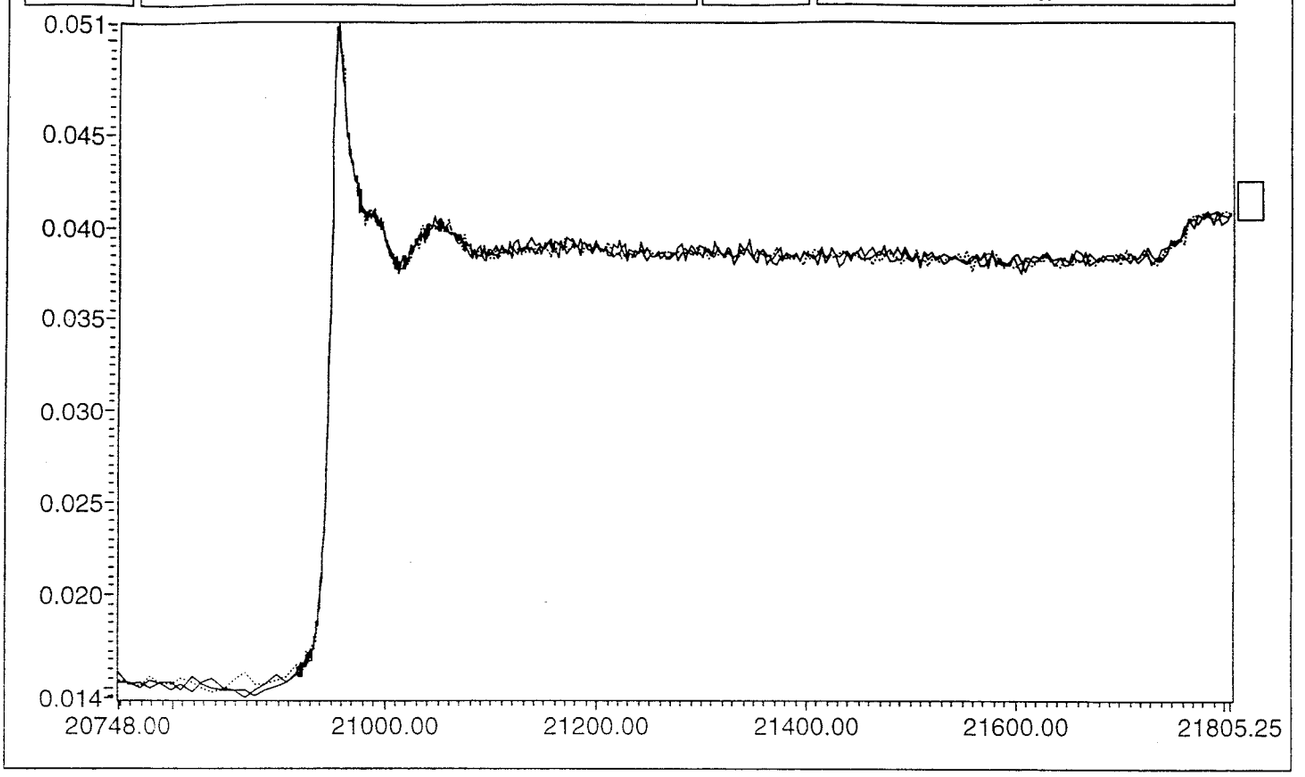
M.Nugent 6/12/2000

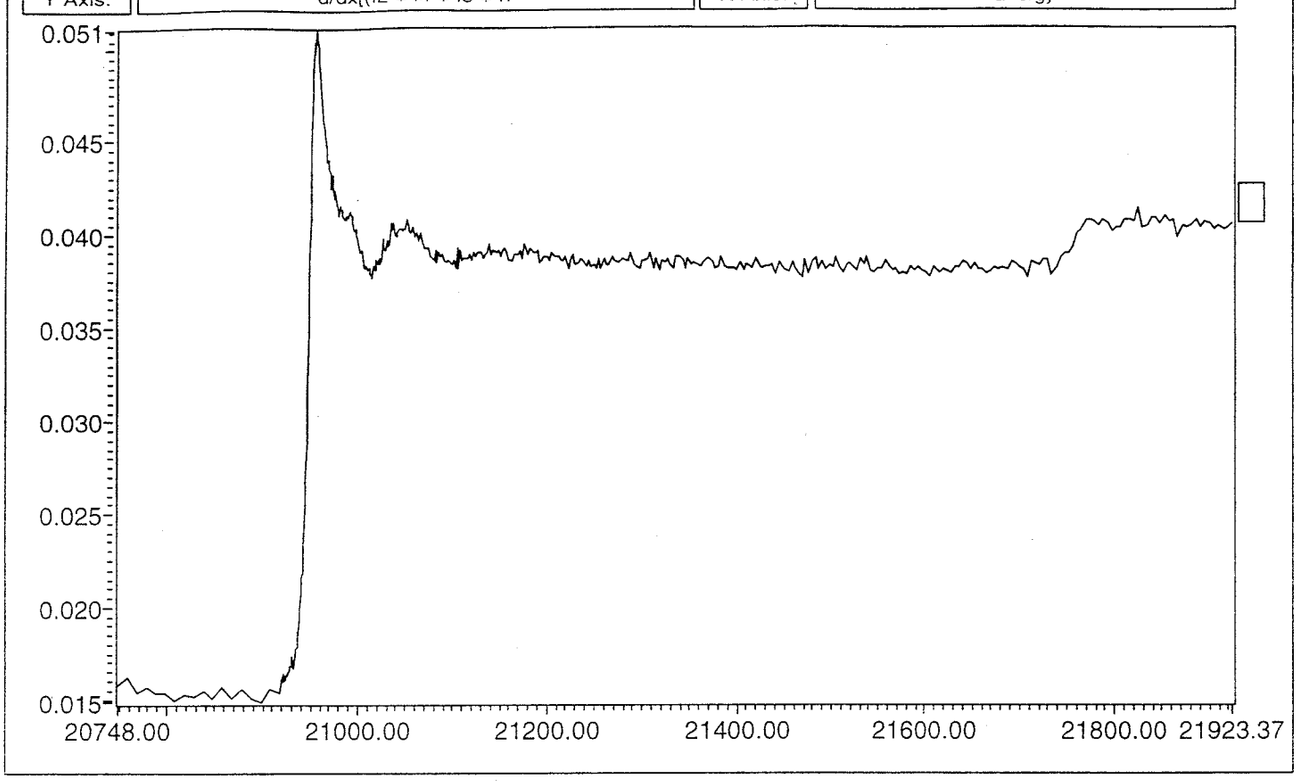
Ciea.005		Date:	3:13 AM 11/7/99
Comments:	CIEA Clinop. pH 3 22 ppm U Moist Full scan Rm T		
Y Axis:	d/dx[(I2 + I4 + I5 + I7	X Axis:	Energy



Notes:

MNugent

