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Q200312260005

Scientific Notebook No. 325: Evolution of the  
Near Field Environment (04/12/1999 through  
05/20/2003)

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Any correspondence should include the code number printed at the bottom of this page as well as the book title stamped at the bottom of the spine.

## Contents

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INVESTIGATOR	SIGNATURE	INITIAL
ROBERT CHERRINGTON	<i>Robert Cherrington</i>	RC
James D. Pridemore	<i>James D. Pridemore</i>	JP
Bradley A. Werling	<i>Bradley A. Werling</i>	BW



4/12/99 *mc*

Evolution of the Near Field Environment  
Project No. 20-1402-561

Initial Entry 4/12/99 by Robert Channing *mc*

This laboratory notebook chronicles the  
investigation of the Evolution of the Near  
Field Environment research project.

*mc* 4/12/99

4/12/99

Procedure - cont'd. re

- e) Use a vacuum pump attached to the gas outlet of the vessel to remove air from the vessel.
- f) Raise and set the temperature of the solution in the reaction vessel to 150 °C using the temperature controller.
- g) Start stirrer and allow reagents to react for about 10 days.

Boiled pH adjusted H<sub>2</sub>O to boil for 25 minutes.

Measured Ca-acetate 25.2022 g  
 U-acetate 42.4015 g  
 Na-metasilicate 28.4003 g

Placed reagents in teflon liner.  
 Added 1302 g of degassed H<sub>2</sub>O

Placed liner w/ reagents into reaction vessel and secured into heating unit. Set temperature to 150 °C and stirring speed to first mark on instrument dial.

Vacuumed off the top layer of air from reaction vessel.

Beginning temp of reagent soln. @ 62 °C.  
 Began ramping temp @ 1030.  
 Reached 150 °C @ 1130  
 Peaked @ 173 °C 110 psi before returning to 150 °C.

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4/12/99

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The synthesis of uranophane  $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 5\text{H}_2\text{O}]$  will be attempted using the following procedure. If successful, the uranophane will be used in thermodynamic and coprecipitation experiments.

## URANOPHANE SYNTHESIS

re 4/12/99

WRITTEN BY: J.D. Prikrýl  
 REVISION NO.: 5

DATE WRITTEN: March 7, 1995  
 DATE REVISED: April 8, 1999

OBJECTIVE: to synthesize uranophane  $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 5\text{H}_2\text{O}]$  for use in thermodynamic and coprecipitation experiments.

EQUIPMENT: Parr Model 4522 stirred reaction vessel (2000 ml capacity)  
 Teflon liners for reaction vessels  
 Parr Model 4843 temperature/pressure controller  
 ORION pH/mV/ISE/°C meter  
 Ross combination pH electrode  
 Analytical balance (Mettler PM 4600)  
 Hot plate  
 Vacuum pump

SUPPLIES: pH buffers (pH= 1,2,4)  
 ultrapure water  
 glassware and plasticware as needed  
 2000 1000 ml erlenmeyer flasks with stoppers  
 4/12/99 fittings, valves, and tubing as necessary  
 No. 5 Whatman filter paper

REAGENTS: Uranyl acetate dihydrate  $[\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ ; MW=424.15 (lot 33749/1) 1094  
 Sodium metasilicate nonahydrate  $(\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O})$ ; MW=284.20 (lot 9228534)  
 Calcium acetate monohydrate  $[\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$ ; MW=176.19 (lot 944231)  
 9 M HCl - diluted from lot 256110

## PROCEDURE:

The procedure for uranophane synthesis is based on Cesbron et al., 1993. Reagents will be mixed in the stoichiometric ratio Ca:U:Si = 2:1:1. Although Cesbron et al. do not give information on the amount of solid and H<sub>2</sub>O used, they state that good crystallinity is obtained if pH is less than 5.

- a) Place 35.2 g Ca-acetate, 42.4 g U-acetate, and 28.4 g sodium metasilicate in a 2000 ml teflon liner.
- b) Transfer approximately 1500 ml of ultrapure H<sub>2</sub>O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- c) Degas the H<sub>2</sub>O by boiling on a hotplate under a fumehood.
- d) Transfer approximately 1300 g of the degassed H<sub>2</sub>O to the teflon liner, place in pressure vessel and seal the vessel.



4/13/99 *MC* Uranophane Synthesis *MC*  
 Procedure - cont'd.

Temperature remaining constant @ 150°C  
 and psi @ ~~70~~<sup>70</sup> - observed @ 0800.  
*MC*  
 4/13/99

4/22/99 Observations:

Turned off heat to reactor @ 1620, left stirrer on.  
 Removed reaction vessel from heater once cooled. Liquid  
 content was yellow, very fine crystals attached to  
 stirrer & lip of vessel.

Decanted vessel liquid into 3 funnels w/ No. 5  
 Whatman filter paper & 500 mL erlenmeyer flasks.  
 Filtered residue are small, consistent in size & color,  
 yellow.

Placed wet crystals into 500 mL beaker & air  
 dried over night under hood.

Beaker labeled - URANOPHANE

Analytical results of synthesized material using XRD  
 instrument in Div 01.

page 7 - 11

*MC*  
 4/22/99

4/14/99 *MC* Heavy Liquid Mineral Separation *MC*

4/14/99 *MC* Procedure

Purpose: Pulverized quartz will be separated from  
 non-quartz impurities utilizing different  
 density liquids.

Materials: 500 mL separatory funnel  
 #4 Whatman filter paper  
 glass funnel  
 (2) 500 mL flasks

Reagents: *N,N*-dimethyl formamide - Lot No. 8816.39 Fisher Chemical  
 Bromoform - Lot No. 980087 Fisher ~~Chemical~~ Chemical  
 Acetone - Lot No. 993009 Fisher Chemical 4/14/99

Procedure: 1. A mixture of heavy liquid Bromoform, and  
 diluent *N,N*-dimethyl formamide, is prepared  
 using the following formula for volume ratio:  

$$V_d = V_h \times \frac{D_h - D_f}{D_h - D_d}$$

where  $V_d$  = volume of diluent  
 $V_h$  = volume of heavy liquid  
 $D_h$  = density of heavy liquid, bromoform  
 $D_f$  = density of liquid desired  
 $D_d$  = density of diluent

2. Transfer heavy liquid mixture to sep. funnel  
 clamped on metal stand. Add some clean polyethylene  
 cover with cap and mix well. Wait 30 min.  
 for complete density separation. Perform under  
 a hood.

ne

3. Open stopper of sep. funnel and release settled heavy impurities into a flask through a funnel + filter paper.
4. Release clean clinoptilolite into separate flask <sup>4/14/99</sup> through funnel + filter. Rinse sep. funnel with acetone and capture + rinse onto filter.
5. Repeat procedure until all clinoptilolite is clean.
6. Remove filter + dry under hood.
7. Rinse clinoptilolite with D.I. water several times + dry in oven.

Calculations:

$$\frac{(2.00)(2.8899 - 2.75)}{(2.75 - 0.93445)} = \frac{27.80}{1.81555}$$

$$= 15.3 \text{ mL}$$

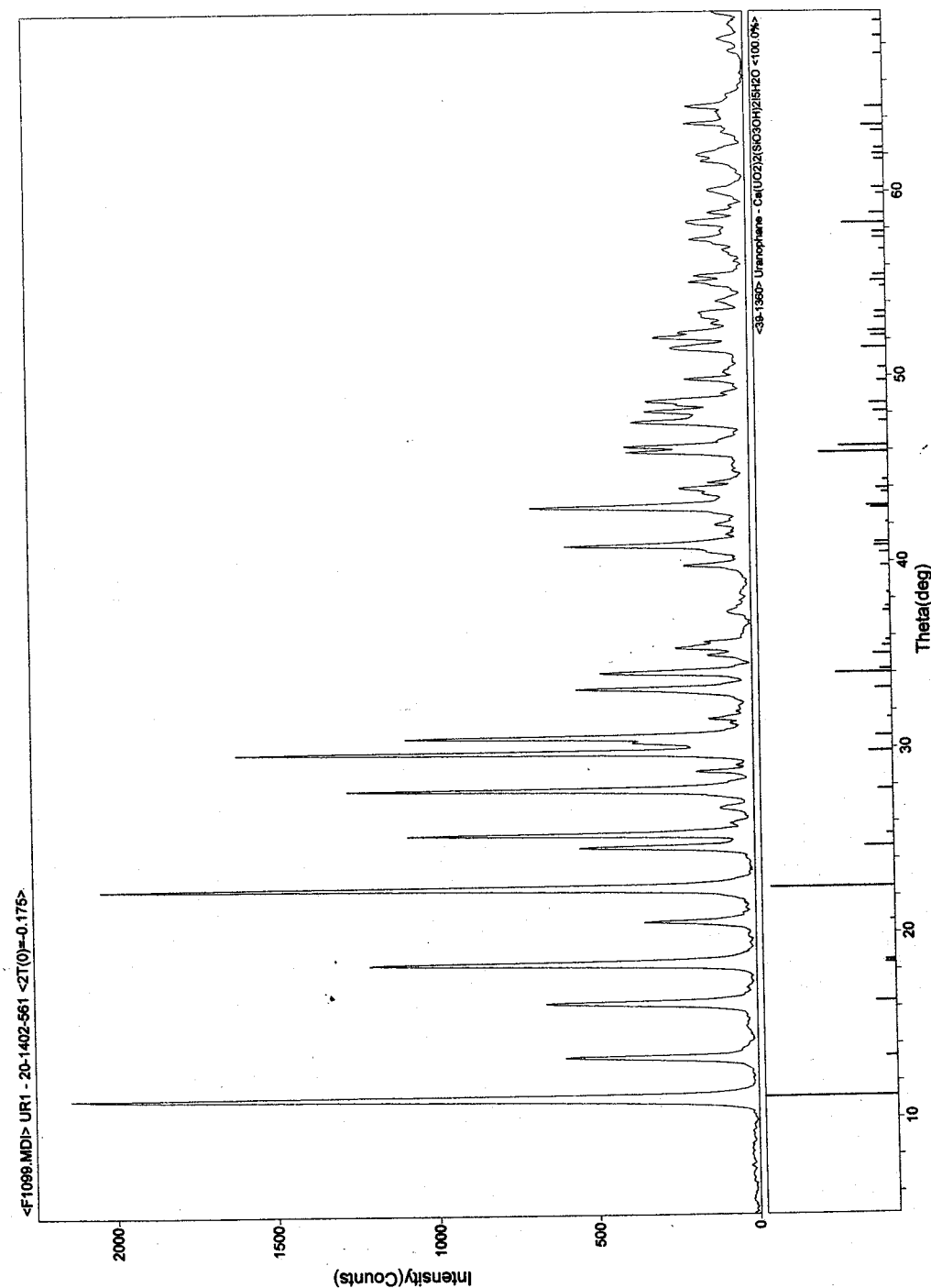
ne  
4/14/99

Uranophane Synthesis - cont'd from pg. 4

Results:

4/29/99

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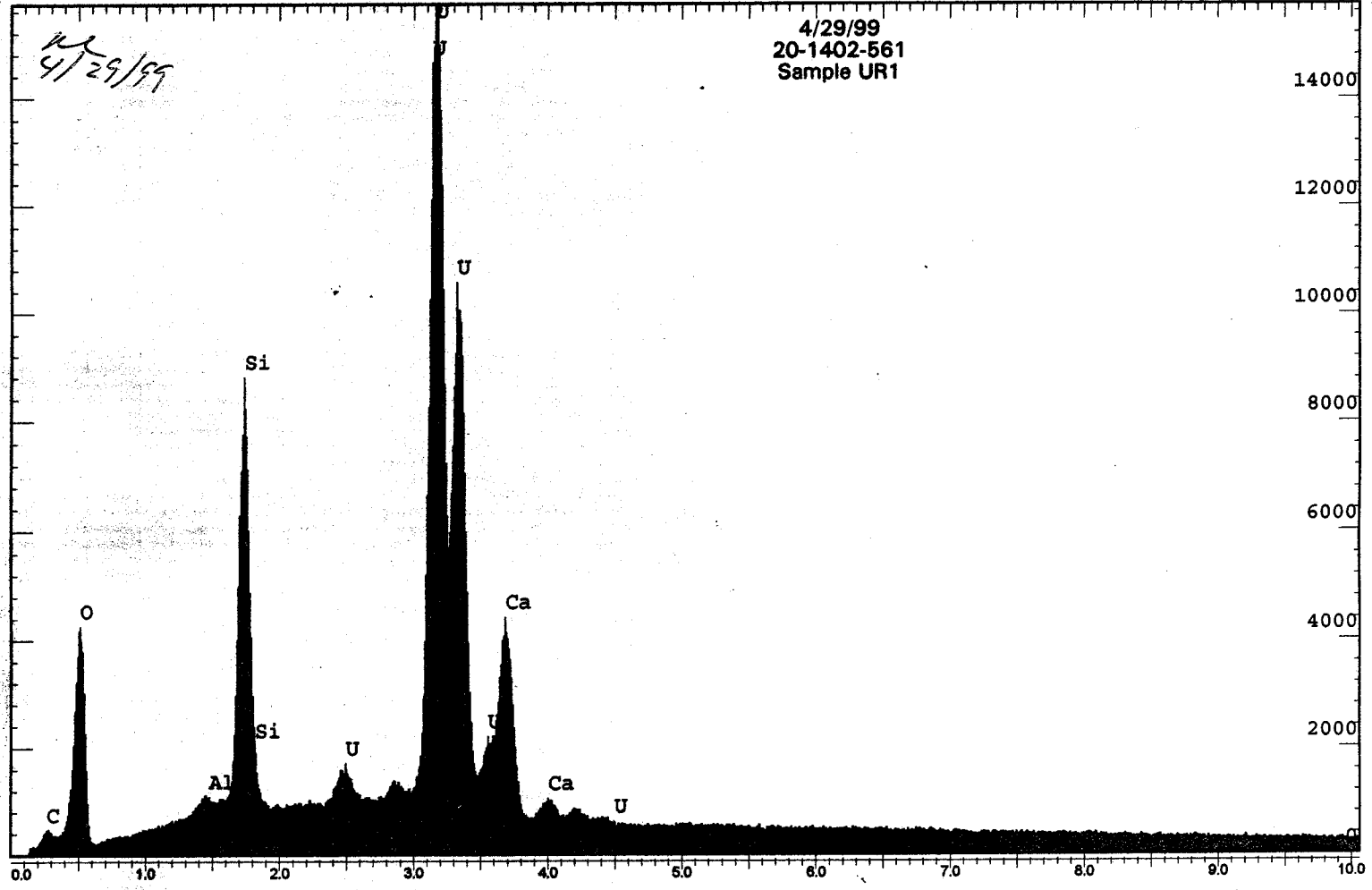
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4/29/99



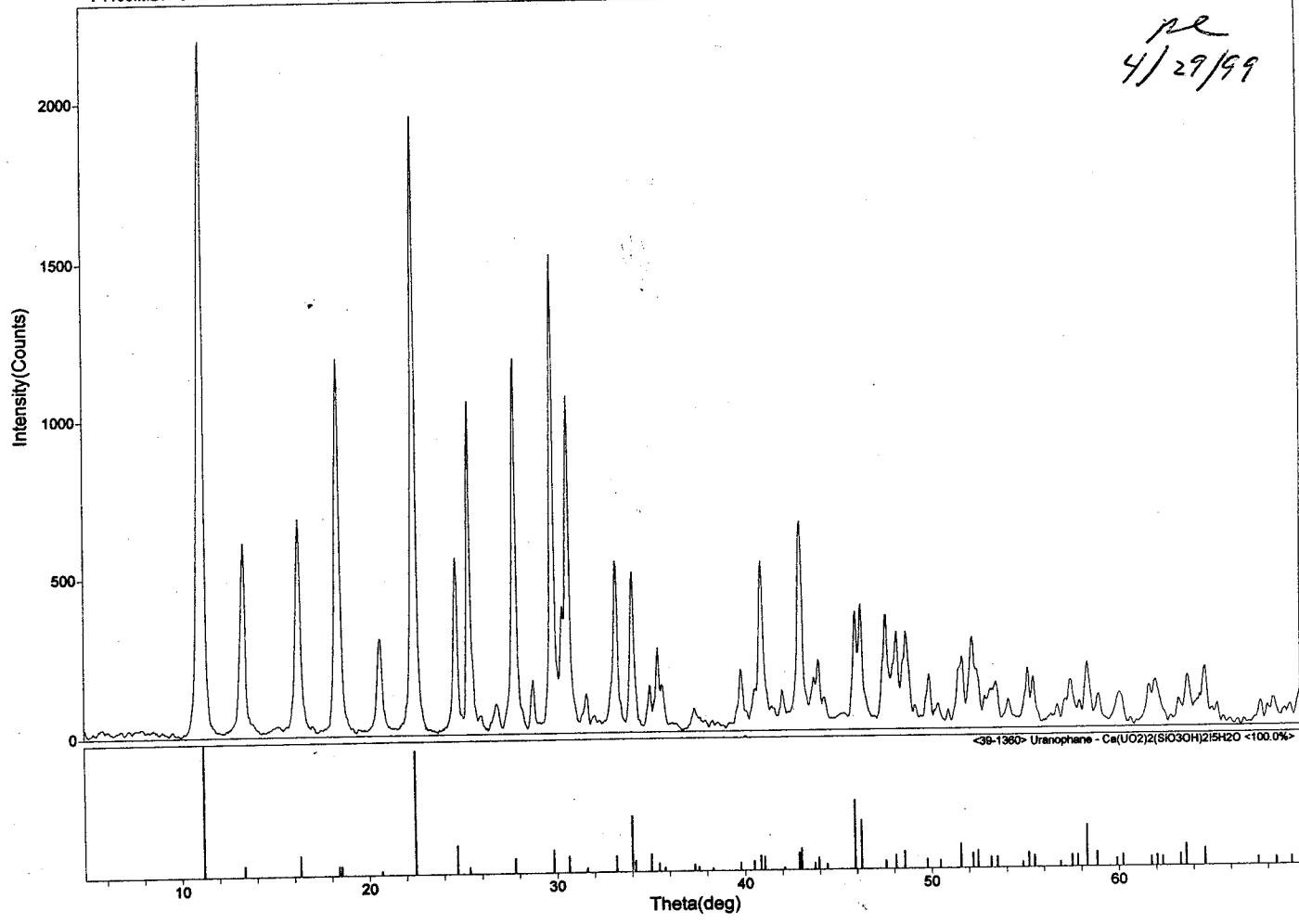
*Uranophane hydrate - Results cont'd*

*Uranophane hydrate - Results cont'd*

Spectrum: JFS1091      Range: 20 keV      Total Counts=1110386.    Linear    Auto-VS=15688



<F1100.MDI> UR2 - 20-1402-561 <2T(0)=-0.185>



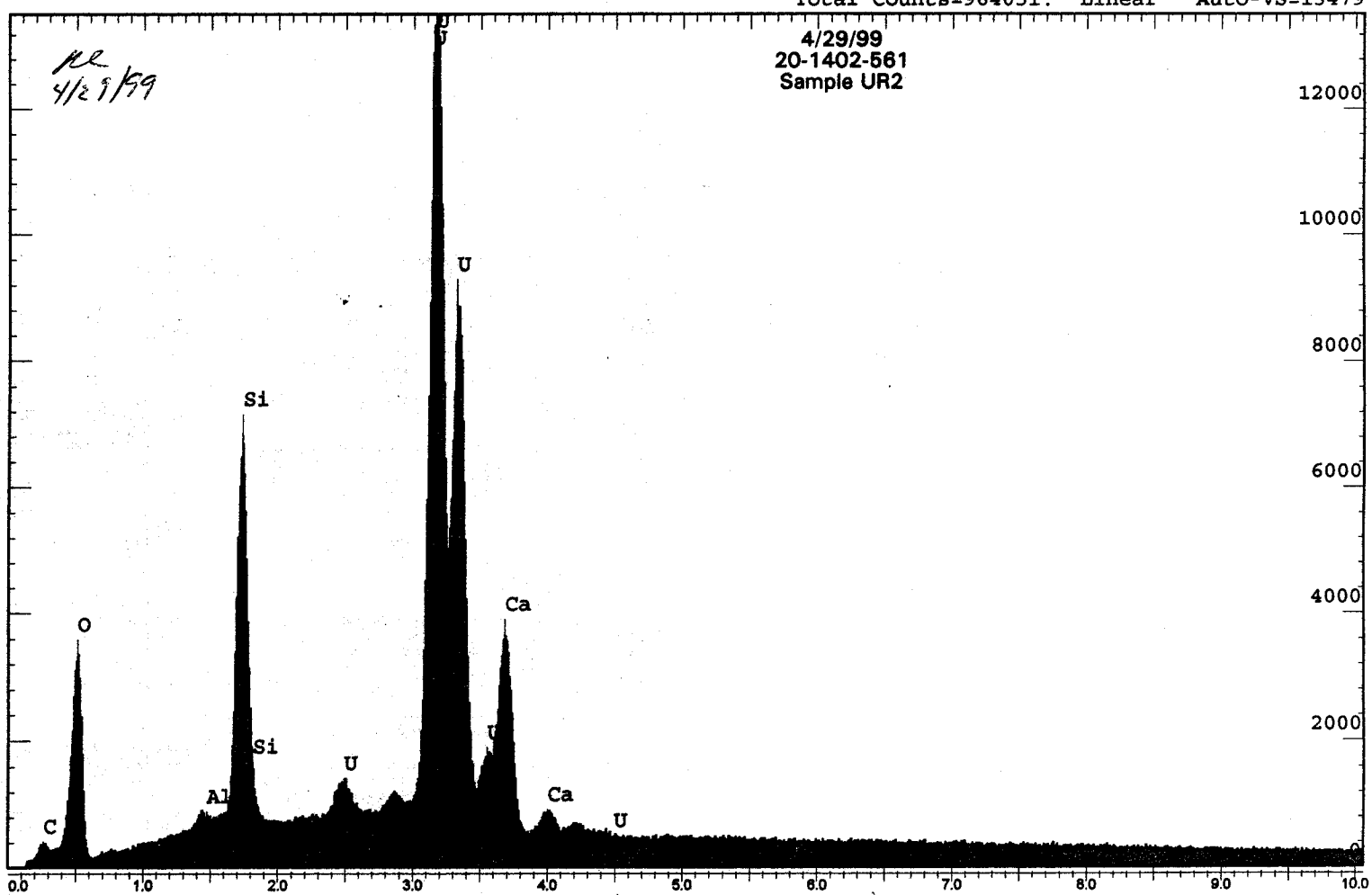
RC

Uranophane Synthesis - Results cont'd

Spectrum: JFS1092

Range: 20 keV

Total Counts=964051. Linear Auto-VS=13479



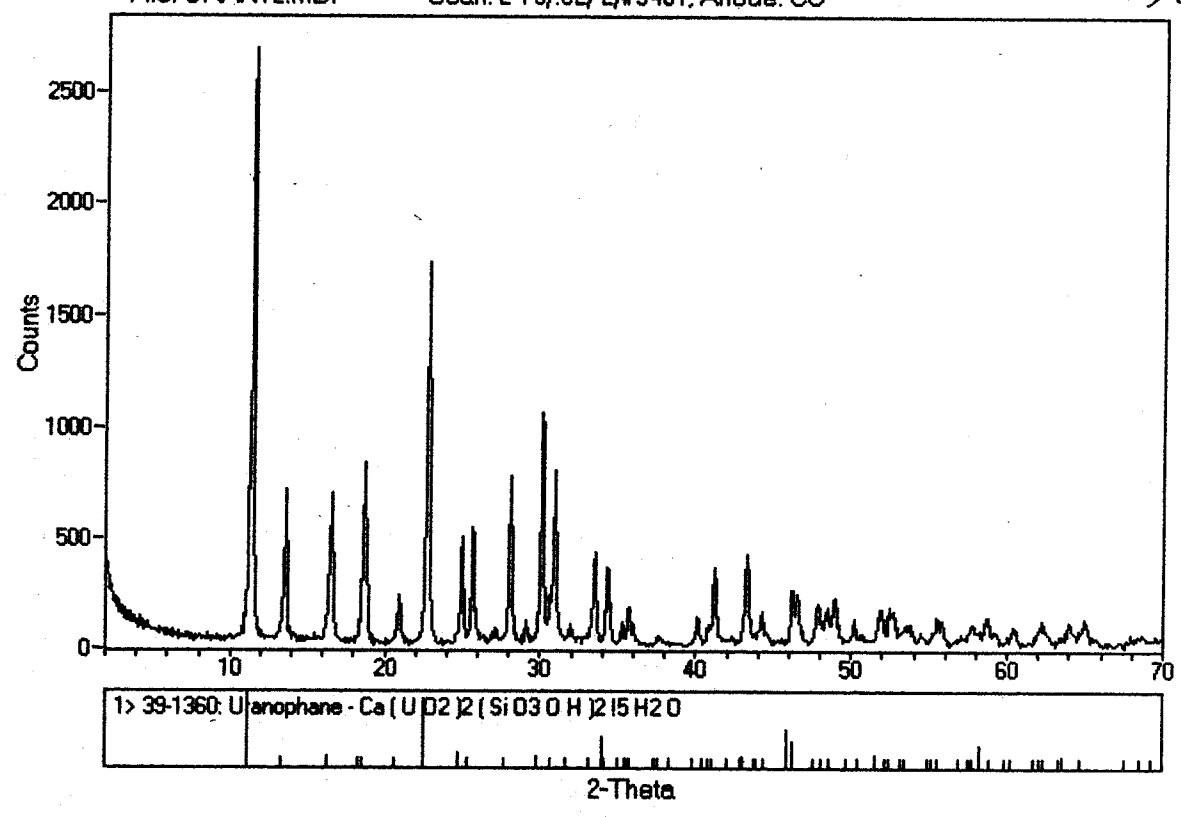
RC

Uranophane Synthesis - Results cont'd

ID: URAN\*SYN\*12  
File: URAN12.MDI

Scan: 2-70/02/ 2/#3401, Anode: CU

RC 4/29/99



Southwest Research Institute - Thu Apr 29 1999 82:40pm



4/30/99

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Synthesis of Uranophane  $[Ca(UO_2)_2(SiO_3OH)_2 \cdot 5H_2O]$   
Second Batch

Procedure: same as from page 2 of this laboratory notebook.

re Observations:

4/30/99

Equipment & Supplies: same as from page 2 with the following exceptions:

Uranyl acetate dihydrate  
Lot No. 386908/1 43398

Reagents: Sodium Metasilicate 28.65g  
Calcium acetate 35.22g  
9 M HCl 1300.00 mL  
Uranyl acetate dihydrate 42.41g

Observation: Began synthesis @ 1030. 4/30/99  
Water turned off @ 0825 5/10/99

5/10/99

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5/17/99

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Crystal material collected had similar characteristics as the first synthesized batch, however, small silver, grey specks were observed floating on surface of water in reaction vessel container. Appears to be from corrosion of metal parts, particularly the stirrer blades.

Skipped top surface of water using filter paper wrapped around a glass stirring rod. Most was removed.

Decanted about half the water through Whatman No. 1 filter in a glass funnel.

cont'd. page 14 re

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5/3/99

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Reagent preparation for silica analysis on the Milton Roy Spectronic 1201 spectrophotometer (UV, Vis)

Materials:

Analytical balance - Mettler AE 240  
100 mL volumetric flask  
Hot plate/stirrer  
Medicine dropper  
250 mL polyethylene bottles  
pH meter - Orion 920A / Orion 8103 combination probe  
D.I. water

Reagents:

Ammonium molybdate  
A.C.S. certified  
Fisher Scientific  
Lot No. 914771  
Oxalic acid

A.C.S. certified  
Fisher Scientific  
Lot No. 906135B

Sodium hydroxide - 6 M  
prepared 7/24/98 - A.V.

re 5/3/99

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Observations - cont'd from page 12

Discarded decanted water.  
Poured remaining contents of reaction vessel into a Buchner funnel apparatus with a Whatman No. 5 filter. Crystal contents had more specks of corrosion product intermixed within.

Product was allowed to air dry <sup>ne 5/17/99</sup> for about 1 1/2 hours.  
Crystals collected into a 250 ml beaker and placed in oven

5/20/99  
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Crystals observed under microscope contained numerous black specks of suspected corrosion materials.

5/21/99  
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Multiple additions of nanopure water to crystal batch in a 250 ml beaker and then sonicated caused dark particles to float on surface and then were decanted into hazardous waste containers.

No dark material is visible to the naked eye. Crystals were placed in drying oven over the weekend.

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Very few dark specks are observed under the microscope.

JP  
5/24/99 ~~Material was~~ <sup>ne 5/24/99</sup>

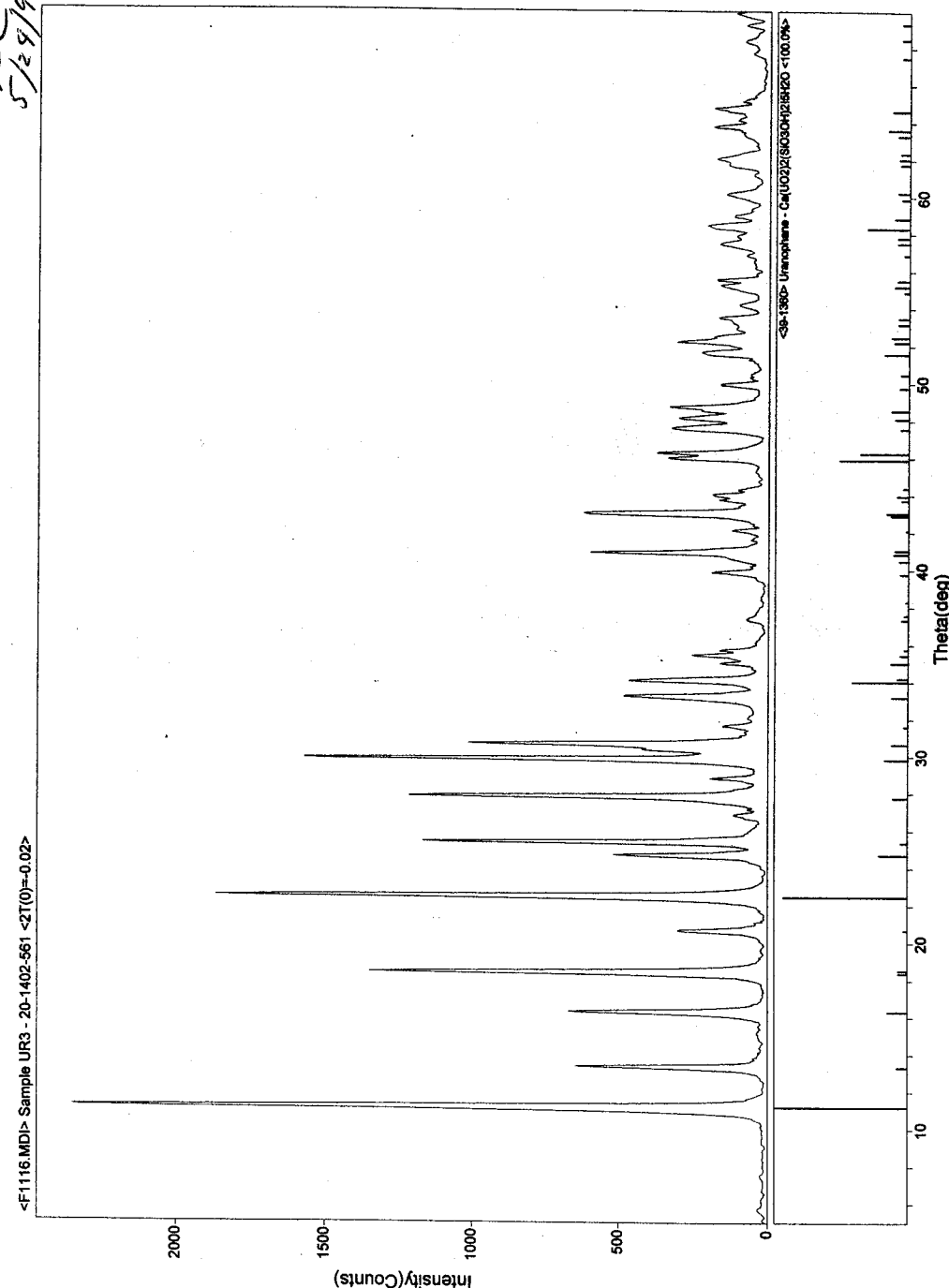
Batch is ~~combined~~ characterized using the XRD instrument. Results are as follows:

5/24/99 JP

Material was added to beaker labeled - URANOPHANE

5/24/99  
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Results:

NE  
5/24/99



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5/7/99  
nePreparation of silica standards for calibration of  
Milton Roy Spectronic 1201 spectrophotometer

Purpose: Prior to performing an analysis ne 5/7/99

Equipment & supplies: Milton Roy Spectronic 1201  
 1 cm cuvettes (Fisher Scientific)  
 necessary glassware & accessories as needed  
 - glassware treated with 1+1 HNO<sub>3</sub> &  
 rinsed with high purity H<sub>2</sub>O.  
 Analytical balance (Mettler PM4600)  
 Polyethylene bottles  
 Hot plate (Corning model PC220)  
 ne Corning  
 5/17/99 PH meter (Orion model 920A using  
 Orion 8103 combination pH  
 probe)  
 Nessler tubes matched, 50 ml, EXAX)

Reagents:  
 SiO<sub>2</sub> soln 10 ppm (Prepared 3/22/99 J.P.)  
 NaOH soln 6M (Prepared 7/24/98) (A.J.)  
 Ammonium Molybdate soln. (Prep 5/3/99)  
 Oxalic acid soln. (prepared 3/22/99 J.P.)  
 Reducing agent (prepared 5/4/99 A.J.)

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Procedure:

## 8.2 Heteropoly Blue Method

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In a low concentration modification the yellow molybdosilicic acid color is reduced by 1-amino-2-naphthol-4-sulfonic acid to a more intense heteropoly blue (815 nm). The minimum silica concentration detectable by this method is about 20 ug/L and the optimum concentration range lies between 50 ug/L and 2.5 mg/L.

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## 8.2.1 Preparation of calibration curve

- (1) Prepare a series of silica standards from 0 to 2.5 mg/L based on a 57 ml sample by accurately pipetting calculated volumes of standard silica solution (1 ml = 10 ug SiO<sub>2</sub>) into 50 ml Nessler tubes. If digestion (8.2.2) is used, add to each standard 200 mg NaHCO<sub>3</sub> and 2.4 ml 1N HSO<sub>4</sub>. Add water to a total volume of 50 ml.
- (2) Perform steps 8.1.1(2) thru 8.1.1(4).

10 ppm SiO<sub>2</sub> stock  
 .15 = 26.3  
 .25 ml = 43.86  
 .5 ml = 87.7  
 1 ml = 175.4  
 2 ml = 350.8  
 5 ml = 877.2  
 10 = 1754.4  
 14 = 2457.0  
 3 ml = 526.2  
 4 ml = 701.6  
 6 ml = 1052.4  
 7 ml = 1228.0  
 8 ml = 1403.2

## 8.1.1 Preparation of calibration curve

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- (1) Prepare a series of silica standards from 0 to 25 mg/L based on a 55 ml sample by accurately pipetting calculated volumes of standard silica solution (1 ml = 100 ug SiO<sub>2</sub>) into 50 ml Nessler tubes. If digestion (8.1.2) is used, add to each standard 200 mg NaHCO<sub>3</sub> and 2.4 ml 1N HSO<sub>4</sub>, to compensate for silica introduced by these reagents and for the effect of the salt on color intensity. Add water to a total volume of 50 ml.
- (2) Add rapidly to each standard 1 ml of 1+1 HCl and 2 ml ammonium molybdate reagent using volumetric pipettes. Mix by inverting at least 6 times.
- (3) Let stand for 5 to 10 minutes.
- (4) Add 2 ml oxalic acid solution to each standard using a volumetric pipette and mix thoroughly.
- (5) After 2 minutes but before 15 minutes from the addition of the oxalic acid solution, transfer 2 to 3 ml of each standard to a spectrophotometer cell.



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Procedure: cont'd.

- (6) Set spectrophotometer to a wavelength of 410 nm and adjust spectrophotometer to zero absorbance with water.
- (7) Construct a standard curve by either,
  - (a) Reading the absorbance of each standard on the spectrophotometer and plotting absorbance values versus the concentration of each standard or,
  - (b) Performing a linear curve fit test as described in Section 4.6.2 of the Operator's Manual for the Milton Roy Spectronic 1201 Spectrophotometer. If this technique is used the spectrophotometer measures the absorbance and prompts the operator for the concentration of each standard. When the spectrophotometer has the data for all standards, it mathematically constructs a standard curve using the absorbance and concentration of each standard. It then calculates slope and intercept values for the standard curve.

#### 8.1.2 Digestion

If molybdate unreactive silica is present and its inclusion in the analysis is desired, include this step, otherwise proceed to 8.1.3.

- (1) Place 50 ml sample, or a smaller sample portion diluted to 50 ml, in a 100 ml platinum dish using a volumetric pipette.
- (2) Add 200 mg  $\text{NaHCO}_3$  and digest on a steam bath for 1 hour. Cool.
- (3) Add slowly and with stirring 2.4 ml 1N  $\text{H}_2\text{SO}_4$  using volumetric pipettes.
- (4) Immediately transfer to a 50 ml Nessler tube, dilute to the mark with water and proceed to 8.1.3 without delay.

#### 8.1.3 Color development and measurement

- (1) Place 50 ml sample, or a smaller sample portion diluted to 50 ml, in a Nessler tube using a volumetric pipette.
- (2) Add rapidly 1 ml of 1+1 HCl and 2 ml ammonium molybdate reagent using volumetric pipettes.

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Procedure: cont'd.

- Mix by inverting at least 6 times.
- (3) Let stand for 5 to 10 minutes.
  - (4) Add 2 ml oxalic acid solution using a volumetric pipette and mix thoroughly.
  - (5) After 2 minutes but before 15 minutes from the addition of the oxalic acid solution, transfer 2 to 3 ml of sample to a spectrophotometer cell.
  - (6) Determine the concentration of the sample by either,
    - (a) Reading the absorbance of the sample and determining silica concentration from the standard curve prepared in 8.1.1(7a) or,
    - (b) Measuring the absorbance of the sample using the spectrophotometer's linear curve fit test as described in Section 4.6.2 of the Operator's Manual for the Milton Roy Spectronic 1201 Spectrophotometer. After measuring the absorbance of the sample the spectrophotometer will automatically determine the concentration of the sample using the slope and intercept values calculated in 8.1.1(7b).

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Results:

Standard No. 1 prepared by addition of  
0.25 ml of 10 ppm  $\text{SiO}_2$  soln. per 57 ml sample.  
(43.86 ppb  $\text{SiO}_2$ )

Standard No. 2 prepared by addition of  
0.50 ml of 10 ppm  $\text{SiO}_2$  soln. per 57 ml sample.  
(87.7 ppb  $\text{SiO}_2$ )

Standard No. 3 prepared by addition of 1.0 ml  
of 10 ppm  $\text{SiO}_2$  soln. per 57 ml sample.  
(175.4 ppb  $\text{SiO}_2$ )

Standard No. 4 prepared by addition of  
2.0 ml of 10 ppm  $\text{SiO}_2$  soln. per 57 ml sample.  
(350.9 ppb  $\text{SiO}_2$ )

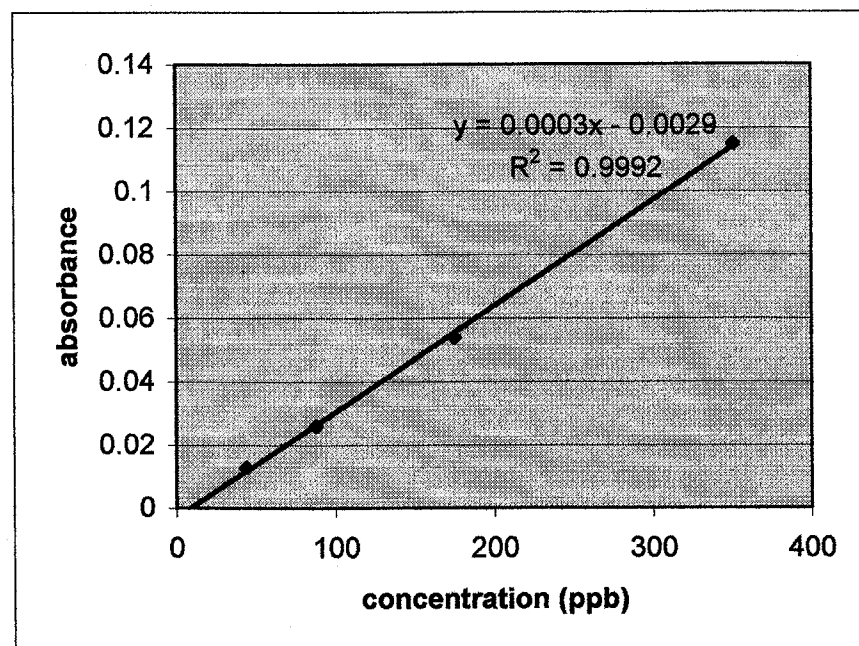
5/7/99  
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Results: Absorbance and Concentration Measurements  
of Standard Solutions analyzed using the  
Milton Roy Spectronic 1201 Spectrophotometer

Standard	Absorbance	Concentration (ppb)
No. 1	0.013	43.86
No. 2	0.026	87.7
No. 3	0.054	175.4
No. 4	0.115	350.7

absorbance concentration

0.013	43.86
0.026	87.7
0.054	175.4
0.115	350.7

5/16/99  
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Preparation of  $\text{CaCO}_3$  soln. to be used in  
actinide coprecipitation experiments.

Materials: 16 polycarbonate bottles  
3 2L Nalgene storage bottles  
1 L Volumetric flask w/stopper  
glass funnel  
D.I. Nanopure  $\text{H}_2\text{O}$   
Analytical balance (Mettler PM 4600)

Reagents:

Calcium carbonate - A.C.S. certified  
Mfg. Fisher Scientific  
Lot No. 986396  
Lot No. 986396

Procedure: Weighed 50 g  $\text{CaCO}_3$  into tared  
1 L volumetric flask w/ funnel.

Added D.I.  $\text{H}_2\text{O}$  to mark.

Poured contents of flask to 1 L PC  
storage bottles (6) and a

Weighed 100 g  $\text{CaCO}_3$  into tared  
2 L Vol. flask w/ funnel.  
5/16/99

Added D.I.  $\text{H}_2\text{O}$  to mark.

Poured contents into 2 L Nalgene  
storage bottles (2).  
5/16/99

Labeled the 1 liter sample bottles as  $\text{CaCO}_3$  A,  
B, C, D, E, F + the 2 liter sample bottles as  
 $\text{CaCO}_3$  1 + 2.

5/11/99  
ML

Procedure - cont'd.

	$\text{CaCO}_3$ g / L H <sub>2</sub> O
Sample No. 1	50.00
No. 2	50.00
No. 3	50.00
No. 4	50.00
No. 5	50.00
No. 6	50.00
No. 7	100.03
No. 8	100.04

ML

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5/11/99  
MLPreparation of 0.02 M  $\text{NaHCO}_3$  soln. to be used for equilibrium studies of calcite.

Materials:

Analytical balance - Mettler AE 240  
 250 mL volumetric flask  
 100 mL graduated cylinder  
 8 L plastic storage bottle  
 glass funnel  
 D.I. water  
 Hot plate

Reagents:

Sodium bicarbonate F.W. = 84.01  
 A.C.S. certified  
 Fisher Scientific  
 Lot No. 936883

Procedure:

Weighted 21.00 g  $\text{NaHCO}_3$  into a tared 250 mL volumetric flask, then filled to mark with D.I. water, mixed & heated slightly to dissolve all visible crystals.

Cooled & then added D.I. water to mark. This produced a 1 M soln.

Measured 160 mL 1 M  $\text{NaHCO}_3$  soln. in a 100 mL graduated cylinder and transferred into an 8 L plastic storage bottle.

Added 7.840 L of D.I. water measured with a 1000 mL flask into the 8 L bottle to produce a 0.02 M  $\text{NaHCO}_3$  soln. Labeled.

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Purpose: Prepare U, Si, Ca standards for chemical analysis. Standards will be used in the characterization of lab synthesized uranophane.

(525/2) re

(525/12) re

re 5/24/99

Object

Reagents: Uranyl acetate lot No. 386908/1 43398  
Sodium silicate lot No. 942853A  
Calcium acetate lot No. 944221

## Supplies &amp; Materials:

100 mL beakers

Ultrapure water

Analytical balance - Mettler ~~AA4600~~ AE240

re weighing paper  
5/24/99 Drying oven  
Stir bars

## Procedure: Prepare 3 standards in the following

Ca: U: Si molar ratios - 2:1:1, 2:1:2,  
2:2:1

② To 3 beakers add following reagents in amounts specified:

Std. 1 (2:1:1) - 1.7619 g Ca acetate  
2.1207 g U acetate  
1.421 g Na silicate

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## Procedure - cont'd.

Std. 2 (2:1:2) - 1.7619 g Ca acetate  
2.1207 g U acetate  
2.842 g Na silicate

Std. 3 (2:2:1) - 1.7619 g Ca acetate  
4.2415 g U acetate  
1.421 g Na silicate

③ Add about 100 mL H<sub>2</sub>O to each beaker  
& mix to achieve homogeneity

④ Place beaker in drying oven at ~60°C  
& allow solution to evaporate.

⑤ Remove solid & place in plastic vials  
labeled as follows:

U-Ca-Si Std. 1

Al-Ca-Si Std. 2

U-Ca-Si Std. 3

Observations: Uranyl acetate dihydrate  
Mfg. Fluka  
lot No. 386908/1 43398  
Assay >99% (T)

Sodium silicate (meta)  
Fisher Chemical  
lot No. 942853A  
Fisher certified

5/24/99  
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observations - cont'd.

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Calcium acetate  
Fisher Chemical  
Lot. No. 944231  
Fisher certified

(Std. 1) Tared beaker + weighed Ca acetate 1.76185 g  
U acetate 2.1213 g  
Na silicate 1.4286 g

(Std. 2) Tared beaker + weighed Ca acetate 1.7624 g  
U acetate 2.1210 g  
Na silicate 2.8436 g

(Std. 3) Tared beaker + weighed Ca Acetate 1.7624 g  
U acetate 4.2424 g  
Na silicate 1.4227 g

Water added, stirred reagents and placed  
beakers in drying oven.

a separation of white and yellow crystals was  
observed on the sides of the beaker. It was  
determined that a uniform, homogeneous  
mixture of the reagents was going to be  
difficult to obtain.

The reagents will be weighed as before, but  
placed in a pulverizing mill, with no water  
or solvents to be added.