Ciea.002		Date:	12:43 AM 11/7/99
Comments:	CIEA Clinop. pH 3 22 ppm U Moist Full scan Rm T		
Y Axis:	d/dx[(I2 + I4 + I5 + I7	X Axis:	Energy



Notes:

M.Nugent 6/12/2000

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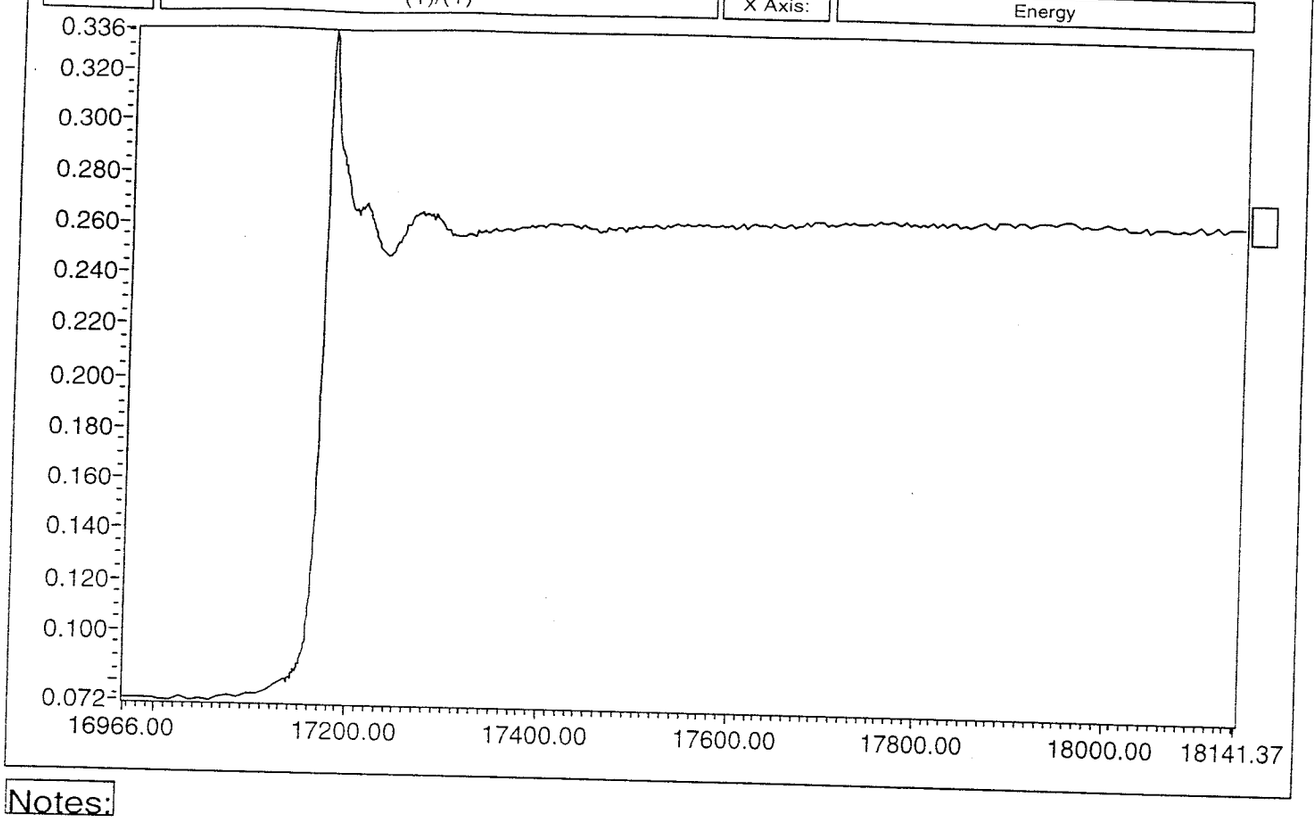
6/12/2000 M Nugent