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The preparation of U, Si, + Ca standards modified  
from the ~~prev~~ procedure on page 26 of this notebook.  
5/25/99

The reagents for Std. No. 1 were weighed and placed  
in the mill container, processed for 7 min. and then  
collected into a plastic sample container.

The reagents were packed solid, probably due to  
adsorbed moisture from the air and from structural  
H<sub>2</sub>O in the hydrates.

Std. No. 2 reagents were weighed and placed in  
a 50 ml beaker and placed in the drying oven overnight.  
Std. No. 3 likewise was weighed & placed in the drying  
oven.

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Dried reagents were placed in mill container for  
~ 7 min. Powder was placed in <sup>glass</sup> plastic containers.  
5/26/99

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5/26/99



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Preparation of uranophane and standards  
for chemical analysis in Div 01.

~0.3g  
Aliquots of uranophane samples and  
standards were placed in glass vials  
and delivered to Div 01 for chemical  
analysis of major species. The glass  
vials were labeled as follows:

Label	Sample
UR1	URANOPHANE
UR2	URAN * SYN
UR3	STD 1
UR4	URANOPHANE
UR5	STD 2
UR6	URAN * SYN * 3
UR7	STD 1
UR8	URANOPHANE
UR9	STD 2
UR10	URANOPHANE
UR11	STD 3
UR12	STD 3

The sample will undergo whole rock  
dissolution followed by ICP analysis

5/26/99 JP

Below are copies of the DIV 01 SAMPLE LIST/  
CHAIN OF CUSTODY forms filled out for  
the whole rock analysis of uranophane.

Client Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:			
James D. Pinkert CNWRA / Div 20 Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78246-6105		<input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> Other:			
Client Purchase Order/Other ID		Site/Zone ID		Analysis Requested			
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time (mm/dd/yyyy)	Matrix Type	Sample Type	# of Containers	Whole Rock Analysis by ICP and major species	REMARKS
UR1	5/26/99		Mineral		1	X	Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Other (Specify)
UR2					1	X	
UR3					1	X	
UR4					1	X	
UR5					1	X	
UR6					1	X	
UR7					1	X	
UR8					1	X	
UR9					1	X	
UR10					1	X	
Matrix Types: A - Air, P - Product, S - Soil, T - Tissue, W - Water Sample Types: DM - Dissolved Material, EP - Enrichment Process FB - Field Blank, MSD - Matrix Spike Duplicate, MB - Matrix Spike TB - Trip Blank, TM - Total Material, ES - Environmental Samples FD - Field Duplicate							Relinquished by (Signature) Received by (Signature) Relinquished by (Signature) Received by (Signature)
Relinquished by (Signature) Received by (Signature)							
Relinquished by (Signature) Received by (Signature)							
Relinquished by (Signature) Received by (Signature)							
Div 01 COC Form 01-01-001, Rev 1/97							Page 1 of 2

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7/26/99  
pc

RC  
7/26/9

Sample ID
NBS688

Work Order: 15783

Analysis	Concentration Oxides	True Value	Recovery
Al <sub>2</sub> O <sub>3</sub>	17.5%	17.36%	100.8%
BaO	203	223	91.0%
CaO	12.3%	12.17%	101.1%
CoO	73	62	117.2%
Cr <sub>2</sub> O <sub>3</sub>	490	485	101.1%
CuO	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	10.4%	10.35%	100.5%
K <sub>2</sub> O	1930	1870	103.2%
MgO	8.48%	8.40%	101.0%
MnO	1720	1670	103.0%
Na <sub>2</sub> O	2.16%	2.15%	100.5%
NiO	205	201	102.1%
P <sub>2</sub> O <sub>5</sub>	1500	1340	111.9%
SiO <sub>2</sub>	49.9%	48.4%	103.1%
SrO	214	200	107.2%
TiO <sub>2</sub>	1.16%	1.17%	99.1%
V <sub>2</sub> O <sub>5</sub>	462	430	107.4%
ZnO	99	105	94.7%

NA- Not Applicable.

7/26/99  
RL

SOUTHWEST RESEARCH INSTITUTE  
LABORATORY CONTROL SAMPLE

RL  
7/26/99

Sample ID  
NBS278

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: NA

Client: Division 20  
Date Received: NA  
Project No.: 20-1402-561  
Work Order: 15783

Analysis	Concentration Oxides	True Value	Recovery
Al <sub>2</sub> O <sub>3</sub>	13.6%	14.15%	96.1%
BaO	1040	1230	84.6%
CaO	9960	9830	101.3%
CoO	NA	NA	NA
Cr <sub>2</sub> O <sub>3</sub>	NA	NA	NA
CuO	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	2.03%	2.04%	99.5%
K <sub>2</sub> O	4.11%	4.16%	98.8%
MgO	2350	2300	102.2%
MnO	518	520	99.7%
Na <sub>2</sub> O	4.81%	4.84%	99.4%
NiO	NA	NA	NA
P <sub>2</sub> O <sub>5</sub>	NA	NA	NA
SiO <sub>2</sub>	72.7%	73.1%	99.5%
SrO	79	75	105.5%
TiO <sub>2</sub>	2300	2450	93.9%
V <sub>2</sub> O <sub>5</sub>	NA	NA	NA
ZnO	NA	NA	NA

NA- Not Applicable.

RL  
7/26/99

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR1

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123619

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.45%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	278	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	537	----
NiO	66	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	15.8%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	53.9%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

RL  
7/26/99

Sample ID  
UR2

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123620

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.42%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	133	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	<150	----
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	15.5%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	54.7%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

7/26/99  
re

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

re  
7/26/99

Sample ID  
UR3

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123621

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	13.1%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	6.82%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	7.00%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	26.3%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123622

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Sample ID  
UR4

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.81%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	309	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	614	----
NiO	84	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	17.0%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	57.7%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

re  
7/26/99

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR5

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123623

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	10.9%
CoO	85	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	11.5%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	11.4%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	21.3%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Solid  
Lab System ID: 123624

Client: Division 20  
Date Received: 05/26/99  
Project No.: 20-1402-561  
Work Order: 15783

Sample ID  
UR6

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.42%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	101	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	<150	----
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	14.9%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	54.8%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----



**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Solid

Lab System ID: 123625

Client: Division 20

Date Received: 05/26/99

Project No.: 20-1402-561

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	13.1%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	6.78%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	6.96%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	26.2%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Solid

Lab System ID: 123626

Client: Division 20

Date Received: 05/26/99

Project No.: 20-1402-561

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.52%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	273	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	591	----
NiO	107	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	16.2%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	54.5%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR9

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Solid

Lab System ID: 123627

Client: Division 20

Date Received: 05/26/99

Project No.: 20-1402-561

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	11.3%
CoO	91	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	11.9%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	11.8%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	21.6%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	525	----

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Solid

Lab System ID: 123628

Client: Division 20

Date Received: 05/26/99

Project No.: 20-1402-561

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	6.43%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	376	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	436	----
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	16.1%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	54.2%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----



**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

MC  
7/26/99

Sample ID  
UR11

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 05/26/99

Matrix: Solid

Project No.: 20-1402-561

Lab System ID: 123629

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	9.28%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	4.67%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	4.90%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	37.6%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 05/26/99

Matrix: Solid

Project No.: 20-1402-561

Lab System ID: 123630

Work Order: 15783

Analysis	Concentration Oxides (mg/Kg)	% Concentration Oxides
Al <sub>2</sub> O <sub>3</sub>	<1000	----
BaO	<60	----
CaO	----	8.93%
CoO	<65	----
Cr <sub>2</sub> O <sub>3</sub>	<75	----
CuO	<250	----
Fe <sub>2</sub> O <sub>3</sub>	<2000	----
K <sub>2</sub> O	<300	----
MgO	<400	----
MnO	<65	----
Na <sub>2</sub> O	----	4.55%
NiO	<65	----
P <sub>2</sub> O <sub>5</sub>	<600	----
SiO <sub>2</sub>	----	4.70%
SrO	<60	----
TiO <sub>2</sub>	<85	----
U <sub>3</sub> O <sub>8</sub>	----	36.3%
V <sub>2</sub> O <sub>5</sub>	<90	----
ZnO	<65	----

8/12/99  
AC

Gravimetric analysis of synthesized uranophane  
for determination of H<sub>2</sub>O content.

Objective: To determine water content of synthesized  
uranophane.

Equipment + supplies:

muffle furnace - Fisher Scientific Isotemp 184-A

Quartz crucibles (10mL cap.) Fisher

Analytical balance - Mettler

Dissicator -

Uranophane - synthesized per 325/12 lab-notebook.

Procedure:

① Placed 0.25g of Uranophane into weighed quartz  
crucibles. Recorded weights as follows:

	Wt. of Crucible	Wt. of Uranophane	Wt. of sample
sample 1	12.6464 g	12.8932 g	0.2468 g
sample 2	12.2494 g 12.8932 g 8/12/99	12.5063 g	0.2569 g
sample 3	11.5348 g	11.7809 g	0.2461 g

② Placed crucibles w/ samples in muffle furnace  
for 2 hrs @ 900°C.

③ Samples were cooled in dissicator and then  
weighed. Weights as follows:

8/12/99  
RC

Procedure - contd.

## ③ Weight of Samples after Heating:

sample 1 12.8632 g  
sample 2 12.4756 g  
sample 3 11.7512 g

12/12/99  
RC

Calculations:

wt. of Crucible before heat - wt. of Crucible after heat = wt. of sample  
sample 1 12.6464 g - 12.8932 g = 0.2468 g  
sample 2 12.2494 g - 12.5063 g = 0.2569 g  
sample 3 11.5248 g - 11.7809 g = 0.2461 g

wt. of Crucible before heat - wt. of Crucible after heat = Wt. of H<sub>2</sub>O evaporated  
sample 1 12.8932 g - 12.8632 g = 0.0300 g  
sample 2 12.5063 g - 12.4756 g = 0.0307 g  
sample 3 11.7809 g - 11.7512 g = 0.0297 g

$$\frac{\text{Wt. of H}_2\text{O}}{\text{Wt. of Uranophane}} \times 100 = \% \text{ H}_2\text{O in sample}$$
  
sample 1  $\frac{0.0300}{0.2468} \times 100 = 12.16\%$   
sample 2  $\frac{0.0307}{0.2569} \times 100 = 11.95\%$   
sample 3  $\frac{0.0297}{0.2461} \times 100 = 12.07\%$

Average % H<sub>2</sub>O = 12.06%8/12/99  
RC

Dissolution of synthesized uranophane

Objective: To dissolve synthesized uranophane (6C325/12) in dilute HCl for analysis of major elements by Div. 01.

Reagents &amp; Supplies:

- 0.1 M HCl from Lot No. 478110 Conc. Fisher Chemical
- Volumetric flasks (100 mL + 16)
- PP sample bottles (125 mL)
- Analytical balance - Mettler AE 240
- Hot plate/stirrer - Corning Corning 8/12/99

Procedure:

① Prepared ~0.1 M HCl by adding 2.5 mL 9 M HCl to 250 mL flask volumetric flask and adding to mark w/ D.I. water.

② Added 0.5 g Uranophane to fared 100 mL vol. flask. Weights of uranophane samples are as follows:

sample 1 0.4984 g  
sample 2 RC 8/12/99  
sample 3 RC 8/12/99

Determined that 0.5 g of sample was too large for needed analysis. Discarded ~~sample~~ prepared sample in Radioactive waste container ~~kept~~ kept under the hood. Proceeded to prepare smaller quantity samples.

8/12/99  
RC

## Procedure - contd.

- ③ Added 0.1g uranophane to tared 100ml volumetric flask. Weighed 0.0984g. Labeled sample 1.

Added 0.1g uranophane to tared 100ml vol. flask. Weighed 0.1018g. Labeled sample 2.

Added 0.2g uranophane to tared 100ml vol. flask. Weighed 0.2067g. Labeled sample 3.

- ④ Washed inside wall of flask with 0.1M HCl. added ~60ml to each flask. Heated on hot plate and agitated soln. by hand.

8/16/99  
RC

Observation: After 2 days of stirring, uranophane did not dissolve completely, or a precipitate was formed; small white, threadlike substance settled to bottom of the flasks. A trial 0.1g uranophane sample was combined with a 0.2M  $\text{HNO}_3$  in a 100ml vol. flask and stirred. Some white substance remained but much less than with the HCl soln.

Prepped flasks of uranophane + HCl held in the drop in case further analysis is requested to determine the character of the residue.

3 new samples of uranophane +  $\text{HNO}_3$  to be prepared as follows:

8/16/99  
RC

## Procedure - contd.

- ① 0.1g uranophane added to clean 100ml volumetric flask. Weighed 0.1092g. Filled to mark w/D.I. water. Added magnetic stir bar in flask and placed on stir plate. Labeled sample 1. Added 1.5ml conc.  $\text{HNO}_3$  (0.2M).

- ② 0.1g uranophane added to 100ml vol. flask. Weighed 0.1033g. Filled to mark w/D.I. water. Added magnetic stir bar and placed on stir plate. Labeled sample 2. Added 1.5ml conc.  $\text{HNO}_3$  (0.2M).

- ③ 0.2g uranophane added to 100ml flask. Weighed 0.2039g. Filled to mark w/D.I. water. Placed on stir plate. Labeled sample 3. Added 1.5ml conc.  $\text{HNO}_3$  (0.2M).

8/17/99  
RC

Preparation of aqueous samples of dissolved synthesized uranophane (525/12) for analysis by ICP for major and minor cation species by Division 01.

## Procedure:

- ① Prepared U standards <sup>concentrations 8/11/00</sup> using previously prepared  $\text{UO}_2(\text{NO}_3)_2$  soln. prepared 1/11/96 (GC-11-220) of  $5 \times 10^{-3} \text{M}$ . (1,190ppm U)

a) 178.5ppm U — 15ml rep. std. in 100ml vol. 8/17/00

Std. check for Ca, K, Mg, Na, Al, Ba, Fe, Sb, Co, V, and Ni prepared from Rep. standard Certiprep, lot no. 15-154 AS, exp. date 7/00.

8/17/99  
RL Procedure - contd.

Sld. check for Si prepared from AA ref. std.  
Fisher Scientific, lot no. 994097-24, exp.  
date 3/01.

③ Prepared secondary Ca std. soln. by adding  
10 mL Ref. Std. lot no. 15-154AS to 100 mL  
volumetric flask, filling to mark w/ 0.2 M HNO<sub>3</sub>  
soln. PPM = 50.

⑤ Prepared secondary Si std. soln. by adding 10 mL  
of ref. std. lot no. 994097-24 to 100 mL vol.  
flask, filling to mark w/ 0.2 M HNO<sub>3</sub> soln.

⑥ Prepared Standard Check No. 1 by adding 15 mL  
U ref. std. to 100 mL vol. flask,  
5 mL Ca (50 ppm) secondary std.  
5 mL Si (100 ppm), filled to mark w/ 0.2 M HNO<sub>3</sub>.

⑦ Prepared Standard Check No. 2 by adding:  
10 mL U (1,190 ppm),  
10 mL Ca (50 ppm),  
10 mL Si (100 ppm), to 100 mL vol. flask &  
filling to mark w/ 0.2 M HNO<sub>3</sub>.

⑧ Prepared Std. Check No. 3 by adding:  
5 mL U (1,190 ppm),  
20 mL Ca (50 ppm),  
15 mL Si (100 ppm) to 100 mL vol. flask &  
adding to mark w/ 0.2 M HNO<sub>3</sub>.

⑨ Prepared Std. Check No. 4 by adding:  
2 mL U (1,190 ppm),  
25 mL Ca (50 ppm),  
20 mL Si (100 ppm) to 100 mL vol. flask &

8/17/99  
RL Procedure - contd.

adding to mark w/ 0.2 M HNO<sub>3</sub>.

8/18/99  
RL

Prepared standard checks were labeled as follows  
in 30 mL plastic bottles (20 mL soln, ea.):

Labeled	Species Conc.
20-1C	U = <sup>178.5</sup> <del>60.7</del> ppm; Ca = 2.5 ppm; Si = 5 ppm; Na = <sup>12.5</sup> <del>3.1</del> ppm

20-2C	U = 119 ppm; Ca = 5.0 ppm; Si = 10 ppm; Na = 5.0 ppm
-------	---

20-3C	U = 59.5 ppm; Ca = 10 ppm; Si = 15.0 ppm; Na = 10.0 ppm
-------	--

20-4C	U = 23.8 ppm; Ca = <sup>12.5</sup> <del>5.0</del> ppm; Si = 20 ppm; Na = 15.0 ppm <sup>5/18/99</sup>
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Procedure for preparing dissolved ceramphone samples  
for analysis by Dir. A:

① Samples 1, 2, and 3 were filtered through  
No. 1 Whatman filter paper. Approx. 20 mL  
were filtered.

② Each sample was diluted by a factor of 10 by  
pipetting 10 mL of filtered sample using a  
class A vol. pipette into a 100 mL vol.  
flask and adding to mark w/ a 0.2 M HNO<sub>3</sub> soln.

③ Two 20 mL samples were taken from <sup>8/18/99</sup>the each  
diluted samples per step 2 and placed in  
30 mL plastic containers. A 20 mL sample of

8/18/99  
PC

8/18/99

Each of the 4 Check Standards were placed  
in 30 mL plastic bottles and labeled:Std 1 - GC-20-1C  
Std 2 GC-20-2C  
Std 3 GC-20-3C  
Std 4 GC-20-4C9/21/99  
PCResults: Div. 01 ICP Analysis for cations - Uranghane + Standard  
Uranghane unknown sampleSOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEETPC  
9/21/99Sample ID  
20-1A

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 08/18/99

Matrix: Liquid

Project No.: 20-1402-561

Lab System ID: 128595

Work Order: 16271

① Samples delivered to Div. 01 for analysis.

Client Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:				
BOB CHERRINGTON CNVRA/OIV. 20 BLOC. 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		<input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> Other:				
Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact:				
Sample ID		Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP Analysis	REMARKS
20-1A	8/18/99			DM	DM	1	X	Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Other (Specify)  Project is per from sample analysis 100% RSP Cat 41 Uranghane B  Question RSC Join Bridge at Cherrington X5501
20-1B	"			"	"	1	X	
20-1C	"			"	"	1	X	
20-2A	"			"	"	1	X	
20-2B	"			"	"	1	X	
20-2C	"			"	"	1	X	
20-3A	"			"	"	1	X	
20-3B	"			"	"	1	X	
20-3C	"			"	"	1	X	
20-4C	"			"	"	1	X	
Matrix Types: A - Air, P - Product, S - Soil, T - Tissue, W - Water		Relinquished by (Signature):		Blank Project No:				
Sample Types: DM - Dissolved Metals; ER - Equipment Rinse; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; EB - Environmental Samples; FD - Field Duplicate		Received by (Signature):		Received by SWRI Lab (Signature):				
Relinquished by Sample (Signature):		Relinquished by (Signature):		Samples Disposed by:				
Received by (Signature):		Comments:		Date/Time: 8/18/99 - 15:35				

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

Page 1 of 1

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.5	0.5
Antimony	<0.05	0.05
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.05	0.05
Boron	<0.25	0.25
Cadmium	<0.025	0.025
Calcium	6.01	0.5
Chromium	<0.05	0.05
Cobalt	<0.025	0.025
Copper	<0.025	0.025
Iron	<0.5	0.5
Lanthanum	<0.025	0.025
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<0.5	0.5
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.025	0.025
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<0.5	0.5
Selenium	<0.05	0.05
Silicon	7.35	0.5
Silver	<0.05	0.05
Sodium	0.679	0.25
Strontium	<0.025	0.025
Sulfur	0.106	0.1
Thallium	<0.25	0.25
Thorium	<0.25	0.25
Tin	<0.05	0.05
Titanium	<0.25	0.25
Tungsten	<0.1	0.1
Uranium	59.6	0.5
Vanadium	<0.025	0.025
Yttrium	<0.025	0.025
Zinc	0.036	0.025
Zirconium	<0.25	0.25

NOTE: This sample was initially analyzed in a non-compliant run. There was not enough "original" sample remaining to re-analyze it undiluted. Therefore, it was diluted by a factor of five and the reporting limits are adjusted due to this dilution.



9/21/99  
pc

Results: Uranophane unknown sample

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

pc  
9/21/99

Sample ID  
20-1B

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128596

Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	<0.01	0.01
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	5.46	0.1
Chromium	<0.01	0.01
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.1	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	<0.1	0.1
Manganese	<0.005	0.005
Molybdenum	0.018	0.005
Nickel	<0.005	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.01	0.01
Silicon	7.21	0.1
Silver	<0.01	0.01
Sodium	0.621	0.05
Strontium	<0.005	0.005
Sulfur	0.126	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	58.5	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.018	0.005
Zirconium	<0.05	0.05

9/21/99  
pc

Results: Uranophane unknown sample

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

pc  
9/21/99

Sample ID  
20-2A

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128598

Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	<0.01	0.01
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	5.38	0.1
Chromium	<0.01	0.01
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	0.102	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	<0.1	0.1
Manganese	<0.005	0.005
Molybdenum	0.027	0.005
Nickel	<0.005	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.01	0.01
Silicon	6.99	0.1
Silver	<0.01	0.01
Sodium	0.586	0.05
Strontium	<0.005	0.005
Sulfur	0.122	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	56.8	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.039	0.005
Zirconium	<0.05	0.05

9/21/99  
PC

Results: Uranophane unknown sample

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

PC  
9/21/99

Sample ID  
20-2B

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 08/18/99

Matrix: Liquid

Project No.: 20-1402-561

Lab System ID: Y28599

Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	<0.01	0.01
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	5.33	0.1
Chromium	<0.01	0.01
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.1	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	<0.1	0.1
Manganese	<0.005	0.005
Molybdenum	0.027	0.005
Nickel	<0.005	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.01	0.01
Silicon	6.99	0.1
Silver	<0.01	0.01
Sodium	0.563	0.05
Strontium	<0.005	0.005
Sulfur	0.120	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	56.8	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.033	0.005
Zirconium	<0.05	0.05

9/21/99  
PC

Results: Uranophane unknown sample

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

PC  
9/21/99

Sample ID  
20-3A

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 08/18/99

Matrix: Liquid

Project No.: 20-1402-561

Lab System ID: 128601

Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	<0.01	0.01
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	10.0	0.1
Chromium	<0.01	0.01
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.1	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	<0.1	0.1
Manganese	<0.005	0.005
Molybdenum	0.028	0.005
Nickel	<0.005	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.01	0.01
Silicon	13.6	0.1
Silver	<0.01	0.01
Sodium	0.630	0.05
Strontium	<0.005	0.005
Sulfur	0.119	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	111	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.027	0.005
Zirconium	<0.05	0.05

9/21/99  
RC

Results: Urangane unknown sample

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

RC  
9/21/99

Sample ID  
20-3B

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128602

Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	<0.01	0.01
Arsenic	<0.01	0.01
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	10.0	0.1
Chromium	<0.01	0.01
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.1	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	<0.1	0.1
Manganese	<0.005	0.005
Molybdenum	0.027	0.005
Nickel	<0.005	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	<0.1	0.1
Selenium	<0.01	0.01
Silicon	13.6	0.1
Silver	<0.01	0.01
Sodium	0.630	0.05
Strontium	<0.005	0.005
Sulfur	0.112	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	111	0.1
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	0.021	0.005
Zirconium	<0.05	0.05

9/21/99  
RC

Results: Prepared standard No. 1

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

RC  
9/21/99

Sample ID  
20-1C

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128597

Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	<0.1	0.1
Antimony	0.031	0.01
Arsenic	<0.01	0.01
Barium	0.107	0.005
Beryllium	<0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	<0.005	0.005
Calcium	2.78	0.1
Chromium	0.011	0.01
Cobalt	0.025	0.005
Copper	0.009	0.005
Iron	<0.1	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	2.54	0.1
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	0.019	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	2.13	0.1
Selenium	<0.01	0.01
Silicon	5.22	0.1
Silver	<0.01	0.01
Sodium	9.19	0.2
Strontium	<0.005	0.005
Sulfur	<0.02	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	181	0.1
Vanadium	0.026	0.005
Yttrium	<0.005	0.005
Zinc	0.034	0.005
Zirconium	<0.05	0.05

9/21/99  
nc

Results: Prepared standard No. 2

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128600

Sample ID  
20-2C  
Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	0.248	0.1
Antimony	0.050	0.01
Arsenic	<0.01	0.01
Barium	0.210	0.005
Beryllium	0.005	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	0.010	0.005
Calcium	5.23	0.1
Chromium	0.018	0.01
Cobalt	0.051	0.005
Copper	0.035	0.005
Iron	0.388	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	5.18	0.1
Manganese	0.011	0.005
Molybdenum	<0.005	0.005
Nickel	0.041	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	4.46	0.1
Selenium	<0.01	0.01
Silicon	10.3	0.1
Silver	0.017	0.01
Sodium	19.2	0.2
Strontium	<0.005	0.005
Sulfur	<0.02	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	118	0.1
Vanadium	0.056	0.005
Yttrium	<0.005	0.005
Zinc	0.029	0.005
Zirconium	<0.05	0.05

9/21/99  
nc

Results: Prepared standard No. 3

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 128603

Sample ID  
20-3C  
Client: Division 20  
Date Received: 08/18/99  
Project No.: 20-1402-561  
Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	0.429	0.1
Antimony	0.124	0.01
Arsenic	0.021	0.01
Barium	0.419	0.005
Beryllium	0.010	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	0.010	0.005
Calcium	10.6	0.1
Chromium	0.023	0.01
Cobalt	0.103	0.005
Copper	0.051	0.005
Iron	0.154	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	10.4	0.1
Manganese	0.028	0.005
Molybdenum	<0.005	0.005
Nickel	0.082	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	9.82	0.1
Selenium	0.012	0.01
Silicon	15.5	0.1
Silver	0.022	0.01
Sodium	33.1	0.2
Strontium	<0.005	0.005
Sulfur	<0.02	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	59.2	0.1
Vanadium	0.103	0.005
Yttrium	<0.005	0.005
Zinc	0.056	0.005
Zirconium	<0.05	0.05

9/21/99  
PC

Results: Prepared standard No. 4

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

PC  
9/21/99

Sample ID  
20-4C

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 08/18/99

Matrix: Liquid

Project No.: 20-1402-561

Lab System ID: 128604

Work Order: 16271

Analysis	Sample Result (mg/L)	Detection Limit (mg/L)
Aluminum	0.535	0.1
Antimony	0.149	0.01
Arsenic	0.026	0.01
Barium	0.520	0.005
Beryllium	0.013	0.005
Bismuth	<0.01	0.01
Boron	<0.05	0.05
Cadmium	0.013	0.005
Calcium	13.0	0.1
Chromium	0.027	0.01
Cobalt	0.127	0.005
Copper	0.065	0.005
Iron	0.223	0.1
Lanthanum	<0.005	0.005
Lead	<0.02	0.02
Lithium	<0.005	0.005
Magnesium	12.9	0.1
Manganese	0.037	0.005
Molybdenum	<0.005	0.005
Nickel	0.102	0.005
Palladium	<0.02	0.02
Phosphorus	<0.05	0.05
Potassium	12.7	0.1
Selenium	0.015	0.01
Silicon	20.5	0.1
Silver	0.026	0.01
Sodium	43.5	0.2
Strontium	<0.005	0.005
Sulfur	<0.02	0.02
Thallium	<0.05	0.05
Thorium	<0.05	0.05
Tin	<0.01	0.01
Titanium	<0.05	0.05
Tungsten	<0.02	0.02
Uranium	23.7	0.1
Vanadium	0.128	0.005
Yttrium	<0.005	0.005
Zinc	0.058	0.005
Zirconium	<0.05	0.05

18  
9/22/99  
PC

Uranophane will be synthesized using silicic acid in place of the previously used sodium silicate reagent. The uranophane product will be characterized with special attention toward detecting the presence of Na and S contaminants.

Procedure:

URANOPHANE SYNTHESIS

PC  
9/18/99

WRITTEN BY: J.D. Prikryl  
REVISION NO.: 6

DATE WRITTEN: March 7, 1995  
DATE REVISED: Sept. 16, 1999

OBJECTIVE: to synthesize uranophane  $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 5\text{H}_2\text{O}]$  for use in thermodynamic and coprecipitation experiments.

EQUIPMENT: Parr Model 4522 stirred reaction vessel (2000 ml capacity)  
Teflon liners for reaction vessels  
Parr Model 4843 temperature/pressure controller  
ORION pH/mV/ISE/°C meter  
Ross combination pH electrode  
Analytical balance (Mettler PM 4600)  
Hot plate  
Vacuum pump

SUPPLIES: pH buffers (pH= 1,2,4)  
ultrapure water  
glassware and plasticware as needed  
2000 ml erlenmeyer flasks with stoppers  
Fittings, valves, and tubing as necessary

REAGENTS: Uranyl acetate dihydrate  $[\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ ; MW=424.15 (lot 286908/1 42298)  
Silicic Acid  $\text{SiO}_2 \cdot x\text{H}_2\text{O}$  = about 60 (lot )  
Calcium acetate monohydrate  $[\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$ ; MW=176.19 (lot 944231)  
9 M HCl - diluted from lot 956110

PROCEDURE:

The procedure for uranophane synthesis is based on Cesbron et al., 1993. Reagents will be mixed in the stoichiometric ratio Ca:U:Si = 2:1:1. Although Cesbron et al. do not give information on the amount of solid and  $\text{H}_2\text{O}$  used, they state that good crystallinity is obtained if pH is less than 5.

- Place 35.2 g Ca-acetate, 42.4 g U-acetate, and 2.5 g silicic acid in a 2000 ml teflon liner.
- Transfer approximately 1500 ml of ultrapure  $\text{H}_2\text{O}$  to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the  $\text{H}_2\text{O}$  by boiling on a hotplate under a fumehood..
- Transfer approximately 1350 g of the degassed  $\text{H}_2\text{O}$  to the teflon liner, place in pressure vessel and seal the vessel.



9/18/99  
pc

## Procedure - contd.

- e) Use a vacuum pump attached to the gas outlet of the vessel to remove air from the vessel.
- f) Raise and set the temperature of the solution in the reaction vessel to 150 °C using the temperature controller.
- g) Start stirrer and allow reagents to react for about 10 days.

Weighed mangl acetate 42.4487 g  
 Weighed calcium acetate 25.2868 g  
 Weighed silicic acid 2.5386 g  
 Weighed D.I. water 1,059.6 g  
 (CO<sub>2</sub> free)

Reaction vessel reached 150 °C at 11:30 AM 9/20/99.  
pc 9/18/99

9/28/99  
pc

Dissolution of synthesized mangraphane in 0.1M HCl  
 to collect and quantify insoluble residue from  
 synthesized mangraphane (525/2 and 525/1/2)  
 Procedure:

Weighed and transferred 0.5045g "Mangraphane"  
 to 250 mL vol. flask and filled to mark  
 with 0.1M HCl soln.  
 Added stir bar and placed on stir plate  
 under the hood. Stirred soln. approx. 17 hrs.

9/22/99  
pc

Small undissolved residue was visible in the soln.

Poured mangraphane soln. into a Fisherbrand  
 micropore filter apparatus using a vacuum  
 pump and filter flask.

Pulled air through filter after filtering the 250 mL

9/22/99  
pc

of liquid to dry the residue.

Small quantity of yellow/white solid residue was  
 scrapped with a metal spatula. A <sup>SEM</sup> ~~SEM~~ sample  
 plug was pressed onto the scrapped powder and  
 sample was sent for <sup>SEM</sup> ~~SEM~~ analysis.  
 9/22/99

9/24/99  
pc

## Results of SEM analysis on undissolved residue from Uplane:

File: C:\SIGMA\QUASAR\JFS1196.RPT

[ANALYSIS REPORT]

pc  
9/24/99

## GENERAL CONDITIONS

Result File : JFS1196  
 File Version : 1  
 Background Method : Fit  
 Decon Method : Gaussian  
 Decon ChiSquared : 63.54  
 Analysis Date : 23-SEP-99  
 Microscope : SEM  
 Comments : Sample CH

Element	Line	Weight%	K-Ratio	Cnts/s	Atomic%
Al	Ka	1.52	0.0130	27.04	1.68
Si	Ka	87.98	0.7922	1596.90	93.38
Cl	Ka	0.42	0.0024	3.95	0.35
Ca	Ka	1.03	0.0081	10.29	0.77
Cr	Ka	2.48	0.0207	16.99	1.42
Fe	Ka	2.53	0.0215	13.35	1.35
Ni	Ka	0.45	0.0039	1.77	0.23
Cu	Ka	1.08	0.0090	3.40	0.51
U	Ma	2.51	0.0163	12.08	0.31

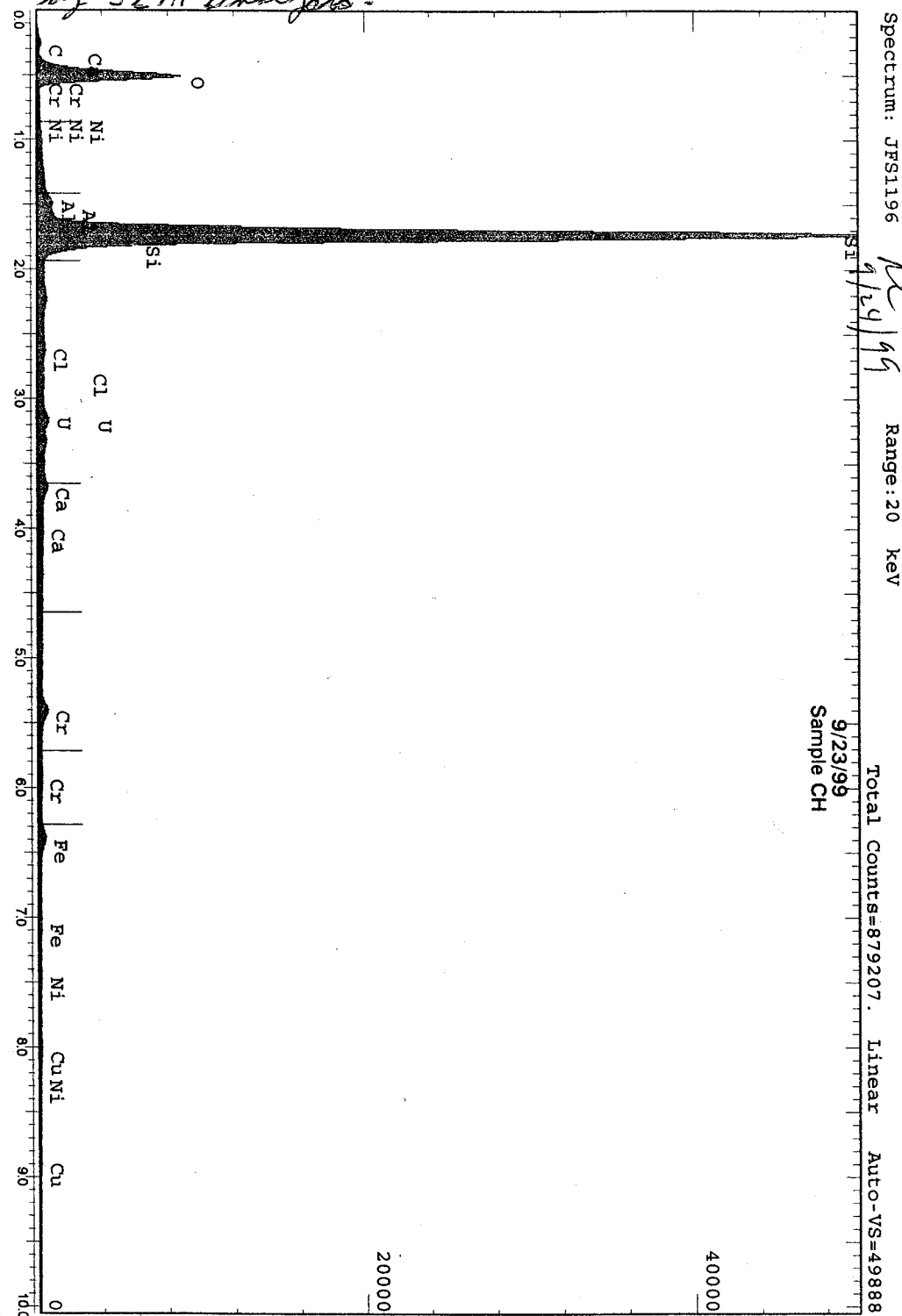
Total 100.00

pc  
9/24/99

9/24/99  
rc

Results - cont'd.

Spectrum graph of undischarged uranophane residue from synthesized uranophane (525/2 and 525/12) by SEM analysis:

9/30/99  
rc

Observation from synthesized uranophane experiment 525/59:

Reaction vessel held a temperature of  $150^{\circ}\text{C}$  for 12 days until heater was shut off on 9/30/99 at 9:00 AM. Vessel was allowed to cool until next day.

10/1/99  
rc

Teflon liner containing product was removed from reaction vessel. Numerous grey specks of material were observed floating on surface of product liquid. The steel stirring blades appeared to have large amounts of corrosive material on them. While the stirring apparatus shaft was discolored, it appears the corrosion occurred only at the blade surfaces.

The grey floating particles were removed using a piece of #5 filter paper to skim off the top surface.

The contents of the teflon liner was poured onto a buchner filtering apparatus using a Whatman #5 filter paper and a vacuum flask and pump.

The crystal material was generally a <sup>10/1/99</sup> yellow color but with greyish tint. Some small particles of orange-yellow color remained settled in the bottom of the teflon liner. These larger orange-yellow flakes were not mixed with the collected product and were discarded.

The product was washed off the filter paper using ultrapure DI water into a 500 mL beaker.

10/4/99  
RL observations - contd.

Uranophane product was filtered through a No. 70 mesh macropore filter into a 1-L filter flask. The small corrosion flakes were collected for possible future analysis. This sample is being held in a petri dish labeled "Syn. Uphase Corrosion Particles."

10/5/99  
RL The collected uranophane product was put in the drying oven overnight.

Another batch of uranophane was prepared for synthesizing in the reaction vessel using the same procedure and reagents as described on 325/59. The reagent lot numbers were the same, with the exception of uranyl acetate.

Uranyl acetate

Fluka Chemika

Analysis No. 386908/1 4.3398

Weights of reagents used for uranophane synthesis:

Calcium acetate	35.19 g
Uranyl acetate	42.53 g
Silicic acid	2.50 g
D.I Water	1,100 g

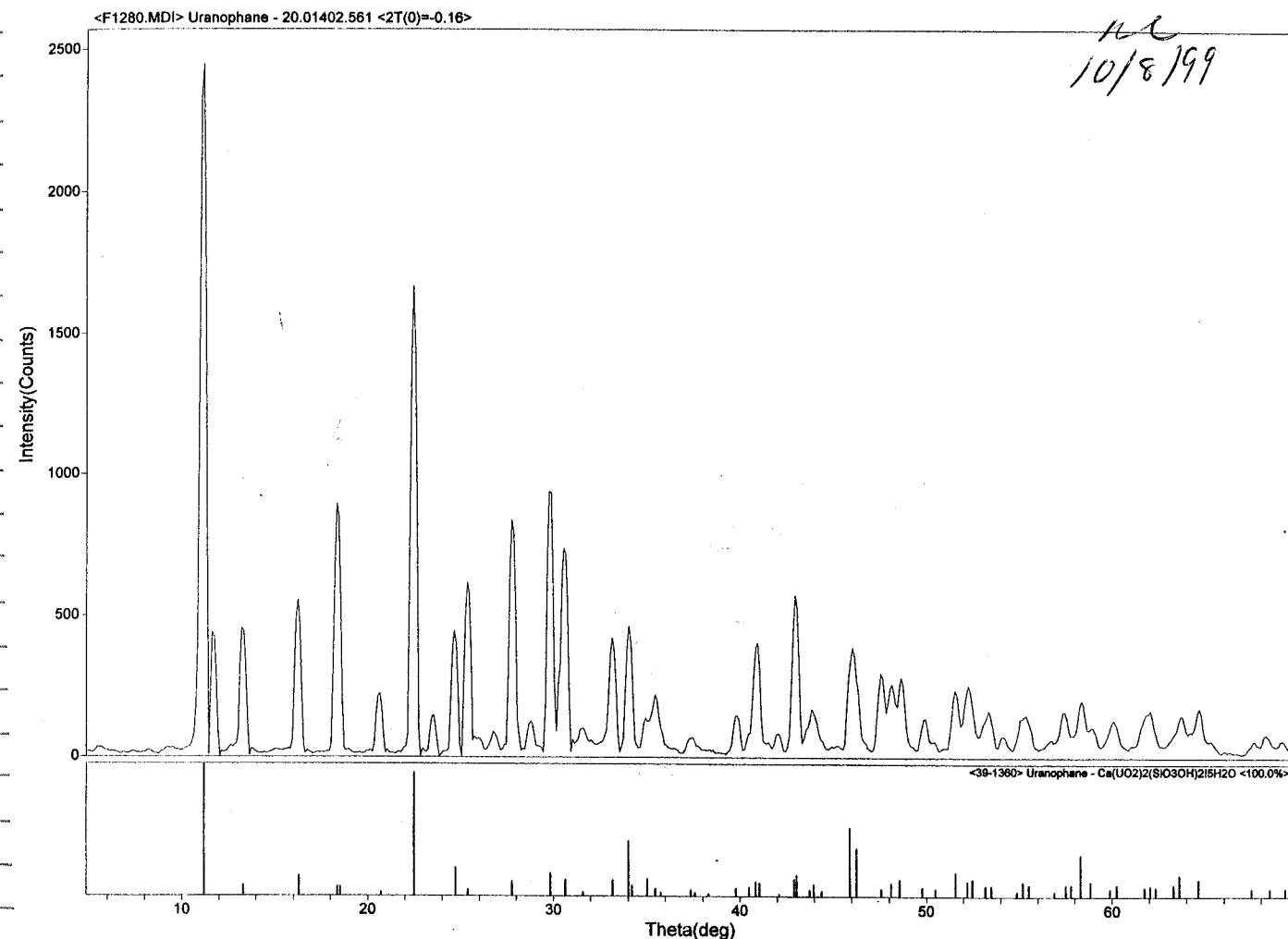
The stainless steel stirring blade used in the reaction vessel was removed and replaced with an in-lab. fabricated blade made from a teflon bottle cap.

The reaction vessel reached 150°C at 1530.

10/6/99  
RL Observation:

A sample of ~1 gram of uranophane was sent to Div. for XRD analysis. Sample was labeled "20-Uphase."

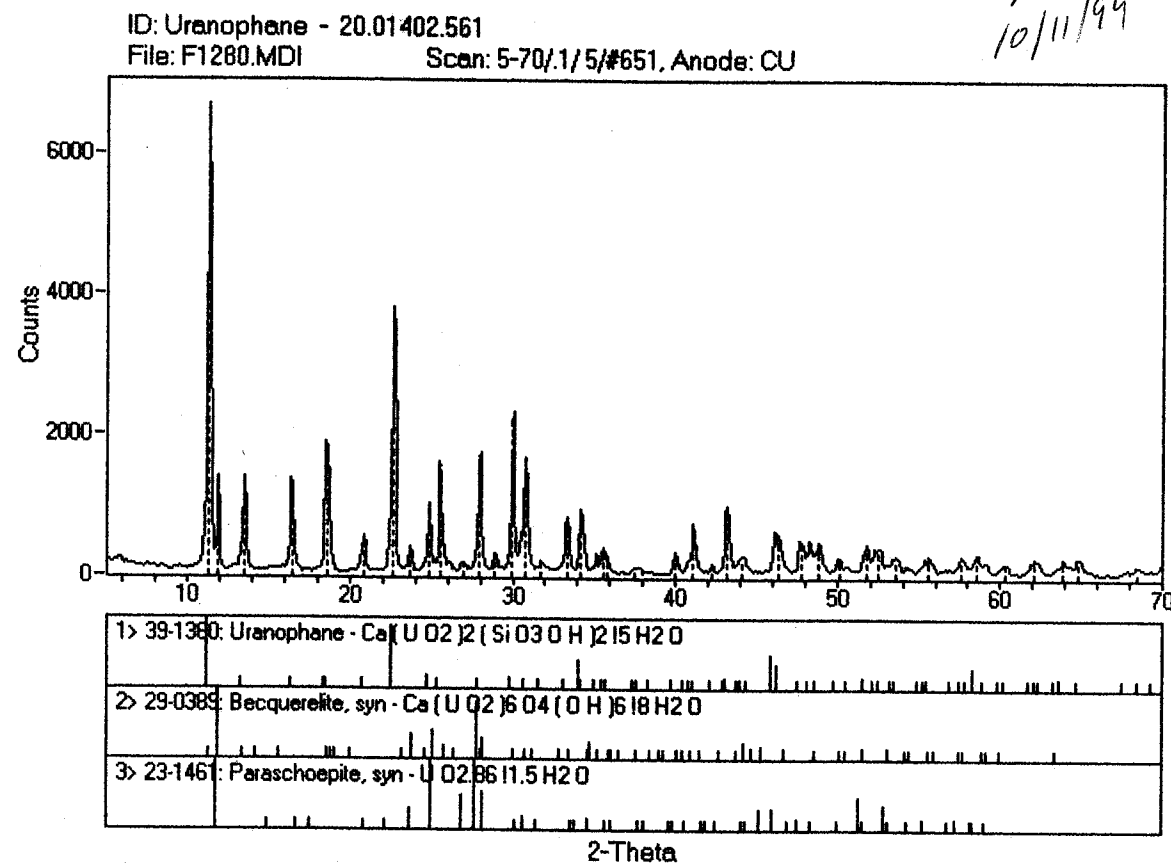
10/8/99  
RL Results: XRD analysis of syn. uranophane, "20-Uphase".



10/11/99  
re

Results - cont'd.

Matched spectrograms of minerals from the JADE library to syn. uranophane XRD analysis.



re 10/11/99

10/19/99  
re

Observations - cont'd.

Reaction vessel was shut down at 0830 and allowed to cool until 1500. The teflon container was removed and placed in the hood. Small black/grey specks of contaminant were observed floating on the liquid surface, as in the previous experiment. The water was decanted into the radioactive waste containers and the yellow/orange crystal product was placed onto a buchner filtering apparatus and allowed to air dry overnight. Small black specks were intermingled throughout the product.

10/20/99  
re

Observations - cont'd.

All reaction vessel parts that had been in contact with the reaction solution were cleaned and inspected for corrosion. No obvious deterioration was found. A large quantity of black powder was found deposited on the inside walls of a nylon bushing used to support the stirring rod.

It was determined that a sample of the product would be sent for XRD analysis. A sample was prepared and labeled "20-Uphane 2".