Cied.002 | Date: 9:58 PM 11/6/99

Comments: Clinp. Ion exchanged pH 3 Room T 100 ppm U Full
NB. Sample appears dry.

Y Axis: (1)/(1) | X Axis: Energy



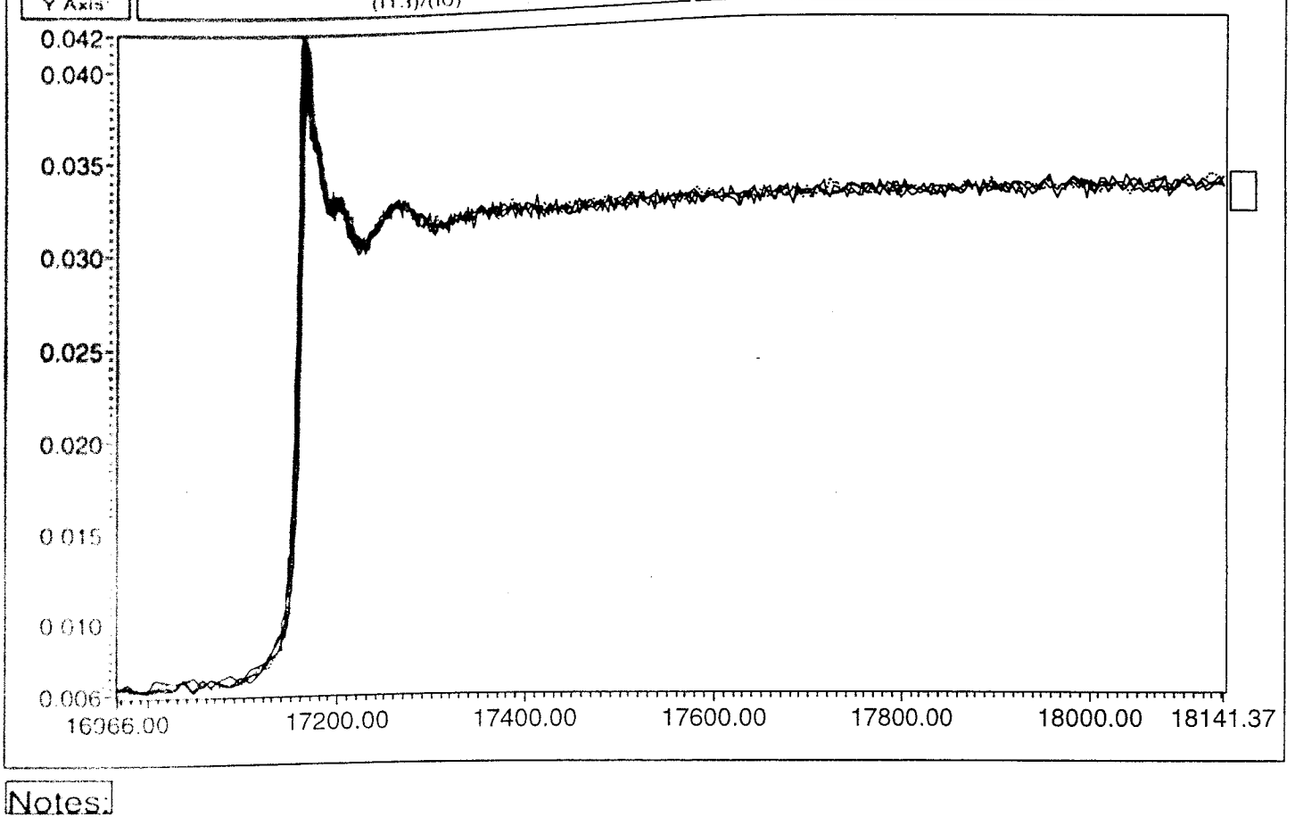
Notes:

M Nugent

csaa.016 | Date: 5:42 AM 11/6/99

Comments: Clinop csaa 11 ppm U pH 6 full scan Room T

Y Axis: (113)/(10) | X Axis: Energy



Notes:

M. Nugent 6/12/2000

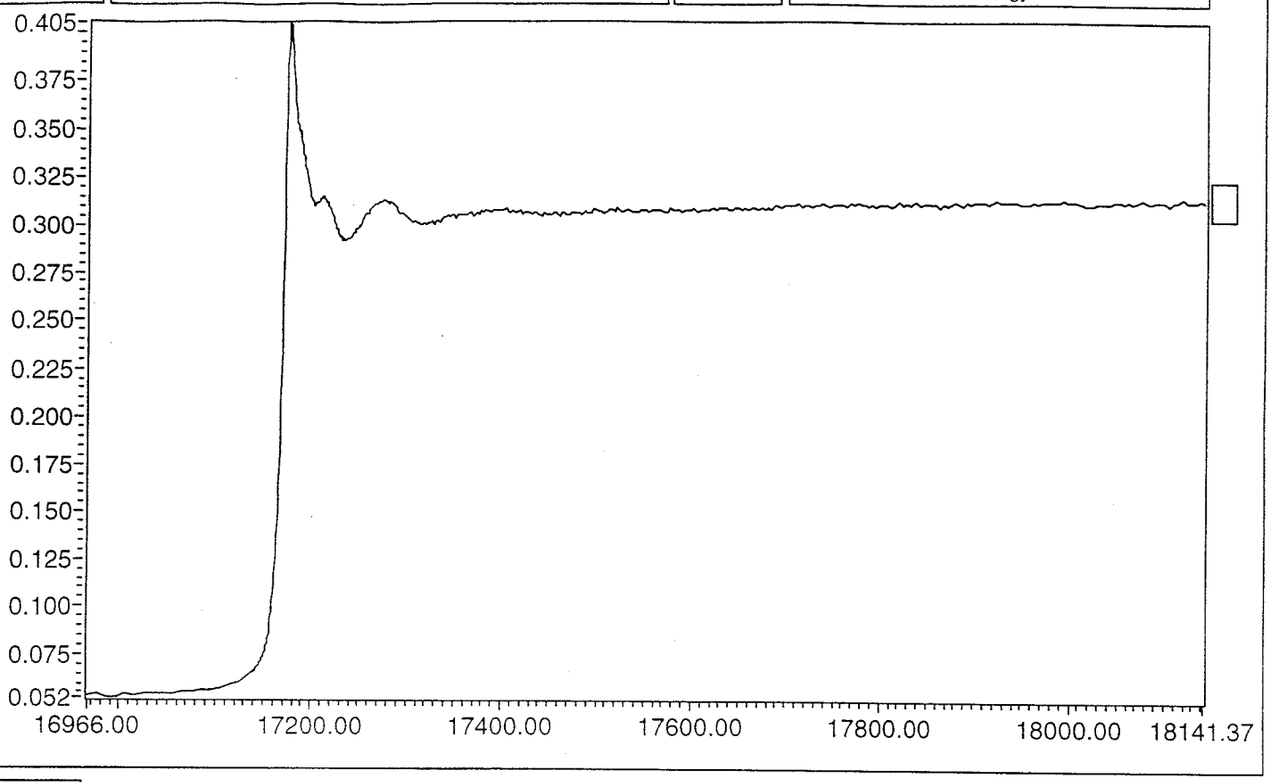
M.Nugent

92 M.Nugent 6/12/2000

csaa.008 | Date: 10:35 PM 11/5/99

Comments: Clinop. csaa 11 ppm U pH 6 full scan Room T

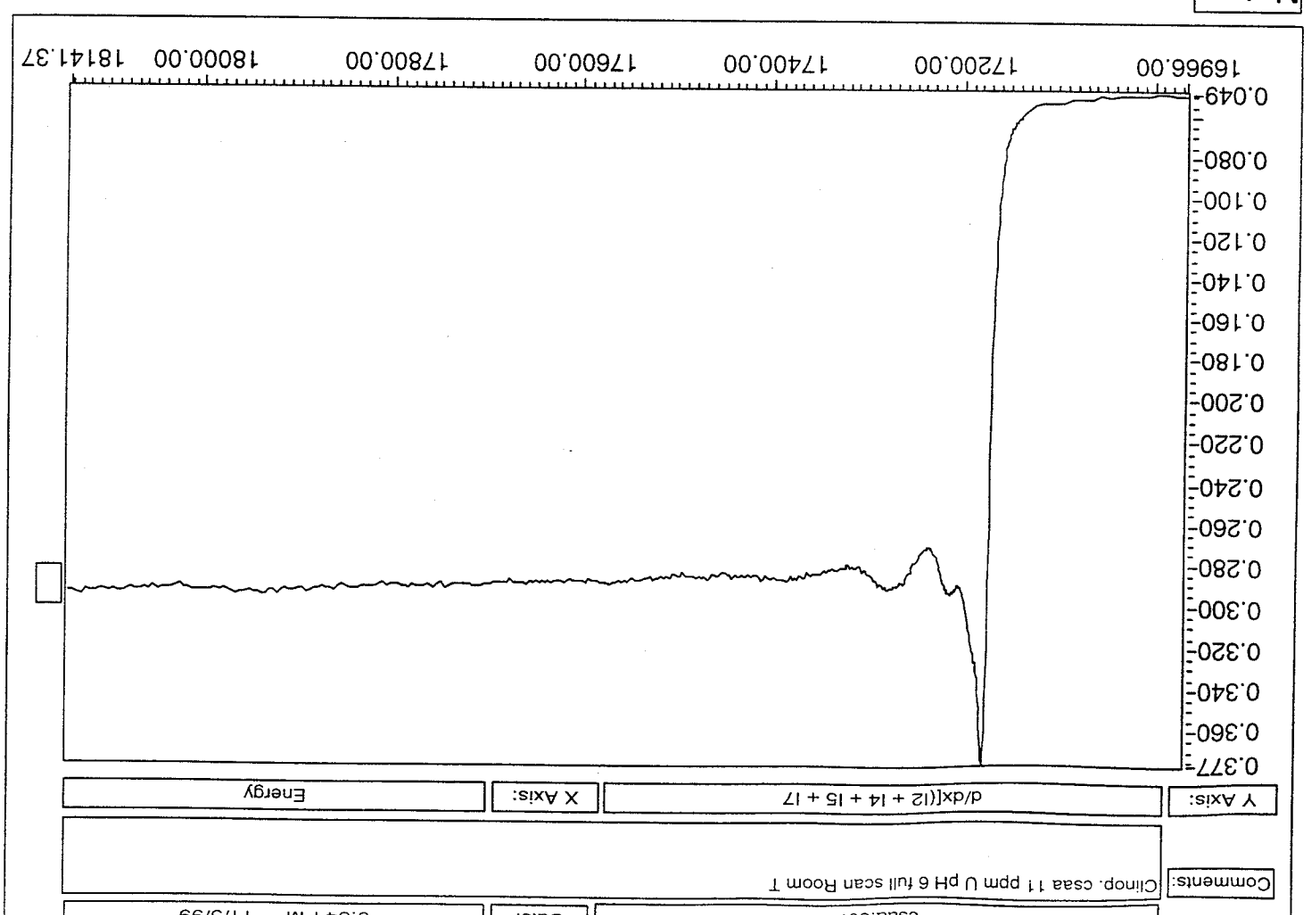
Y Axis: (1)/(1) | X Axis: Energy



Notes:

M.Nugent

Notes:



Y Axis: d/dx[(12 + 14 + 15 + 17 | X Axis: Energy

Comments: Clinop. csaa 11 ppm U pH 6 full scan Room T

csaa.007 | Date: 9:34 PM 11/5/99

93 M.Nugent 6/12/2000

Scientific Notebook No. 370

Pages 94 through 159 intentionally left blank

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M Nugent
6/14/2000

This notebook is in current compliance with QAP-001.

E. C. R

6/27/2000

I have reviewed this notebook again and it still find it to be in compliance with QAP 001. There is sufficient information so that another qualified staff could repeat the activity. E-C-R 8/28/2000