Another batch of uranophane reagents will be attempted using the reaction vessel minus the stirring mechanism. A different container head <sup>will be</sup> ~~was~~ used that has only one threaded opening, which will be plugged with a nylon threaded plug. The reagent mixture will not be stirred and the silicic acid reagent will be replaced with sodium silicate. Approximately 1360 mL of  $\text{H}_2\text{O}$  will be added to assure complete dissolution of the reagents.

10/21/99  
re

## Uranophane synthesis - batch No. 5

Procedure: The same procedure and reagents/equipment was used as per 325/2, with the exception of having ~~removed the~~ used a reaction vessel head that did not have a stirring rod mechanism.

Reagents: Uranyl Acetate - Analysis No. 386908/1  
Calcium acetate - Lot No. 944231  
Sodium silicate - Lot No. 942853A

Wts. of reagents:

Uranyl acetate	42.4281 g
Calcium acetate	35.2459 g
Sodium silicate	28.2438 g
CO <sub>2</sub> free ultrapure H <sub>2</sub> O	1,380.00 g

Reaction vessel reached 150°C at ~1500  
10/21/99.

11/2/99  
re

Observations: Uranophane product was collected 11/2/99. The solid material looked uniform in size of crystals and color, light yellow. No contaminants were observed in the solid or the liquid of the reaction vessel.

Solid was mixed with ~150 mL ultrapure D.I. water 3 times in a 250 mL beaker. The solid was allowed to settle each time and the clear liquid was decanted into the hazardous waste bottles.

The solid product was dried in the oven overnight and crushed, then collected in a glass sample.

10/28/99  
re

## Uranophane synthesis - batch No. 6

Procedure: Same as per 325/2. Equipment was modified by removing stirring rod mechanism. Black powder was found coating the threads of the stirring rod. The powder appeared to be burned thread lubricant rather than corrosion product. The threads were cleaned and plugged with nylon threaded plugs.

Reagents: Uranyl acetate - Analysis No. 386908/1  
Calcium acetate - Lot No. 944231  
Sodium silicate - Lot No. 942853A

Wt. of reagents:

re	Calcium	
10/28/99	Uranyl acetate	35.36 g
	Na silicate	28.75 g
	Uranyl acetate	42.57 g
	CO <sub>2</sub> free water	~ 1400 g

11/9/99  
re

Observations: Product was removed from reaction vessel. No contamination was observed, although some of the liquid reagents had apparently overflowed from the teflon container into the steel holding vessel, causing some crystal formation to occur on the steel vessel bottom. This spilled material was not collected as part of the sample.

11/10/99  
re

The uranophane product was filtered using the Buchner filter apparatus and a No. 2 Whatman paper. The collected product was washed several times in a beaker using nanopure water.

11/11/99  
re

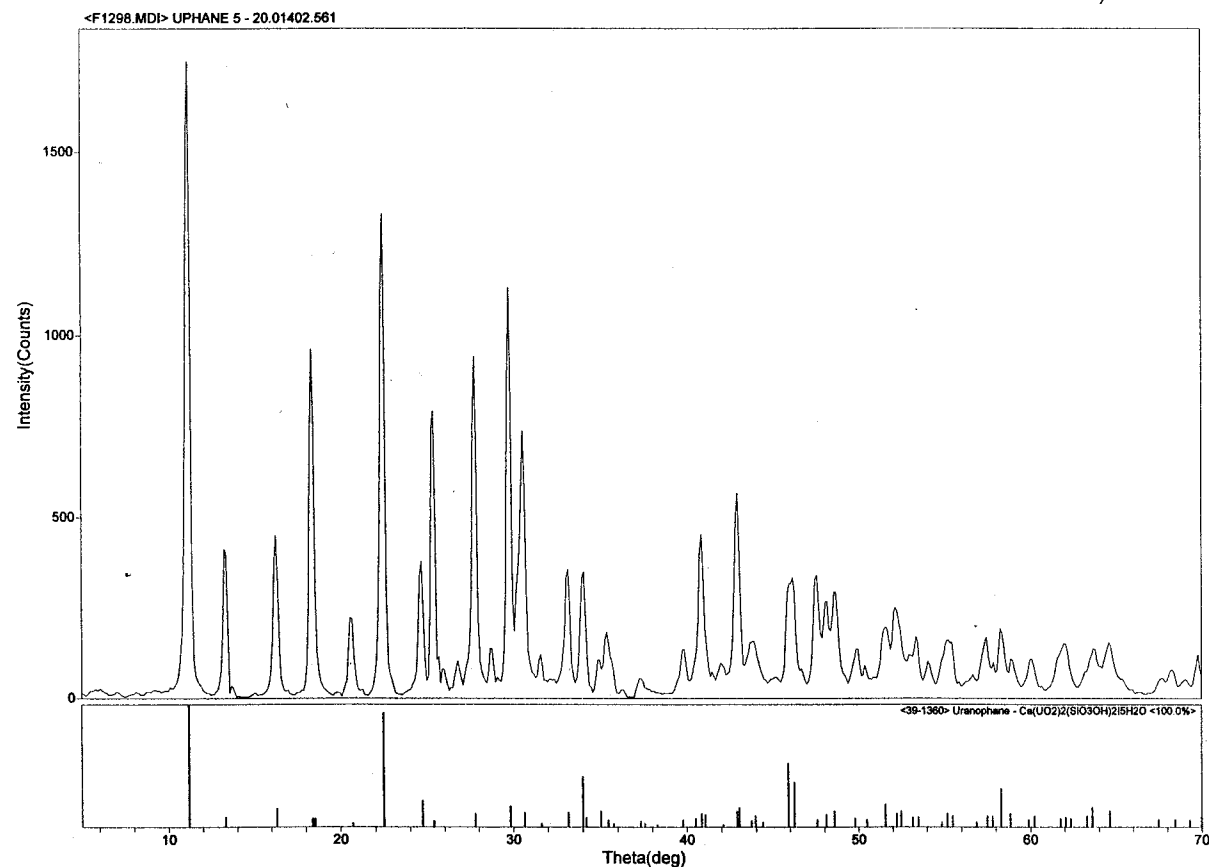
Product was dried in the oven, then collected. Total collected product = 24.52 g and was labeled "Syn. Uranophane No. 6 325/69".



11/2/99  
RC  
Observations - cont'd from pg 68: Uranophane No. 5  
11/2/99  
jar and labeled "Syn. Uranophane No. 5 225/68."  
Sample stored in hood 57-2.

11/18/99  
RC  
11/12/99  
Approximately 2 grams each of samples Syn. Uranophane No. 5 and Syn. Uranophane No. 6 were analyzed by XRD.

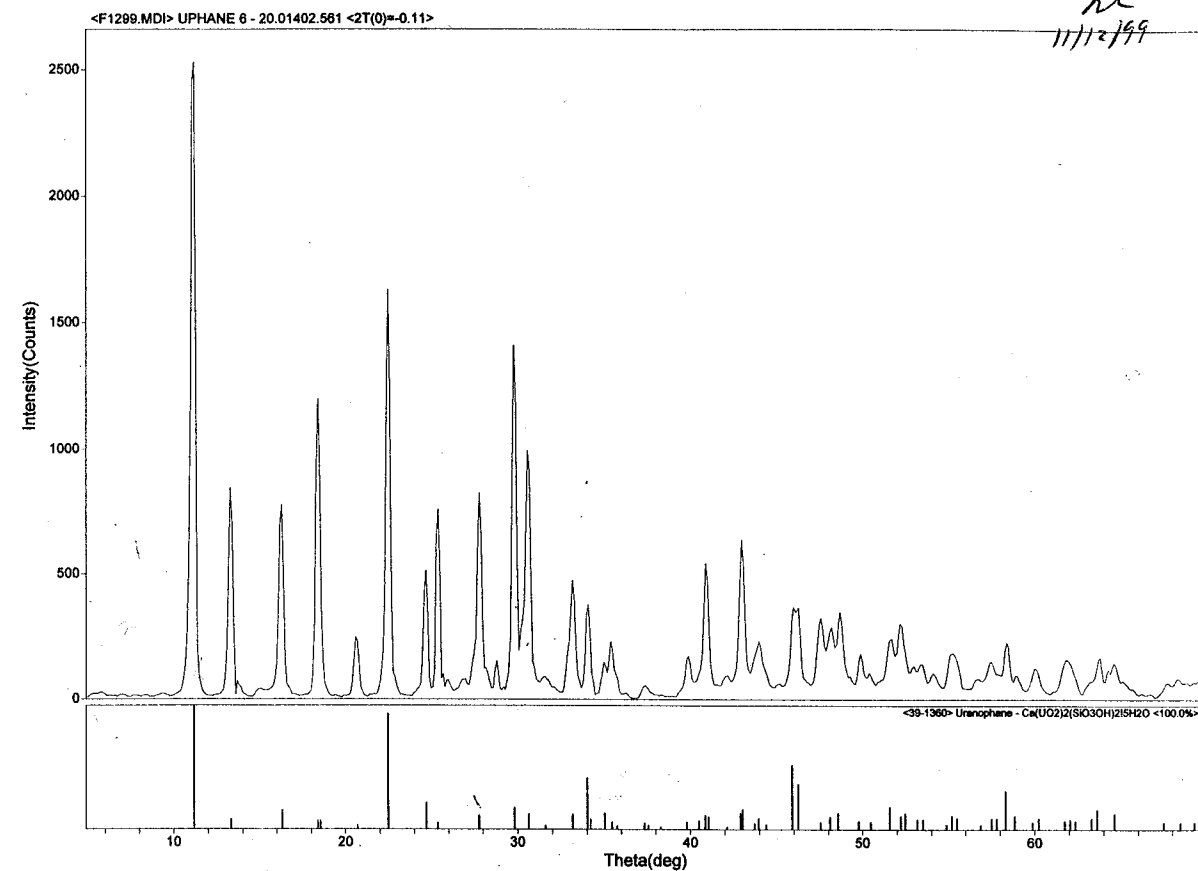
Results of XRD analysis:  
Uranophane No. 5



Southwest Research Institute

&lt;winjadedata&gt; Thursday, Nov 11, 1999 @04:06p

Results -  
cont'd. Uranophane No. 6



Southwest Research Institute

&lt;winjadedata&gt; Thursday, Nov 11, 1999 @04:12p

RC  
11/12/99

11/15/99  
 Dissolution of Syn Uranophane No. 5 and No. 6 for samples to be analyzed by Div. 01 for elemental composition.

Materials & reagents:

0.1 M HCl (prepared 9/9/99 by RC)  
 Syn. Uranophane No. 5 (325/68)  
 Syn. Uranophane No. 6 (325/69)  
 Analytical balance - Mettler AE 240  
 100 mL volumetric flasks  
 Stirrer - Corning  
 Nanopure D.I. water

Procedure: Weighed approx. 0.1 g of Uranophane No. 5 + No. 6 and placed in 100 mL volumetric flasks, 3 samples of each batch.

11/15/99  
 Weighed approx. 0.2 g of Uranophane No. 5 + No. 6 into 100 mL vol. flasks, 1 sample for each batch.

Samples	Sample Wt. (grams)
20-5a	0.1042
20-5b	0.1066
20-5c	0.2060
20-6a	0.1072
20-6b	0.1073
20-6c	0.2080

Weighed samples were added to 100 mL vol. flasks and filled to mark with 0.1 M HCl. Solutions were stirred on stirring plates for 4 hours and then poured into individually marked 60 mL polypropylene containers and sent to Div. 01 for analysis.

11/16/99  
 Uranium, silicon and calcium standards were sent to Div. 01 with Syn. Uranophane solutions. These standards were prepared and are labeled as per 20-325/45-47.

2/21/00  
 Preparation of  $10^{-3}$  molar aqueous silica solution:

Objective - make a  $10^{-3}$  molar aqueous silica soln. for use in uranophane solubility and co-precipitation experiments.

Method - dissolve silicic acid in nanopure  $H_2O$ .

Materials & Equipment -

- silicic acid ( $SiO_2 \cdot xH_2O$ )  
 Fisher Chemical  
 Lot No. 953433

- nanopure  $H_2O$   
 - Fisher versabath  
 - 1 L PP bottles  
 - 20 L PP storage bottle  
 - drying oven  
 - Whatman No. 5 filter paper + plastic funnel.

Procedure:

1. Transferred approx. 5 grams of silicic acid to a 125 mL beaker and placed in drying oven overnight to remove adsorbed water.

2/23/00  
 m

2. Added 1 L of nanopure  $H_2O$  to 20 L PP bottles.

3. Added approximately 0.07 gram of the oven dried silicic acid to tared 1 L bottles and

2/23/00  
re

procedure - cont'd.

3. capped tightly.

4. Placed 10 of the liter bottles in a water bath/shaker set at 90°C for 1 week. Loosened caps.

5. Removed bottles and allowed to cool. Filtered contents through a 45 <sup>2/23/00</sup> micron filter using a Fisherbrand filtering apparatus.6. Placed remaining 10 bottles in the water bath/shaker under the same conditions as above.  
Removed <sup>2/23/00</sup>

7. Removed bottles and filtered contents.

8. Filtered silica solution was placed in the 20 L PP storage bottle and labeled as "Silica Solution."

3/28/00  
re

PH measurement of Silica soln.

Orion model 920A pH meter calibrated using a 3 pt. standard calibration with 4.0, 7.0, and 10.0 pH buffer solns.

slope = 99.6

measured pH value of silica soln. = 5.56

re  
2/22/00

Results: Uranophane 5 ICP analysis

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-SA	135311	Aluminum	0.100	U	mg/L	0.1		
20-SA	135311	Antimony	0.010	U	mg/L	0.01		
20-SA	135311	Arsenic	0.010	U	mg/L	0.01		
20-SA	135311	Barium	0.005	U	mg/L	0.005		
20-SA	135311	Beryllium	0.005	U	mg/L	0.005		
20-SA	135311	Bismuth	0.020	U	mg/L	0.02		
20-SA	135311	Boron	0.100	U	mg/L	0.1		
20-SA	135311	Cadmium	0.005	U	mg/L	0.005		
20-SA	135311	Calcium	44.4		mg/L	0.1		
20-SA	135311	Chromium	0.010	U	mg/L	0.01		
20-SA	135311	Cobalt	0.005	U	mg/L	0.005		
20-SA	135311	Copper	0.010	U	mg/L	0.01		
20-SA	135311	Iron	0.250	U	mg/L	0.25		
20-SA	135311	Lanthanum	0.005	U	mg/L	0.005		
20-SA	135311	Lead	0.050	U	mg/L	0.05		
20-SA	135311	Lithium	0.005	U	mg/L	0.005		
20-SA	135311	Magnesium	0.100	U	mg/L	0.1		
20-SA	135311	Manganese	0.050	U	mg/L	0.05		
20-SA	135311	Molybdenum	0.136		mg/L	0.005		
20-SA	135311	Nickel	0.007		mg/L	0.005		
20-SA	135311	Palladium	0.020	U	mg/L	0.02		
20-SA	135311	Phosphorus	0.050	U	mg/L	0.05		
20-SA	135311	Potassium	0.100	U	mg/L	0.1		
20-SA	135311	Selenium	0.050	U	mg/L	0.05		
20-SA	135311	Silicon	63.6		mg/L	1		
20-SA	135311	Silver	0.010	U	mg/L	0.01		
20-SA	135311	Sodium	0.753		mg/L	0.1		
20-SA	135311	Strontium	0.005		mg/L	0.005		
20-SA	135311	Sulfur	0.200	U	mg/L	0.2		
20-SA	135311	Thallium	0.050	U	mg/L	0.05		
20-SA	135311	Thorium	0.500	U	mg/L	0.5		
20-SA	135311	Tin	0.010	U	mg/L	0.01		
20-SA	135311	Titanium	0.050	U	mg/L	0.05		
20-SA	135311	Tungsten	0.020	U	mg/L	0.02		
20-SA	135311	Uranium	518		mg/L	10		
20-SA	135311	Vanadium	0.005	U	mg/L	0.005		
20-SA	135311	Yttrium	0.005	U	mg/L	0.005		
20-SA	135311	Zinc	0.005	U	mg/L	0.005		
20-SA	135311	Zirconium	0.050	U	mg/L	0.05		

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-SA	135311 Duplicate	Aluminum	0.103		mg/L	0.1		
20-SA	135311 Duplicate	Antimony	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Arsenic	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Barium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Beryllium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Bismuth	0.020	U	mg/L	0.02		
20-SA	135311 Duplicate	Boron	0.100	U	mg/L	0.1		
20-SA	135311 Duplicate	Cadmium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Calcium	44.2		mg/L	0.1		
20-SA	135311 Duplicate	Chromium	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Cobalt	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Copper	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Iron	0.250	U	mg/L	0.25		
20-SA	135311 Duplicate	Lanthanum	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Lead	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Lithium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Magnesium	0.100	U	mg/L	0.1		
20-SA	135311 Duplicate	Manganese	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Molybdenum	0.137		mg/L	0.005		
20-SA	135311 Duplicate	Nickel	0.007		mg/L	0.005		
20-SA	135311 Duplicate	Palladium	0.020	U	mg/L	0.02		
20-SA	135311 Duplicate	Phosphorus	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Potassium	0.100	U	mg/L	0.1		
20-SA	135311 Duplicate	Selenium	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Silicon	63.7		mg/L	1		
20-SA	135311 Duplicate	Silver	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Sodium	0.749		mg/L	0.1		
20-SA	135311 Duplicate	Strontium	0.005		mg/L	0.005		
20-SA	135311 Duplicate	Sulfur	0.200	U	mg/L	0.2		
20-SA	135311 Duplicate	Thallium	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Thorium	0.500	U	mg/L	0.5		
20-SA	135311 Duplicate	Tin	0.010	U	mg/L	0.01		
20-SA	135311 Duplicate	Titanium	0.050	U	mg/L	0.05		
20-SA	135311 Duplicate	Tungsten	0.020	U	mg/L	0.02		
20-SA	135311 Duplicate	Uranium	513		mg/L	10		
20-SA	135311 Duplicate	Vanadium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Yttrium	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Zinc	0.005	U	mg/L	0.005		
20-SA	135311 Duplicate	Zirconium	0.050	U	mg/L	0.05		

re  
3/28/00

Results: Uranophore No. 5 ICP analysis

PROJECT # 20.01402.561

3/28/00

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-5B	135312	Aluminum	0.100	U	mg/L	0.1		
20-5B	135312	Antimony	0.010	U	mg/L	0.01		
20-5B	135312	Arsenic	0.010	U	mg/L	0.01		
20-5B	135312	Barium	0.005	U	mg/L	0.005		
20-5B	135312	Beryllium	0.005	U	mg/L	0.005		
20-5B	135312	Bismuth	0.020	U	mg/L	0.02		
20-5B	135312	Boron	0.100	U	mg/L	0.1		
20-5B	135312	Cadmium	0.005	U	mg/L	0.005		
20-5B	135312	Calcium	43.0	U	mg/L	0.1		
20-5B	135312	Chromium	0.011	U	mg/L	0.01		
20-5B	135312	Cobalt	0.005	U	mg/L	0.005		
20-5B	135312	Copper	0.011	U	mg/L	0.01		
20-5B	135312	Iron	0.250	U	mg/L	0.25		
20-5B	135312	Lanthanum	0.005	U	mg/L	0.005		
20-5B	135312	Lead	0.050	U	mg/L	0.05		
20-5B	135312	Lithium	0.005	U	mg/L	0.005		
20-5B	135312	Magnesium	0.100	U	mg/L	0.1		
20-5B	135312	Manganese	0.050	U	mg/L	0.05		
20-5B	135312	Molybdenum	0.134	U	mg/L	0.005		
20-5B	135312	Nickel	0.011	U	mg/L	0.005		
20-5B	135312	Palladium	0.020	U	mg/L	0.02		
20-5B	135312	Phosphorus	0.050	U	mg/L	0.05		
20-5B	135312	Potassium	0.100	U	mg/L	0.1		
20-5B	135312	Selenium	0.050	U	mg/L	0.05		
20-5B	135312	Silicon	61.6	U	mg/L	1		
20-5B	135312	Silver	0.010	U	mg/L	0.01		
20-5B	135312	Sodium	0.781	U	mg/L	0.1		
20-5B	135312	Strontium	0.005	U	mg/L	0.005		
20-5B	135312	Sulfur	0.200	U	mg/L	0.2		
20-5B	135312	Thallium	0.050	U	mg/L	0.05		
20-5B	135312	Thorium	0.500	U	mg/L	0.5		
20-5B	135312	Tin	0.010	U	mg/L	0.01		
20-5B	135312	Titanium	0.050	U	mg/L	0.05		
20-5B	135312	Tungsten	0.020	U	mg/L	0.02		
20-5B	135312	Uranium	504	U	mg/L	10		
20-5B	135312	Vanadium	0.005	U	mg/L	0.005		
20-5B	135312	Yttrium	0.005	U	mg/L	0.005		
20-5B	135312	Zinc	0.455	U	mg/L	0.005		
20-5B	135312	Zirconium	0.050	U	mg/L	0.05		

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-5C	135313	Aluminum	0.100	U	mg/L	0.1		
20-5C	135313	Antimony	0.010	U	mg/L	0.01		
20-5C	135313	Arsenic	0.010	U	mg/L	0.01		
20-5C	135313	Barium	0.005	U	mg/L	0.005		
20-5C	135313	Beryllium	0.005	U	mg/L	0.005		
20-5C	135313	Bismuth	0.020	U	mg/L	0.02		
20-5C	135313	Boron	0.100	U	mg/L	0.1		
20-5C	135313	Cadmium	0.005	U	mg/L	0.005		
20-5C	135313	Calcium	82.5	U	mg/L	0.1		
20-5C	135313	Chromium	0.042	U	mg/L	0.01		
20-5C	135313	Cobalt	0.013	U	mg/L	0.005		
20-5C	135313	Copper	0.010	U	mg/L	0.01		
20-5C	135313	Iron	0.250	U	mg/L	0.25		
20-5C	135313	Lanthanum	0.050	U	mg/L	0.05		
20-5C	135313	Lead	0.050	U	mg/L	0.05		
20-5C	135313	Lithium	0.005	U	mg/L	0.005		
20-5C	135313	Magnesium	1.00	U	mg/L	1		
20-5C	135313	Manganese	0.050	U	mg/L	0.05		
20-5C	135313	Molybdenum	0.263	U	mg/L	0.005		
20-5C	135313	Nickel	0.021	U	mg/L	0.005		
20-5C	135313	Palladium	0.020	U	mg/L	0.02		
20-5C	135313	Phosphorus	0.050	U	mg/L	0.05		
20-5C	135313	Potassium	0.100	U	mg/L	0.1		
20-5C	135313	Selenium	0.050	U	mg/L	0.05		
20-5C	135313	Silicon	120	U	mg/L	1		
20-5C	135313	Silver	0.010	U	mg/L	0.01		
20-5C	135313	Sodium	1.79	U	mg/L	0.1		
20-5C	135313	Strontium	0.010	U	mg/L	0.005		
20-5C	135313	Sulfur	0.200	U	mg/L	0.2		
20-5C	135313	Thallium	0.050	U	mg/L	0.05		
20-5C	135313	Thorium	0.500	U	mg/L	0.5		
20-5C	135313	Tin	0.010	U	mg/L	0.01		
20-5C	135313	Titanium	0.050	U	mg/L	0.05		
20-5C	135313	Tungsten	0.020	U	mg/L	0.02		
20-5C	135313	Uranium	974	U	mg/L	10		
20-5C	135313	Vanadium	0.005	U	mg/L	0.005		
20-5C	135313	Yttrium	0.005	U	mg/L	0.005		
20-5C	135313	Zinc	0.050	U	mg/L	0.05		
20-5C	135313	Zirconium	0.500	U	mg/L	0.5		

Results: Uranophore No. 6 ICP analysis

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-6A	135314	Aluminum	0.104	U	mg/L	0.1		
20-6A	135314	Antimony	0.010	U	mg/L	0.01		
20-6A	135314	Arsenic	0.010	U	mg/L	0.01		
20-6A	135314	Barium	0.005	U	mg/L	0.005		
20-6A	135314	Beryllium	0.005	U	mg/L	0.005		
20-6A	135314	Bismuth	0.020	U	mg/L	0.02		
20-6A	135314	Boron	0.100	U	mg/L	0.1		
20-6A	135314	Cadmium	0.005	U	mg/L	0.005		
20-6A	135314	Calcium	42.1	U	mg/L	0.1		
20-6A	135314	Chromium	0.015	U	mg/L	0.01		
20-6A	135314	Cobalt	0.008	U	mg/L	0.005		
20-6A	135314	Copper	0.010	U	mg/L	0.01		
20-6A	135314	Iron	0.250	U	mg/L	0.25		
20-6A	135314	Lanthanum	0.050	U	mg/L	0.05		
20-6A	135314	Lead	0.050	U	mg/L	0.05		
20-6A	135314	Lithium	0.005	U	mg/L	0.005		
20-6A	135314	Magnesium	1.00	U	mg/L	1		
20-6A	135314	Manganese	0.050	U	mg/L	0.05		
20-6A	135314	Molybdenum	0.136	U	mg/L	0.005		
20-6A	135314	Nickel	0.010	U	mg/L	0.005		
20-6A	135314	Palladium	0.020	U	mg/L	0.02		
20-6A	135314	Phosphorus	0.050	U	mg/L	0.05		
20-6A	135314	Potassium	0.100	U	mg/L	0.1		
20-6A	135314	Selenium	0.050	U	mg/L	0.05		
20-6A	135314	Silicon	61.3	U	mg/L	1		
20-6A	135314	Silver	0.010	U	mg/L	0.01		
20-6A	135314	Sodium	0.814	U	mg/L	0.1		
20-6A	135314	Strontium	0.005	U	mg/L	0.005		
20-6A	135314	Sulfur	0.200	U	mg/L	0.2		
20-6A	135314	Thallium	0.050	U	mg/L	0.05		
20-6A	135314	Thorium	0.500	U	mg/L	0.5		
20-6A	135314	Tin	0.010	U	mg/L	0.01		
20-6A	135314	Titanium	0.050	U	mg/L	0.05		
20-6A	135314	Tungsten	0.020	U	mg/L	0.02		
20-6A	135314	Uranium	502	U	mg/L	10		
20-6A	135314	Vanadium	0.005	U	mg/L	0.005		
20-6A	135314	Yttrium	0.005	U	mg/L	0.005		
20-6A	135314	Zinc	0.050	U	mg/L	0.05		
20-6A	135314	Zirconium	0.050	U	mg/L	0.05		

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-6B	135315	Aluminum	0.100	U	mg/L	0.1		
20-6B	135315	Antimony	0.010	U	mg/L	0.01		
20-6B	135315	Arsenic	0.010	U	mg/L	0.01		
20-6B	135315	Barium	0.005	U	mg/L	0.005		
20-6B	135315	Beryllium	0.005	U	mg/L	0.005		
20-6B	135315	Bismuth	0.020	U	mg/L	0.02		
20-6B	135315	Boron	0.100	U	mg/L	0.1		
20-6B	135315	Cadmium	0.005	U	mg/L	0.005		
20-6B	135315	Calcium	42.8	U	mg/L	0.1		
20-6B	135315	Chromium	0.015	U	mg/L	0.01		
20-6B	135315	Cobalt	0.005	U	mg/L	0.005		
20-6B	135315	Copper	0.010	U	mg/L	0.01		
20-6B	135315	Iron	0.250	U	mg/L	0.25		
20-6B	135315	Lanthanum	0.050	U	mg/L	0.05		
20-6B	135315	Lead	0.050	U	mg/L	0.05		
20-6B	135315	Lithium	0.005	U	mg/L	0.005		
20-6B	135315	Magnesium	1.00	U	mg/L	1		
20-6B	135315	Manganese	0.050	U	mg/L	0.05		
20-6B	135315	Molybdenum	0.143	U	mg/L	0.005		
20-6B	135315	Nickel	0.015	U	mg/L	0.005		
20-6B	135315	Palladium	0.020	U	mg/L	0.02		
20-6B	135315	Phosphorus	0.050	U	mg/L	0.05		
20-6B	135315	Potassium	0.100	U	mg/L	0.1		
20-6B	135315	Selenium	0.050	U	mg/L	0.05		
20-6B	135315	Silicon	61.9	U	mg/L	1		
20-6B	135315	Silver	0.010	U	mg/L	0.01		
20-6B	135315	Sodium	1.03	U	mg/L	0.1		
20-6B	135315	Strontium	0.005	U	mg/L	0.005		
20-6B	135315	Sulfur	0.200	U	mg/L	0.2		
20-6B	135315	Thallium	0.050	U	mg/L	0.05		
20-6B	135315	Thorium	0.500	U	mg/L	0.5		
20-6B	135315	Tin	0.010	U	mg/L	0.01		
20-6B	135315	Titanium	0.050	U	mg/L	0.05		
20-6B	135315	Tungsten	0.020	U	mg/L	0.02		
20-6B	135315	Uranium	508	U	mg/L	10		
20-6B	135315	Vanadium	0.005	U	mg/L	0.005		
20-6B	135315	Yttrium	0.005	U	mg/L	0.005		
20-6B	135315	Zinc	0.050	U	mg/L	0.05		
20-6B	135315	Zirconium	0.050	U	mg/L	0.05		

Results: Uranophane No. 6 analysis

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-6C	135316	Aluminum	0.100	U	mg/L	0.1		
20-6C	135316	Antimony	0.010	U	mg/L	0.01		
20-6C	135316	Arsenic	0.010	U	mg/L	0.01		
20-6C	135316	Barium	0.005	U	mg/L	0.005		
20-6C	135316	Beryllium	0.005	U	mg/L	0.005		
20-6C	135316	Bismuth	0.020	U	mg/L	0.02		
20-6C	135316	Boron	0.100	U	mg/L	0.1		
20-6C	135316	Cadmium	0.005	U	mg/L	0.005		
20-6C	135316	Calcium	83.3		mg/L	0.1		
20-6C	135316	Chromium	0.048		mg/L	0.01		
20-6C	135316	Cobalt	0.009		mg/L	0.005		
20-6C	135316	Copper	0.010	U	mg/L	0.01		
20-6C	135316	Iron	0.250	U	mg/L	0.25		
20-6C	135316	Lanthanum	0.050	U	mg/L	0.05		
20-6C	135316	Lead	0.050	U	mg/L	0.05		
20-6C	135316	Lithium	0.005	U	mg/L	0.005		
20-6C	135316	Magnesium	1.00	U	mg/L	1		
20-6C	135316	Manganese	0.050	U	mg/L	0.05		
20-6C	135316	Molybdenum	0.261		mg/L	0.005		
20-6C	135316	Nickel	0.016		mg/L	0.005		
20-6C	135316	Palladium	0.020	U	mg/L	0.02		
20-6C	135316	Phosphorus	0.050	U	mg/L	0.05		
20-6C	135316	Potassium	0.100	U	mg/L	0.1		
20-6C	135316	Selenium	0.050	U	mg/L	0.05		
20-6C	135316	Silicon	121		mg/L	1		
20-6C	135316	Silver	0.010	U	mg/L	0.01		
20-6C	135316	Sodium	1.62		mg/L	0.1		
20-6C	135316	Strontium	0.010		mg/L	0.005		
20-6C	135316	Sulfur	0.200	U	mg/L	0.2		
20-6C	135316	Thallium	0.050	U	mg/L	0.05		
20-6C	135316	Thorium	0.500	U	mg/L	0.5		
20-6C	135316	Tin	0.010	U	mg/L	0.01		
20-6C	135316	Titanium	0.050	U	mg/L	0.05		
20-6C	135316	Tungsten	0.020	U	mg/L	0.02		
20-6C	135316	Uranium	991		mg/L	10		
20-6C	135316	Vanadium	0.005	U	mg/L	0.005		
20-6C	135316	Yttrium	0.005	U	mg/L	0.005		
20-6C	135316	Zinc	0.050	U	mg/L	0.05		
20-6C	135316	Zirconium	0.500	U	mg/L	0.5		

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Results: Ca, U, Si standards ICP analysis

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-1C	135317	Aluminum	0.183		mg/L	0.1		
20-1C	135317	Antimony	0.029		mg/L	0.01		
20-1C	135317	Arsenic	0.010	U	mg/L	0.01		
20-1C	135317	Barium	0.107		mg/L	0.005		
20-1C	135317	Beryllium	0.005	U	mg/L	0.005		
20-1C	135317	Bismuth	0.020	U	mg/L	0.02		
20-1C	135317	Boron	0.100	U	mg/L	0.1		
20-1C	135317	Cadmium	0.005	U	mg/L	0.005		
20-1C	135317	Calcium	2.49		mg/L	0.1		
20-1C	135317	Chromium	0.010	U	mg/L	0.01		
20-1C	135317	Cobalt	0.025		mg/L	0.005		
20-1C	135317	Copper	0.012		mg/L	0.01		
20-1C	135317	Iron	0.250	U	mg/L	0.25		
20-1C	135317	Lanthanum	0.084		mg/L	0.005		
20-1C	135317	Lead	0.050	U	mg/L	0.05		
20-1C	135317	Lithium	0.005	U	mg/L	0.005		
20-1C	135317	Magnesium	2.35		mg/L	1		
20-1C	135317	Manganese	0.005	U	mg/L	0.005		
20-1C	135317	Molybdenum	0.013		mg/L	0.005		
20-1C	135317	Nickel	0.019		mg/L	0.005		
20-1C	135317	Palladium	0.020	U	mg/L	0.02		
20-1C	135317	Phosphorus	0.050	U	mg/L	0.05		
20-1C	135317	Potassium	1.92		mg/L	0.1		
20-1C	135317	Selenium	0.050	U	mg/L	0.05		
20-1C	135317	Silicon	5.04		mg/L	0.1		
20-1C	135317	Silver	0.010	U	mg/L	0.01		
20-1C	135317	Sodium	9.70		mg/L	1		
20-1C	135317	Strontium	0.005	U	mg/L	0.005		
20-1C	135317	Sulfur	0.200	U	mg/L	0.2		
20-1C	135317	Thallium	0.050	U	mg/L	0.05		
20-1C	135317	Thorium	0.500	U	mg/L	0.5		
20-1C	135317	Tin	0.010	U	mg/L	0.01		
20-1C	135317	Titanium	0.050	U	mg/L	0.05		
20-1C	135317	Tungsten	0.020	U	mg/L	0.02		
20-1C	135317	Uranium	173		mg/L	10		
20-1C	135317	Vanadium	0.025		mg/L	0.005		
20-1C	135317	Yttrium	0.005	U	mg/L	0.005		
20-1C	135317	Zinc	0.016		mg/L	0.005		
20-1C	135317	Zirconium	0.050	U	mg/L	0.05		

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-2C	135318	Aluminum	0.275		mg/L	0.1		
20-2C	135318	Antimony	0.047		mg/L	0.01		
20-2C	135318	Arsenic	0.010	U	mg/L	0.01		
20-2C	135318	Barium	0.212		mg/L	0.005		
20-2C	135318	Beryllium	0.005	U	mg/L	0.005		
20-2C	135318	Bismuth	0.020	U	mg/L	0.02		
20-2C	135318	Boron	0.100	U	mg/L	0.1		
20-2C	135318	Cadmium	0.006		mg/L	0.005		
20-2C	135318	Calcium	4.93		mg/L	0.1		
20-2C	135318	Chromium	0.012		mg/L	0.01		
20-2C	135318	Cobalt	0.049		mg/L	0.005		
20-2C	135318	Copper	0.026		mg/L	0.01		
20-2C	135318	Iron	0.250	U	mg/L	0.25		
20-2C	135318	Lanthanum	0.009		mg/L	0.005		
20-2C	135318	Lead	0.050	U	mg/L	0.05		
20-2C	135318	Lithium	0.005	U	mg/L	0.005		
20-2C	135318	Magnesium	4.86		mg/L	1		
20-2C	135318	Manganese	0.011		mg/L	0.005		
20-2C	135318	Molybdenum	0.005	U	mg/L	0.005		
20-2C	135318	Nickel	0.040		mg/L	0.005		
20-2C	135318	Palladium	0.020	U	mg/L	0.02		
20-2C	135318	Phosphorus	0.050	U	mg/L	0.05		
20-2C	135318	Potassium	4.29		mg/L	0.1		
20-2C	135318	Selenium	0.050	U	mg/L	0.05		
20-2C	135318	Silicon	9.89		mg/L	0.1		
20-2C	135318	Silver	0.010	U	mg/L	0.01		
20-2C	135318	Sodium	20.0		mg/L	1		
20-2C	135318	Strontium	0.005	U	mg/L	0.005		
20-2C	135318	Sulfur	0.200	U	mg/L	0.2		
20-2C	135318	Thallium	0.050	U	mg/L	0.05		
20-2C	135318	Thorium	0.500	U	mg/L	0.5		
20-2C	135318	Tin	0.010	U	mg/L	0.01		
20-2C	135318	Titanium	0.050	U	mg/L	0.05		
20-2C	135318	Tungsten	0.020	U	mg/L	0.02		
20-2C	135318	Uranium	114		mg/L	10		
20-2C	135318	Vanadium	0.049		mg/L	0.005		
20-2C	135318	Yttrium	0.005	U	mg/L	0.005		
20-2C	135318	Zinc	0.021		mg/L	0.005		
20-2C	135318	Zirconium	0.050	U	mg/L	0.05		

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Results: Ca, U, Si standard ICP analysis

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-3C	135319	Aluminum	0.456		mg/L	0.1		
20-3C	135319	Antimony	0.120		mg/L	0.01		
20-3C	135319	Arsenic	0.019		mg/L	0.01		
20-3C	135319	Barium	0.425		mg/L	0.005		
20-3C	135319	Beryllium	0.010		mg/L	0.005		
20-3C	135319	Bismuth	0.020	U	mg/L	0.02		
20-3C	135319	Boron	0.100	U	mg/L	0.1		
20-3C	135319	Cadmium	0.011		mg/L	0.005		
20-3C	135319	Calcium	10.1		mg/L	0.1		
20-3C	135319	Chromium	0.022		mg/L	0.01		
20-3C	135319	Cobalt	0.100		mg/L	0.005		
20-3C	135319	Copper	0.053		mg/L	0.01		
20-3C	135319	Iron	0.250	U	mg/L	0.25		
20-3C	135319	Lanthanum	0.006		mg/L	0.005		
20-3C	135319	Lead	0.050	U	mg/L	0.05		
20-3C	135319	Lithium	0.005	U	mg/L	0.005		
20-3C	135319	Magnesium	9.78		mg/L	1		
20-3C	135319	Manganese	0.029		mg/L	0.005		
20-3C	135319	Molybdenum	0.005	U	mg/L	0.005		
20-3C	135319	Nickel	0.081		mg/L	0.005		
20-3C	135319	Palladium	0.020	U	mg/L	0.02		
20-3C	135319	Phosphorus	0.050	U	mg/L	0.05		
20-3C	135319	Potassium	9.52		mg/L	0.1		
20-3C	135319	Selenium	0.050	U	mg/L	0.05		
20-3C	135319	Silicon	15.0		mg/L	0.1		
20-3C	135319	Silver	0.019		mg/L	0.01		
20-3C	135319	Sodium	33.3		mg/L	1		
20-3C	135319	Strontium	0.005	U	mg/L	0.005		
20-3C	135319	Sulfur	0.200	U	mg/L	0.2		
20-3C	135319	Thallium	0.050	U	mg/L	0.05		
20-3C	135319	Thorium	0.500	U	mg/L	0.5		
20-3C	135319	Tin	0.010	U	mg/L	0.01		
20-3C	135319	Titanium	0.050	U	mg/L	0.05		
20-3C	135319	Tungsten	0.020	U	mg/L	0.02		
20-3C	135319	Uranium	58.2		mg/L	1		
20-3C	135319	Vanadium	0.100		mg/L	0.005		
20-3C	135319	Yttrium	0.005	U	mg/L	0.005		
20-3C	135319	Zinc	0.052		mg/L	0.005		
20-3C	135319	Zirconium	0.050	U	mg/L	0.05		

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-4C	135320	Aluminum	0.547		mg/L	0.1		
20-4C	135320	Antimony	0.146		mg/L	0.01		
20-4C	135320	Arsenic	0.027		mg/L	0.01		
20-4C	135320	Barium	0.532		mg/L	0.005		
20-4C	135320	Beryllium	0.012		mg/L	0.005		
20-4C	135320	Bismuth	0.020	U	mg/L	0.02		
20-4C	135320	Boron	0.100	U	mg/L	0.1		
20-4C	135320	Cadmium	0.014		mg/L	0.005		
20-4C	135320	Calcium	12.5		mg/L	0.1		
20-4C	135320	Chromium	0.026		mg/L	0.01		
20-4C	135320	Cobalt	0.125		mg/L	0.005		
20-4C	135320	Copper	0.066		mg/L	0.01		
20-4C	135320	Iron	0.295		mg/L	0.25		
20-4C	135320	Lanthanum	0.006		mg/L	0.005		
20-4C	135320	Lead	0.050	U	mg/L	0.05		
20-4C	135320	Lithium	0.005	U	mg/L	0.005		
20-4C	135320	Magnesium	12.2		mg/L	1		
20-4C	135320	Manganese	0.038		mg/L	0.005		
20-4C	135320	Molybdenum	0.005	U	mg/L	0.005		
20-4C	135320	Nickel	0.102		mg/L	0.005		
20-4C	135320	Palladium	0.020	U	mg/L	0.02		
20-4C	135320	Phosphorus	0.050	U	mg/L	0.05		
20-4C	135320	Potassium	12.5		mg/L	0.1		
20-4C	135320	Selenium	0.050	U	mg/L	0.05		
20-4C	135320	Silicon	20.0		mg/L	0.1		
20-4C	135320	Silver	0.025		mg/L	0.01		
20-4C	135320	Sodium	44.4		mg/L	1		
20-4C	135320	Strontium	0.005	U	mg/L	0.005		
20-4C	135320	Sulfur	0.200	U	mg/L	0.2		
20-4C	135320	Thallium	0.050	U	mg/L	0.05		
20-4C	135320	Thorium	0.500	U	mg/L	0.5		
20-4C	135320	Tin	0.010	U	mg/L	0.01		
20-4C	135320	Titanium	0.050	U	mg/L	0.05		
20-4C	135320	Tungsten	0.020	U	mg/L	0.02		
20-4C	135320	Uranium	23.2		mg/L	1		
20-4C	135320	Vanadium	0.125		mg/L	0.005		
20-4C	135320	Yttrium	0.005	U	mg/L	0.005		
20-4C	135320	Zinc	0.057		mg/L	0.005		
20-4C	135320	Zirconium	0.050	U	mg/L	0.05		

Results: Div. 01 QC ICP trip

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
Prep Blank	pbw-b28c3	Aluminum	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Antimony	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Arsenic	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Barium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Beryllium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Bismuth	0.020	U	mg/L	0.02		
Prep Blank	pbw-b28c3	Boron	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Cadmium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Calcium	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Chromium	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Cobalt	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Copper	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Iron	0.025	U	mg/L	0.025		
Prep Blank	pbw-b28c3	Lanthanum	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Lead	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Lithium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Magnesium	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Manganese	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Molybdenum	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Nickel	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Palladium	0.020	U	mg/L	0.02		
Prep Blank	pbw-b28c3	Phosphorus	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Potassium	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Selenium	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Silicon	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Silver	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Sodium	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Strontium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Sulfur	0.020	U	mg/L	0.02		
Prep Blank	pbw-b28c3	Thallium	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Thorium	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Tin	0.010	U	mg/L	0.01		
Prep Blank	pbw-b28c3	Titanium	0.050	U	mg/L	0.05		
Prep Blank	pbw-b28c3	Tungsten	0.020	U	mg/L	0.02		
Prep Blank	pbw-b28c3	Uranium	0.100	U	mg/L	0.1		
Prep Blank	pbw-b28c3	Vanadium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Yttrium	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Zinc	0.005	U	mg/L	0.005		
Prep Blank	pbw-b28c3	Zirconium	0.050	U	mg/L	0.05		

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SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
20-5A	135311 Spike	Aluminum	1.97		mg/L	0.1	2	98.3%
20-5A	135311 Spike	Antimony	0.526		mg/L	0.01	0.5	105.2%
20-5A	135311 Spike	Arsenic	2.21		mg/L	0.01	2	110.6%
20-5A	135311 Spike	Barium	2.07		mg/L	0.005	2	103.3%
20-5A	135311 Spike	Beryllium	0.053		mg/L	0.005	0.05	106.3%
20-5A	135311 Spike	Bismuth	0.020	U	mg/L	0.02		
20-5A	135311 Spike	Boron	4.14		mg/L	0.1	4	103.4%
20-5A	135311 Spike	Cadmium	0.055		mg/L	0.005	0.05	109.1%
20-5A	135311 Spike	Calcium	239		mg/L	0.1	200	97.1%
20-5A	135311 Spike	Chromium	0.206		mg/L	0.01	0.2	103.0%
20-5A	135311 Spike	Cobalt	0.512		mg/L	0.005	0.5	102.4%
20-5A	135311 Spike	Copper	0.238		mg/L	0.01	0.25	95.0%
20-5A	135311 Spike	Iron	1.19		mg/L	0.25	1	118.5%
20-5A	135311 Spike	Lanthanum	0.007		mg/L	0.005		
20-5A	135311 Spike	Lead	0.502		mg/L	0.05	0.5	100.3%
20-5A	135311 Spike	Lithium	3.84		mg/L	0.005	4	96.0%
20-5A	135311 Spike	Magnesium	20.2		mg/L	0.1	20	100.8%
20-5A	135311 Spike	Manganese	0.505		mg/L	0.05	0.5	100.9%
20-5A	135311 Spike	Molybdenum	0.138		mg/L	0.005		
20-5A	135311 Spike	Nickel	0.530		mg/L	0.005	0.5	104.6%
20-5A	135311 Spike	Palladium	0.020	U	mg/L	0.02		
20-5A	135311 Spike	Phosphorus	0.050	U	mg/L	0.05		
20-5A	135311 Spike	Potassium	19.5		mg/L	0.1	20	97.6%
20-5A	135311 Spike	Selenium	2.31		mg/L	0.05	2	115.4%
20-5A	135311 Spike	Silicon	105		mg/L	0.1	40	103.0%
20-5A	135311 Spike	Silver	0.046		mg/L	0.01	0.05	92.9%
20-5A	135311 Spike	Sodium	16.4		mg/L	0.1	20	78.1%
20-5A	135311 Spike	Strontium	0.007		mg/L	0.005		
20-5A	135311 Spike	Sulfur	0.200	U	mg/L	0.2		
20-5A	135311 Spike	Thallium	2.19		mg/L	0.05	2	109.6%
20-5A	135311 Spike	Thorium	0.500	U	mg/L	0.5		
20-5A	135311 Spike	Tin	0.010	U	mg/L	0.01		
20-5A	135311 Spike	Titanium	3.94		mg/L	0.05	4	98.4%
20-5A	135311 Spike	Tungsten	0.020	U	mg/L	0.02		
20-5A	135311 Spike	Uranium	925		mg/L	10	400	101.8%
20-5A	135311 Spike	Vanadium	0.508		mg/L	0.005	0.5	101.5%
20-5A	135311 Spike	Yttrium	0.005	U	mg/L	0.005		
20-5A	135311 Spike	Zinc	0.538		mg/L	0.005	0.5	107.6%
20-5A	135311 Spike	Zirconium	0.050	U	mg/L	0.05		

Results: Div. 01 QC ICP trials

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
Lab Control	lcsw-b28e3	Aluminum	1.94		mg/L	0.1	2	97.0%
Lab Control	lcsw-b28e3	Antimony	0.501		mg/L	0.01	0.5	100.2%
Lab Control	lcsw-b28e3	Arsenic	2.03		mg/L	0.01	2	101.4%
Lab Control	lcsw-b28e3	Barium	2.05		mg/L	0.005	2	102.6%
Lab Control	lcsw-b28e3	Beryllium	0.052		mg/L	0.005	0.05	104.7%
Lab Control	lcsw-b28e3	Bismuth	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e3	Boron	4.01		mg/L	0.1	4	100.3%
Lab Control	lcsw-b28e3	Cadmium	0.051		mg/L	0.005	0.05	102.7%
Lab Control	lcsw-b28e3	Calcium	19.7		mg/L	0.1	20	98.5%
Lab Control	lcsw-b28e3	Chromium	0.203		mg/L	0.01	0.2	101.4%
Lab Control	lcsw-b28e3	Cobalt	0.489		mg/L	0.005	0.5	97.7%
Lab Control	lcsw-b28e3	Copper	0.258		mg/L	0.01	0.25	103.1%
Lab Control	lcsw-b28e3	Iron	1.01		mg/L	0.025	1	100.6%
Lab Control	lcsw-b28e3	Lanthanum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e3	Lead	0.492		mg/L	0.05	0.5	98.4%
Lab Control	lcsw-b28e3	Lithium	3.68		mg/L	0.005	4	92.0%
Lab Control	lcsw-b28e3	Magnesium	19.6		mg/L	0.1	20	97.8%
Lab Control	lcsw-b28e3	Manganese	0.515		mg/L	0.005	0.5	103.1%
Lab Control	lcsw-b28e3	Molybdenum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e3	Nickel	0.500		mg/L	0.005	0.5	100.1%
Lab Control	lcsw-b28e3	Palladium	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e3	Phosphorus	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e3	Potassium	16.8		mg/L	0.1	20	83.9%
Lab Control	lcsw-b28e3	Selenium	2.03		mg/L	0.05	2	101.4%
Lab Control	lcsw-b28e3	Silicon	4.01		mg/L	0.1	4	100.3%
Lab Control	lcsw-b28e3	Silver	0.050		mg/L	0.01	0.05	99.2%
Lab Control	lcsw-b28e3	Sodium	16.9		mg/L	0.1	20	84.5%
Lab Control	lcsw-b28e3	Strontium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e3	Sulfur	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e3	Thallium	2.09		mg/L	0.05	2	104.7%
Lab Control	lcsw-b28e3	Thorium	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e3	Tin	0.010	U	mg/L	0.01		
Lab Control	lcsw-b28e3	Titanium	3.97		mg/L	0.05	4	99.3%
Lab Control	lcsw-b28e3	Tungsten	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e3	Uranium	4.10		mg/L	0.1	4	102.5%
Lab Control	lcsw-b28e3	Vanadium	0.493		mg/L	0.005	0.5	98.6%
Lab Control	lcsw-b28e3	Yttrium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e3	Zinc	0.504		mg/L	0.005	0.5	100.8%
Lab Control	lcsw-b28e3	Zirconium	0.050	U	mg/L	0.05		

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
Lab Control	lcsw-b28e4	Aluminum	1.95		mg/L	0.1	2	97.6%
Lab Control	lcsw-b28e4	Antimony	0.497		mg/L	0.01	0.5	99.4%
Lab Control	lcsw-b28e4	Arsenic	2.04		mg/L	0.01	2	101.9%
Lab Control	lcsw-b28e4	Barium	2.06		mg/L	0.005	2	102.9%
Lab Control	lcsw-b28e4	Beryllium	0.052		mg/L	0.005	0.05	104.8%
Lab Control	lcsw-b28e4	Bismuth	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e4	Boron	4.12		mg/L	0.1	4	103.1%
Lab Control	lcsw-b28e4	Cadmium	0.052		mg/L	0.005	0.05	103.0%
Lab Control	lcsw-b28e4	Calcium	20.5		mg/L	0.1	20	102.4%
Lab Control	lcsw-b28e4	Chromium	0.203		mg/L	0.01	0.2	101.7%
Lab Control	lcsw-b28e4	Cobalt	0.492		mg/L	0.005	0.5	98.3%
Lab Control	lcsw-b28e4	Copper	0.258		mg/L	0.01	0.25	103.1%
Lab Control	lcsw-b28e4	Iron	1.00		mg/L	0.025	1	100.3%
Lab Control	lcsw-b28e4	Lanthanum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e4	Lead	0.494		mg/L	0.05	0.5	98.8%
Lab Control	lcsw-b28e4	Lithium	3.77		mg/L	0.005	4	94.4%
Lab Control	lcsw-b28e4	Magnesium	20.3		mg/L	0.1	20	101.5%
Lab Control	lcsw-b28e4	Manganese	0.518		mg/L	0.005	0.5	103.6%
Lab Control	lcsw-b28e4	Molybdenum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e4	Nickel	0.504		mg/L	0.005	0.5	100.7%
Lab Control	lcsw-b28e4	Palladium	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e4	Phosphorus	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e4	Potassium	17.6		mg/L	0.1	20	87.8%
Lab Control	lcsw-b28e4	Selenium	2.05		mg/L	0.05	2	102.7%
Lab Control	lcsw-b28e4	Silicon	4.10		mg/L	0.1	4	102.4%
Lab Control	lcsw-b28e4	Silver	0.050		mg/L	0.01	0.05	100.0%
Lab Control	lcsw-b28e4	Sodium	17.5		mg/L	0.1	20	87.7%
Lab Control	lcsw-b28e4	Strontium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e4	Sulfur	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e4	Thallium	2.13		mg/L	0.05	2	106.4%
Lab Control	lcsw-b28e4	Thorium	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e4	Tin	0.010	U	mg/L	0.01		
Lab Control	lcsw-b28e4	Titanium	4.00		mg/L	0.05	4	100.0%
Lab Control	lcsw-b28e4	Tungsten	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e4	Uranium	3.92		mg/L	0.1	4	98.1%
Lab Control	lcsw-b28e4	Vanadium	0.496		mg/L	0.005	0.5	99.2%
Lab Control	lcsw-b28e4	Yttrium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e4	Zinc	0.509		mg/L	0.005	0.5	101.8%
Lab Control	lcsw-b28e4	Zirconium	0.050	U	mg/L	0.05		

Results: Div. 01 QC ICP analysis trials

SAMPLE ID	SYSTEM ID	ELEMENT	RESULTS	QUALIFIER	UNITS	REPORTING LIMIT	TRUE VALUE	RECOVERY
Lab Control	lcsw-b28e5	Aluminum	1.99		mg/L	0.1	2	99.3%
Lab Control	lcsw-b28e5	Antimony	0.504		mg/L	0.01	0.5	100.9%
Lab Control	lcsw-b28e5	Arsenic	2.05		mg/L	0.01	2	102.3%
Lab Control	lcsw-b28e5	Barium	2.09		mg/L	0.005	2	104.7%
Lab Control	lcsw-b28e5	Beryllium	0.052		mg/L	0.005	0.05	104.0%
Lab Control	lcsw-b28e5	Bismuth	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e5	Boron	4.07		mg/L	0.1	4	101.7%
Lab Control	lcsw-b28e5	Cadmium	0.051		mg/L	0.005	0.05	102.0%
Lab Control	lcsw-b28e5	Calcium	19.9		mg/L	0.1	20	99.4%
Lab Control	lcsw-b28e5	Chromium	0.202		mg/L	0.01	0.2	101.0%
Lab Control	lcsw-b28e5	Cobalt	0.487		mg/L	0.005	0.5	97.4%
Lab Control	lcsw-b28e5	Copper	0.262		mg/L	0.01	0.25	104.7%
Lab Control	lcsw-b28e5	Iron	0.987		mg/L	0.025	1	98.7%
Lab Control	lcsw-b28e5	Lanthanum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e5	Lead	0.490		mg/L	0.05	0.5	98.0%
Lab Control	lcsw-b28e5	Lithium	3.80		mg/L	0.005	4	95.0%
Lab Control	lcsw-b28e5	Magnesium	20.0		mg/L	0.1	20	99.8%
Lab Control	lcsw-b28e5	Manganese	0.515		mg/L	0.005	0.5	103.1%
Lab Control	lcsw-b28e5	Molybdenum	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e5	Nickel	0.498		mg/L	0.005	0.5	99.7%
Lab Control	lcsw-b28e5	Palladium	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e5	Phosphorus	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e5	Potassium	17.7		mg/L	0.1	20	88.4%
Lab Control	lcsw-b28e5	Selenium	2.06		mg/L	0.05	2	103.0%
Lab Control	lcsw-b28e5	Silicon	4.04		mg/L	0.1	4	101.0%
Lab Control	lcsw-b28e5	Silver	0.050		mg/L	0.01	0.05	100.2%
Lab Control	lcsw-b28e5	Sodium	17.2		mg/L	0.1	20	85.9%
Lab Control	lcsw-b28e5	Strontium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e5	Sulfur	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e5	Thallium	2.13		mg/L	0.05	2	106.4%
Lab Control	lcsw-b28e5	Thorium	0.050	U	mg/L	0.05		
Lab Control	lcsw-b28e5	Tin	0.010	U	mg/L	0.01		
Lab Control	lcsw-b28e5	Titanium	4.06		mg/L	0.05	4	101.6%
Lab Control	lcsw-b28e5	Tungsten	0.020	U	mg/L	0.02		
Lab Control	lcsw-b28e5	Uranium	4.21		mg/L	0.1	4	105.3%
Lab Control	lcsw-b28e5	Vanadium	0.493		mg/L	0.005	0.5	98.6%
Lab Control	lcsw-b28e5	Yttrium	0.005	U	mg/L	0.005		
Lab Control	lcsw-b28e5	Zinc	0.501		mg/L	0.005	0.5	100.3%
Lab Control	lcsw-b28e5	Zirconium	0.050	U	mg/L	0.05		

2/28/00

Summary: Uranophane No. 5+6 Cation Analysis  
by ICP

## ICP Analysis

Element	Trial Results (ppm)			Results Average ppm	Reporting Limit (ppm)
	20-5A	20-5B	20-5C		
Ca	44.4	43	82.5	42.5	0.100
U	518	504	974	499.0	10.000
Si	63.6	61.6	120	61.3	0.100
Na	0.753	0.781	1.79	0.8	0.100
Al				0.0	0.100
Sb				0.0	0.010
Ba				0.0	0.005
Co			0.013	0.0	0.005
Cu		0.011		0.0	0.010
La				0.0	0.005
Mg				0.0	0.100
Mn				0.0	0.005
Mo	0.136	0.134	0.263	0.1	0.005
Ni	0.007	0.011	0.021	0.0	0.005
K				0.0	0.100
V				0.0	0.005
Zn		0.455		0.1	0.005

Element	Trial Results (ppm)			Results Average ppm	Reporting Limit (ppm)
	20-6A	20-6B	20-6C		
Ca	42.1	42.8	83.3	42.1	0.100
U	502	508	991	500.3	10.000
Si	61.3	61.9	121	61.1	0.100
Na	0.814	1.03	1.62	0.9	0.100
Al	0.104			0.0	0.100
Sb				0.0	0.010
Ba				0.0	0.005
Co	0.008		0.009	0.0	0.005
Cu				0.0	0.010
La				0.0	0.005
Mg				0.0	0.100
Mn				0.0	0.005
Mo	0.136	0.143	0.261	0.1	0.005
Ni	0.01	0.015	0.016	0.0	0.005
K				0.0	0.100
V				0.0	0.005
Zn				0.0	0.005

Summary: Ca, U, Si standard analysis by ICP

## ICP Analysis Results

Sample I.D.	Element	Results (ppm)	Reporting Limit (ppm)	True Value (ppm)	Recovery %
20-1c	Ca	2.490	0.100	2.5	99.6
20-1c	U	173.000	10.000	179	96.6
20-1c	Si	5.040	0.100	5	100.8
20-1c	Na	9.700	1.000	2.5	388.0
20-1c	Al	0.183	0.100		
20-1c	Sb	0.029	0.010		
20-1c	Ba	0.107	0.005		
20-1c	Co	0.025	0.005		
20-1c	Cu	0.012	0.010		
20-1c	La	0.084	0.005		
20-1c	Mg	2.350	1.000		
20-1c	Mn	U	0.005		
20-1c	Mo	0.013	0.005		
20-1c	Ni	0.019	0.005		
20-1c	K	1.920	0.100		
20-1c	Va	0.025	0.005		
20-1c	Zn	0.016	0.005		

Sample I.D.	Element	Results (ppm)	Reporting Limit (ppm)	True Value (ppm)	Recovery %
20-2c	Ca	4.930	0.100	5.0	98.6
20-2c	U	114.000	10.000	119.0	95.8
20-2c	Si	9.890	0.100	10.0	98.9
20-2c	Na	20.000	1.000	5.0	400.0
20-2c	Al	0.275	0.100		
20-2c	Sb	0.047	0.010		
20-2c	Ba	0.212	0.005		
20-2c	Co	0.049	0.005		
20-2c	Cu	0.026	0.010		
20-2c	La	0.009	0.005		
20-2c	Mg	4.860	1.000		
20-2c	Mn	0.011	0.005		
20-2c	Mo	U	0.005		
20-2c	Ni	0.040	0.005		
20-2c	K	4.290	0.100		
20-2c	Va	0.049	0.005		
20-2c	Zn	0.021	0.005		

Summary: Ca, U, Si standard analysis by ICP

Sample I.D.	Element	Results (ppm)	Reporting Limit (ppm)	True Value (ppm)	Recovery %
20-3c	Ca	10.100	0.100	10.0	101.0
20-3c	U	58.200	10.000	60.0	97.0
20-3c	Si	15.000	0.100	15.0	100.0
20-3c	Na	33.300	1.000	10.0	333.0
20-3c	Al	0.456	0.100		
20-3c	Sb	0.120	0.010		
20-3c	Ba	0.425	0.005		
20-3c	Co	0.100	0.005		
20-3c	Cu	0.053	0.010		
20-3c	La	0.006	0.005		
20-3c	Mg	9.780	1.000		
20-3c	Mn	0.029	0.005		
20-3c	Mo	0.005	0.005		
20-3c	Ni	0.081	0.005		
20-3c	K	9.520	0.100		
20-3c	Va	0.100	0.005		
20-3c	Zn	0.052	0.005		

Sample I.D.	Element	Results (ppm)	Reporting Limit (ppm)	True Value (ppm)	Recovery %
20-4c	Ca	12.5	0.100	12.5	100.0
20-4c	U	23.2	10.000	23.8	97.5
20-4c	Si	20	0.100	20.0	100.0
20-4c	Na	44.4	1.000	15.0	296.0
20-4c	Al	0.547	0.100		
20-4c	Sb	0.146	0.010		
20-4c	Ba	0.532	0.005		
20-4c	Co	0.125	0.005		
20-4c	Cu	0.066	0.010		
20-4c	La	0.006	0.005		
20-4c	Mg	12.2	1.000		
20-4c	Mn	0.038	0.005		
20-4c	Mo	0.005	0.005		
20-4c	Ni	0.102	0.005		
20-4c	K	12.5	0.100		
20-4c	Va	0.125	0.005		
20-4c	Zn	0.057	0.005		

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Preparation of solutions for uranophane solubility studies

#### Preparation of solutions for uranophane solubility studies

Written by: J. D. Prikryl  
Date: 02/25/00

Objective: Prepare a series of solutions containing U, Ca, and SiO<sub>2</sub> for use in the uranophane solubility experiments. The solutions will have a Ca content of 10<sup>-2</sup> M and a SiO<sub>2</sub> content of 10<sup>-3</sup> M. U content will vary from 10<sup>-4</sup> to 10<sup>-7</sup> M.

Method: Addition of chemical reagents containing Ca and U to an approximate 10<sup>-3</sup> M SiO<sub>2</sub> solution.

#### Materials and Equipment:

- previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution
- CaCl<sub>2</sub>·2H<sub>2</sub>O; F.W. 147.02; (lot no. 6A15H) Analytical Scientific
- UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O; F.W. 502.13; (lot no. 8640 KCAP) Analytical Scientific
- CaCO<sub>3</sub>; F.W. 100.09; (lot no. 986-396) Analytical Scientific
- Polypropylene bottles (500 ml) (4)
- Polypropylene bottles (15 ml) (8)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Mettler analytical balance (AE240)
- 100 ml volumetric flask (1)
- 250 ml volumetric flasks (4)
- weighing paper and boats
- Fisher Versabath Model 236

#### Procedure

- Transfer about 50 ml of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution to a 100 ml volumetric flask. Add 0.05021 g of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to the volumetric flask and make up to the mark with the 10<sup>-3</sup> M SiO<sub>2</sub> solution. Label the volumetric flask as 10<sup>-3</sup> M U.
- Label 4 250 ml volumetric flasks as follows:  
  
10<sup>-4</sup> M U  
10<sup>-5</sup> M U  
10<sup>-6</sup> M U  
10<sup>-7</sup> M U
- Transfer about 100 ml of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution into each 250 ml volumetric flask.
- Carefully add the following quantities of reagents and 10<sup>-3</sup> M U solution to the specified flask. Use weighing paper and weigh out reagents as accurately as possible. For the 10<sup>-3</sup> M U solution use plastic weighing boats and rinse boats using the 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Flask Label	CaCl <sub>2</sub> ·2H <sub>2</sub> O	CaCO <sub>3</sub>	10 <sup>-3</sup> M U solution
10 <sup>-4</sup> M U	0.36417 g	0.00230 g	25 g
10 <sup>-5</sup> M U	0.36712 g	0.00029 g	2.5 g
10 <sup>-6</sup> M U	0.36742 g	0.00009 g	0.25 g
10 <sup>-7</sup> M U	0.36745 g	0.00006 g	0.025 g

Procedure cont'd.

5. After adding reagents and  $10^{-3}$  M U solution, fill flasks up to mark using the previously prepared  $10^{-3}$  M  $\text{SiO}_2$  solution.
6. Insure that reagents are completely dissolved and then transfer the solutions to four 500 ml polypropylene bottles labeled as follows:

$10^{-4}$  M U matrix solution  
 $10^{-5}$  M U matrix solution  
 $10^{-6}$  M U matrix solution  
 $10^{-7}$  M U matrix solution

7. Place a teflon stir bar in each bottle and measure and record pH.
8. Place bottles in shaker water bath set at  $25^\circ\text{C}$ . Allow solutions to equilibrate with air by loosely capping bottles.
9. Measure and record pH periodically (e.g., daily) to determine when solutions reach equilibrium with air.
10. When equilibrium is reached record the weight of each U matrix solution bottle.

Bottle	Wt (g)
$10^{-4}$ M U matrix solution	
$10^{-5}$ M U matrix solution	
$10^{-6}$ M U matrix solution	
$10^{-7}$ M U matrix solution	

11. Take two 5ml sample aliquots from each U matrix solution and place in 15 ml polypropylene bottles for chemical analysis. Use an Oxford pipettor and 5 ml plastic tips to take samples. Preserve the samples by adding  $10\ \mu\text{L}$  of 6 M  $\text{HNO}_3$  to each 15 ml sample bottle. Chemical analysis will show the initial Ca,  $\text{SiO}_2$ , and U contents of the solutions before addition of uranophane. Label the samples as shown below.

Label	U matrix solution sampled
UM-4A	$10^{-4}$ M U matrix solution
UM-4B	$10^{-4}$ M U matrix solution
UM-5A	$10^{-5}$ M U matrix solution
UM-5B	$10^{-5}$ M U matrix solution
UM-6A	$10^{-6}$ M U matrix solution
UM-6B	$10^{-6}$ M U matrix solution
UM-7A	$10^{-7}$ M U matrix solution
UM-7B	$10^{-7}$ M U matrix solution

12. After sampling record the weight of each U matrix solution bottle.

Bottle	Wt (g)
$10^{-4}$ M U matrix solution	
$10^{-5}$ M U matrix solution	
$10^{-6}$ M U matrix solution	
$10^{-7}$ M U matrix solution	

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3/31/00

3/20/00 Data: Uranophane Solubility  
 re  
 added 0.0508 g uranyl nitrate to 100ml  $10^{-3}$  M silica solution.

3/31/00 Reagents added in  $10^{-3}$  M  $\text{SiO}_2$  matrix to make up  
 re  
 250 ml of solution:

Flash Label	$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	$\text{CaCO}_3$	$10^{-3}$ M U soln.
$10^{-4}$ M U	0.36415 g	0.00235 g	24.9948 g
$10^{-5}$ M U	0.36728 g	0.00033 g	2.4986 g
$10^{-6}$ M U	0.36740 g	-0- g	0.2462 g
$10^{-7}$ M U	0.36748 g	-0- g	0.0203 g

4/4/00 pH measurement of above solutions (temp. compensated):  
 re  
 pH meter calib. = 99.6 slope

Soln. Label	pH
$10^{-4}$ M U	4.56
$10^{-5}$ M U	5.36
$10^{-6}$ M U	5.86
$10^{-7}$ M U	5.96

Solutions placed in shaker bath set at  $25^\circ\text{C}$ .

4/6/00 pH measurement of solutions after 2 days of equilibrating:  
 re

Soln. Label	pH
$10^{-4}$ M U	4.57
$10^{-5}$ M U	5.36
$10^{-6}$ M U	6.00
$10^{-7}$ M U	5.98

Added  $\text{CaCO}_3$  to following samples to adjust pH to  $\sim 6.0$ :

Sample Label	Wt. $\text{CaCO}_3$ (grams)
$10^{-4}$ M U	0.00238
$10^{-5}$ M U	0.00025



Uranophane Solubility4/11/00  
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pH measurement of solutions after added adjustment.

Soln. Label	pH
$10^{-4}$ M U	6.03
$10^{-5}$ M U	5.93
$10^{-6}$ M U	6.01
$10^{-7}$ M U	5.91

4/12/00  
re

pH measurement of solns. after extra day of equilibration.

Soln. Label	pH
$10^{-4}$ M U	5.95
$10^{-5}$ M U	5.94
$10^{-6}$ M U	5.97
$10^{-7}$ M U	5.92

4/14/00  
re

Prepared samples of U-matrix solutions for analysis by adding 5 ml solution from each labeled U-matrix soln. bottle using an Oxford 5 ml pipettor. Tared the empty 15 ml PP bottles and added sample soln.

Sample Label	Wt. of added soln.	pH re 4/14/00
UM-4A	4.8903	
UM-4B	4.9113	
UM-5A	4.9143	
UM-5B	4.8933	
UM-6A	4.9030	
UM-6B	4.9274	
UM-7A	4.9237	
UM-7B	4.8871	

Labeled the 15 ml PP sample bottles as appropriate, attached radioactive labels, and sent samples to DIV 01 for ICP analysis.

Uranophane Solubility

## Uranophane solubility experiment

re  
4/15/00

Written by: J. D. Prikryl  
Date: 03/23/00

Objective: Determine the solubility of synthesized uranophane.

Method: Monitor the reaction of synthesized uranophane with solutions containing U, Ca, and  $\text{SiO}_2$ .

Materials and Equipment:

- Synthesized uranophane
- Previously prepared U matrix solutions with U concentrations of  $10^{-4}$ ,  $10^{-5}$ ,  $10^{-6}$ , and  $10^{-7}$  M
- Polypropylene bottles (500 ml) (8)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Fisher Versabath Model 236
- Mettler analytical balance (AE240 and PM4600)
- Pipets and pipet tips

## Procedure

1. Label 8 500 ml polypropylene bottles as follows and record weights.

Label	Weight (g)
URS-4A	54.8766
URS-4B	54.5966
URS-5A	54.6724
URS-5B	54.7204
URS-6A	53.9248
URS-6B	54.1179
URS-7A	54.7974
URS-7B	54.7700

2. To each sample bottle add  $0.5 \pm 0.0010$  g of synthesized uranophane. Record the weight of uranophane added to each bottle.

Bottle	Wt of uranophane (g)
URS-4A	0.4997
URS-4B	0.4999
URS-5A	0.5004
URS-5B	0.4991
URS-6A	0.5008
URS-6B	0.5005
URS-7A	0.5003
URS-7B	0.5007

Uranophane Solubility

3. To each bottle add 100±0.05 g of the specified U matrix solution shown in the table below. Record the weight of matrix solution added.

4/12/00 RL

Bottle	U matrix solution	Wt of U matrix solution (g)
URS-4A	10 <sup>-4</sup> M U matrix solution	99.96
URS-4B	10 <sup>-4</sup> M U matrix solution	99.99
URS-5A	10 <sup>-5</sup> M U matrix solution	100.01
URS-5B	10 <sup>-5</sup> M U matrix solution	100.03
URS-6A	10 <sup>-6</sup> M U matrix solution	99.97
URS-6B	10 <sup>-6</sup> M U matrix solution	100.02
URS-7A	10 <sup>-7</sup> M U matrix solution	99.96
URS-7B	10 <sup>-7</sup> M U matrix solution	99.98

4. Record the initial sample weights (bottle + synthesized uranophane + U matrix solution).

4/12/00 RL

Sample	Initial wt (g)
URS-4A	155.24
URS-4B	155.06
URS-5A	155.17
URS-5B	155.23
URS-6A	154.40
URS-6B	154.62
URS-7A	155.27
URS-7B	155.25

5. Cap the bottles and place in a shaking water bath set at 25 °C.
6. At 1 week intervals record the weight of each bottle and then remove 5 ml aliquots from each bottle using a syringe and syringe filter. Transfer the samples to plastic sample cups containing stir bars and measure and record pHs. Transfer the samples from the sample cups to 15 ml polypropylene bottles for analysis of Ca, SiO<sub>2</sub>, and U. Record the weights of the bottles after sampling. Below are tables into which data can be recorded. Preserve samples by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle.

URS-4A

RL

Date / Time	Wt of bottle before sampling (g)	Wt of bottle after sampling (g)	Wt of solution removed (g)	pH of solution removed	Sample Label
4/19/00-1040	155.33	150.05	5.28	4.78	URS-4A-1
4/26/00-1350	150.06	145.03	5.03	4.93	URS-4A-2
5/3/00-1010	145.03	139.98	5.05	—	URS-4A-3
5/10/00-1015	139.93	134.90	5.03	4.77	URS-4A-4
5/18/00-1350	134.88	129.88	5.00	4.76	URS-4A-5
5/25/00-1000	129.86	124.66	5.20	4.92	URS-4A-6
6/10/00-0945	124.64	119.61	5.03	4.81	URS-4A-7
					URS-4A-8

Uranophane Solubility

URS-4B

RL

Date / Time	Wt of bottle before sampling (g)	Wt of bottle after sampling (g)	Wt of solution removed (g)	pH of solution removed	Sample Label
4/19/00-1045	155.08	150.17	4.91	4.78	URS-4B-1
4/26/00-1350	150.17	145.11	5.06	4.82	URS-4B-2
5/3/00-1015	145.11	140.07	5.04	—	URS-4B-3
5/10/00-1025	140.07	134.99	5.08	4.77	URS-4B-4
5/18/00-1400	134.99	129.95	5.04	4.74	URS-4B-5
5/25/00-1010	129.95	124.91	5.04	4.77	URS-4B-6
6/10/00-0950	124.90	119.90	5.00	4.80	URS-4B-7
					URS-4B-8

URS-5A

RL

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
4/19/00-1050	155.16	150.62	4.54	5.25	URS-5A-1
4/26/00-1355	150.62	145.57	5.05	5.25	URS-5A-2
5/3/00-1020	145.61	140.60	5.01	—	URS-5A-3
5/10/00-1030	140.56	135.52	5.04	6.02	URS-5A-4
5/18/00-1405	135.50	130.43	5.07	6.27	URS-5A-5
5/25/00-1015	130.43	125.42	5.01	6.50	URS-5A-6
6/10/00-0955	125.41	120.40	5.01	6.28	URS-5A-7
					URS-5A-8

URS-5B

RL

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
4/19/00-1100	155.21	150.20	5.01	5.20	URS-5B-1
4/26/00-1400	150.20	145.24	5.06	5.27	URS-5B-2
5/3/00-1025	145.25	140.19	5.06	—	URS-5B-3
5/10/00-1030	140.18	135.15	5.03	6.11	URS-5B-4
5/18/00-1410	135.15	130.11	5.04	6.26	URS-5B-5
5/25/00-1000	130.12	125.12	5.00	6.39	URS-5B-6
6/10/00-1000	125.12	120.14	4.98	6.48	URS-5B-7
					URS-5B-8

4/20/00 RL Prepared 6 M HNO<sub>3</sub> soln. by measuring 375 mL of HNO<sub>3</sub> concentrate (lot # 993617 - Fisher Chemical) using a 500 mL volumetric flask and adding to a 1-L vol. flask. Added nanopure H<sub>2</sub>O to mark. Transferred to 1-L PP bottle.

# Uranophane Solubility

URS-6A

pc 4/15/00

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
pc 4/19/00-1300	154.37	149.45	4.92	6.35	URS-6A-1
pc 4/16/00-1405	149.44	144.40	5.04	6.32	URS-6A-2
pc 5/2/00-1023	144.41	139.37	5.04	—	URS-6A-3
pc 5/10/00-1035	139.35	134.31	5.04	6.35	URS-6A-4
pc 5/18/00-1415	134.29	129.26	5.03	6.37	URS-6A-5
pc 5/25/00-1020	129.25	124.18	5.07	6.35	URS-6A-6
pc 6/01/00-1007	124.16	119.16	5.00	6.44	URS-6A-7
					URS-6A-8

URS-6B

pc 4/15/00

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
pc 4/19/00-1305	154.61	149.67	4.94	6.33	URS-6B-1
pc 4/16/00-1410	149.67	144.64	5.03	6.35	URS-6B-2
pc 5/2/00-1030	144.66	139.64	5.02	—	URS-6B-3
pc 5/10/00-1040	139.63	134.59	5.04	6.34	URS-6B-4
pc 5/18/00-1415	134.58	129.55	5.03	6.57	URS-6B-5
pc 5/25/00-1025	129.56	124.52	5.04	6.42	URS-6B-6
pc 6/01/00-1005	124.51	119.50	5.01	6.41	URS-6B-7
					URS-6B-8

URS-7A

pc 4/15/00

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
pc 4/19/00-1310	155.25	150.31	4.94	6.40	URS-7A-1
pc 4/16/00-1420	150.31	145.26	5.05	6.46	URS-7A-2
pc 5/2/00-1023	145.26	140.24	5.02	—	URS-7A-3
pc 5/10/00-1040	140.24	135.21	5.03	6.36	URS-7A-4
pc 5/18/00-1420	135.20	130.16	5.04	6.29	URS-7A-5
pc 5/25/00-1030	130.17	125.15	5.02	6.42	URS-7A-6
pc 6/01/00-1010	125.14	120.12	5.02	6.40	URS-7A-7
					URS-7A-8

5/25/00

URS-7B

pc 4/15/00

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
pc 4/19/00-1315	155.25	150.27	4.98	6.41	URS-7B-1
pc 4/19/00-1425	150.28	145.25	5.03	6.47	URS-7B-2
pc 5/2/00-1040	145.25	140.26	4.99	—	URS-7B-3
pc 5/10/00-1045	140.26	135.24	5.02	6.41	URS-7B-4
pc 5/18/00-1430	135.22	130.21	5.01	6.32	URS-7B-5
pc 5/25/00-1030	130.23	125.20	5.03	6.41	URS-7B-6
pc 6/01/00-1015	125.19	120.16	5.03	6.50	URS-7B-7
					URS-7B-8

6/01/00  
pc

Preparation of standards for uranophane solubility experiment:

Reagents:

Uranium standard 1,000 ppm U

Mfg. SPEX Certiprep

Lot No. 6-239-U

Exp. date 8/31/00

Silicon 1,000 ppm standard

Mfg. SPEX Certiprep

Lot No. 6-271-SI

Exp. date 5/15/01

Calcium 1,000 ppm standard

Mfg. <sup>6/11/00</sup> ~~cert~~ SPEX Certiprep <sup>pc 6/11/00</sup> Fisher Chemical

Lot No. 986825-24

Exp. date 10/2000

Sodium 1,000 ppm standard

Mfg. SPEX Certiprep

Lot No. CLI-141NA

Exp. date 5/15/01

Pipetted required volume of 1,000 ppm (1,000 µg/mL) using volumetric pipettes into 100 mL volumetric flasks, then filling to mark with uranophane D.I. water. Contents of flasks were labeled as "Std. 1, 2, ..." etc. 5 mL aliquots were removed from std. flasks and placed in individual 15 mL PP bottles using a manual expending 5 mL pipette.

6/01/00  
RL

## Data Table - Uranophane Experiment Standard Preparation:

Flask No.	Element	(ppm) Expected Concentration	Vol. of Reference Standard (mL)	Flask Volume (mL)
1	U	800	80	100
1	Si	20	2	100
1	Ca	100	10	100
1	Na	-0-	-0-	100
2	U	10	1	100
2	Si	100	10	100
2	Ca	500	50	100
2	Na	50	5	100
3	U	500	50	100
3	Si	50	5	100
3	Ca	400	40	100
3	Na	10	1	100
4	U	100	10	100
4	Si	10	1	100
4	Ca	600	60	100
4	Na	30	3	100
5	U	50	5	100
5	Si	70	7	100
5	Ca	450	45	100
5	Na	5	0.5	100
6	U	20	2	100
6	Si	30	3	100
6	Ca	430	43	100
6	Na	20	2	100
7a	U	10	1	100
7b	Si	50	5	100
7	U	1	10 mL from 7a	100
7	Si	5	10 mL from 7b	100
7	Ca	50	5	100
7	Na	-0-	-0-	100
8a	U	50	re 5	100

6/1/00

6/01/00  
RL

## Data table - cont'd from pg. 96

Flask No.	Element	Expected Conc (ppm)	Vol. of Ref. Std. (mL)	Flask Vol. (mL)
8	U	0.5	1 mL from 8a	100
8	Si	20	2	100
8	Ca	re 100 50.0	re 10 5.0	100
8	Na	6/1/00 -0-	6/1/00 -0-	100

Labeled the 5 mL<sup>25</sup> check standards placed in 15 mL PP bottles as follows:

Bottle I.D.	5 mL Aliquots from Flask No.
URS-2	re 2
URS-3	6/1/00 3
URS-4	4
URS-5	5
URS-6	6
URS-7	7
URS-8	8

Did not prepare std. sample from flask No. 1 because uranium concentration as prepared is higher than needed.

6/01/00  
RL

Sent prepared standards and uraniumophane solubility samples to Div. 01 for ICP analysis of Si, Ca, + Na; <sup>re 6/1/00</sup> U analysis by ICP-MS.

RL

6/1/00

6/01/00  
me

Uranophane solubility experiment samples and check  
standards "Laboratory Work Order" as received by Div. 1:

Southwest Research Institute

Project: 20.01402.561  
Case #: CNWRA  
SDG: 144397  
Client: DIV 20

## Laboratory Work Order

Work Order Number: 18001

VTSR: 06/01/00

Time: 15:30:00

Revision: 1

Manager: DAMMANN, MIKE  
Logged in by: JMORIN  
Creation Date: 06/01/00

This Work Order was Revised Jun 1 2000 5:19PM

## Notes

AMPLES RECEIVED AT ROOM TEMP.

C NOTE: AS PER METHOD AND SPQP.

PARAMETERS: ICP AND ICP/MS ANALYSIS AS PER COC AND P.M.

NOTE: SEE ATTACHED LETTER FOR ANALYTES OF INTEREST.

NOTE: FOR FURTHER INFORMATION SEE ATTACHED, OR CONTACT PROJECT MANAGER.

NOTE: PROJECT IS NUCLEAR SAFETY RELATED, 10CFR50, PART 21, APPENDIX B.

NOTE: CONTACT JOANN BOYD @ 2169 OR PAGER 351-1617 FOR INSTITUTE QA CONTACT BEFORE STARTING ANY WORK ON THIS PROJECT.

NOTE: HAVE SPQP (LATEST REVISION) ON THE BENCH AT TIME OF PREP OR ANALYSIS OF SAMPLES.

NOTE: ALL PERSONNEL MUST BE QA NUCLEAR CERTIFIED.

NOTE: OUTSIDE OF CONTAINERS WAS SCREENED FOR RADIOACTIVITY, SAMPLES BELOW BACKGROUND LEVEL.

REV#1: CHANGED P.M. TO MIKE DAMMANN. MJM 06/01/2000

System ID	#	Customer ID	CED	Matrix	Required Analyzes
144397	1	UM-4A		LIQUID	ICP, ICP_MS
144398	1	UM-4B		LIQUID	ICP, ICP_MS
144399	1	UM-5A		LIQUID	ICP, ICP_MS
144400	1	UM-5B		LIQUID	ICP, ICP_MS
144401	1	UM-6A		LIQUID	ICP, ICP_MS
144402	1	UM-6B		LIQUID	ICP, ICP_MS
144403	1	UM-7A		LIQUID	ICP, ICP_MS
144404	1	UM-7B		LIQUID	ICP, ICP_MS
144405	1	URS-2		LIQUID	ICP, ICP_MS
144406	1	URS-3		LIQUID	ICP, ICP_MS
144407	1	URS-4		LIQUID	ICP, ICP_MS
144408	1	URS-4A1		LIQUID	ICP, ICP_MS
144409	1	URS-4A3		LIQUID	ICP, ICP_MS
144410	1	URS-4A5		LIQUID	ICP, ICP_MS
144411	1	URS-4A7		LIQUID	ICP, ICP_MS
144412	1	URS-4B1		LIQUID	ICP, ICP_MS
144413	1	URS-4B3		LIQUID	ICP, ICP_MS
144414	1	URS-4B5		LIQUID	ICP, ICP_MS
144415	1	URS-4B7		LIQUID	ICP, ICP_MS
144416	1	URS-5		LIQUID	ICP, ICP_MS
144417	1	URS-5A1		LIQUID	ICP, ICP_MS
144418	1	URS-5A3		LIQUID	ICP, ICP_MS
144419	1	URS-5A5		LIQUID	ICP, ICP_MS
144420	1	URS-5A7		LIQUID	ICP, ICP_MS
144421	1	URS-5B1		LIQUID	ICP, ICP_MS
144422	1	URS-5B3		LIQUID	ICP, ICP_MS
144423	1	URS-5B5		LIQUID	ICP, ICP_MS

Data Must be to QA: 06/12/00  
Absolute Latest Date: 06/14/00Turnaround is 14 Days from VTSR  
Page 1 of 2Page Printed: 1 Jun 2000 17:19:38  
ver (05/17/2000)6/01/00  
me

Contd.

Southwest Research Institute

Project: 20.01402.561  
Case #: CNWRA  
SDG: 144397  
Client: DIV 20

## Laboratory Work Order

Work Order Number: 18001

VTSR: 06/01/00

Time: 15:30:00

Revision: 1

Manager: DAMMANN, MIKE  
Logged in by: JMORIN  
Creation Date: 06/01/00

This Work Order was Revised Jun 1 2000 5:19PM

System ID	#	Customer ID	CED	Matrix	Required Analyzes
144424	1	URS-5B7		LIQUID	ICP, ICP_MS
144425	1	URS-6		LIQUID	ICP, ICP_MS
144426	1	URS-6A1		LIQUID	ICP, ICP_MS
144427	1	URS-6A3		LIQUID	ICP, ICP_MS
144428	1	URS-6A5		LIQUID	ICP, ICP_MS
144429	1	URS-6A7		LIQUID	ICP, ICP_MS
144430	1	URS-6B1		LIQUID	ICP, ICP_MS
144431	1	URS-6B3		LIQUID	ICP, ICP_MS
144432	1	URS-6B5		LIQUID	ICP, ICP_MS
144433	1	URS-6B7		LIQUID	ICP, ICP_MS
144434	1	URS-7		LIQUID	ICP, ICP_MS
144435	1	URS-7A1		LIQUID	ICP, ICP_MS
144436	1	URS-7A3		LIQUID	ICP, ICP_MS
144437	1	URS-7A5		LIQUID	ICP, ICP_MS
144438	1	URS-7A7		LIQUID	ICP, ICP_MS
144439	1	URS-7B1		LIQUID	ICP, ICP_MS
144440	1	URS-7B3		LIQUID	ICP, ICP_MS
144441	1	URS-7B5		LIQUID	ICP, ICP_MS
144442	1	URS-7B7	01/01/00	LIQUID	ICP, ICP_MS
144443	1	URS-8		LIQUID	ICP, ICP_MS

Containers: 47

Samples: 47

Group	Test	Start	Method Date	Deadline	Number	Description	Fridge	Shelf
METALS	ICP	ced	30 Jun 1900	30 Jun 1900	47	ANALYSIS BY ICP	LAB 101	REF 11
METALS	ICP_MS	ced	30 Jun 1900	30 Jun 1900	47	ANALYSIS BY ICP-MS SCAN	LAB 101	REF 11

Work Orders in this SDG

18001

Thermometer: 44  
Temperature: 22me  
6/01/00Data Must be to QA: 06/12/00  
Absolute Latest Date: 06/14/00Turnaround is 14 Days from VTSR  
Page 2 of 2Page Printed: 1 Jun 2000 17:19:38  
ver (05/17/2000)

Client: DIV 20

W0: 18001

FRM-002

Client: DIV 20

W0: 18001

FRM-002



6/26/00  
nr  
Unanophore solubility experiment sample results  
as reported by Div. 01:

nr  
6/26/00

**SOUTHWEST RESEARCH INSTITUTE**  
**SAMPLE ANALYSIS DATA SHEET**

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 18001  
Client: Division 20  
Date Received: 06/01/00  
Project No.: 20.01402.561

Sample ID	Lab System ID	Calcium Result (mg/L)	Sodium Result (mg/L)	Silicon Result (mg/L)	Uranium Result (mg/L)
UM-4A	144397	502	12.6	29.0	22.9
Duplicate result	144397	501	12.5	28.9	----
RPD	144397	0.19%	0.27%	0.26%	----
UM-4B	144398	498	12.4	28.8	23.1
Spike result	144398	694	187	78.1	----
Spike added	144398	200	200	50.0	----
Recovery	144398	98.3%	87.2%	98.7%	----
UM-5A	144399	455	1.22	28.6	2.30
UM-5B	144400	455	1.18	28.8	2.28
UM-6A	144401	882	0.606	29.1	0.197
UM-6B	144402	878	0.537	28.9	0.194
UM-7A	144403	422	0.477	28.8	0.0254
UM-7B	144404	422	0.495	28.8	0.0232
URS-2	144405	489	58.1	95.8	9.55
URS-3	144406	393	11.6	49.7	473
URS-4	144407	584	35.4	10.1	101
URS-4A1	144408	491	15.2	27.4	2.38
Duplicate result	144408	----	----	----	2.44
RPD	144408	----	----	----	2.36%
URS-4A3	144409	497	15.2	29.2	2.15
Spike result	144409	----	----	----	51.4
Spike added	144409	----	----	----	50.0
Recovery	144409	----	----	----	98.4%
URS-4A5	144410	498	15.4	30.1	2.11
URS-4A7	144411	504	15.6	31.0	1.95
URS-4B1	144412	498	15.2	28.0	2.42
URS-4B3	144413	496	15.3	29.4	2.25
URS-4B5	144414	500	15.3	30.4	2.08
URS-4B7	144415	496	15.2	31.2	1.97
URS-5	144416	439	6.15	67.4	50.4
URS-5A1	144417	459	3.87	30.7	0.0792
Duplicate result	144417	463	3.77	30.9	----
RPD	144417	0.71%	2.38%	0.55%	----
URS-5A3	144418	464	3.68	32.6	0.0074
Spike result	144418	659	182	82.8	----
Spike added	144418	200	200	50.0	----
Recovery	144418	97.4%	89.2%	100.4%	----

6/26/00  
nr  
Data - cont'd.

**SOUTHWEST RESEARCH INSTITUTE**  
**SAMPLE ANALYSIS DATA SHEET**

nr  
6/26/00

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 18001  
Client: Division 20  
Date Received: 06/01/00  
Project No.: 20.01402.561

Sample ID	Lab System ID	Calcium Result (mg/L)	Sodium Result (mg/L)	Silicon Result (mg/L)	Uranium Result (mg/L)
URS-5A5	144419	458	3.71	32.9	0.0048
Duplicate result	144419	----	----	----	0.0049
RPD	144419	----	----	----	2.19%
URS-5A7	144420	458	3.80	33.3	0.0051
Spike result	144420	----	----	----	9.99
Spike added	144420	----	----	----	10.0
Recovery	144420	----	----	----	99.9%
URS-5B1	144421	461	3.78	31.0	0.0829
URS-5B3	144422	460	3.66	32.4	0.0078
URS-5B5	144423	454	3.73	32.7	0.0036
URS-5B7	144424	457	3.68	33.2	0.0032
URS-6	144425	420	22.6	29.4	19.5
URS-6A1	144426	889	3.50	32.3	0.0030
URS-6A3	144427	874	3.47	32.9	0.0026
URS-6A5	144428	863	3.49	32.7	0.0026
URS-6A7	144429	872	3.57	33.1	0.0034
URS-6B1	144430	869	3.60	31.6	0.0028
URS-6B3	144431	866	3.80	32.6	0.471
URS-6B5	144432	852	3.53	32.4	0.0018
URS-6B7	144433	859	3.62	32.3	0.0030
URS-7	144434	48.1	0.314	4.91	0.952
URS-7A1	144435	423	3.09	32.0	0.0028
URS-7A3	144436	421	3.17	33.3	0.0022
URS-7A5	144437	417	3.06	33.3	0.0016
Duplicate result	144437	418	3.07	33.3	----
RPD	144437	0.10%	0.29%	0.04%	----
URS-7A7	144438	422	3.09	33.4	0.0054
Spike result	144438	612	180	82.4	----
Spike added	144438	200	200	50.0	----
Recovery	144438	95.4%	88.2%	98.1%	----

nr  
6/26/00

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SwRI      Date Received: 06/01/00  
Matrix: Liquid      Project No.: 20.01402.561  
Work Order: 18001

Sample ID	Lab System ID	Calcium Result (mg/L)	Sodium Result (mg/L)	Silicon Result (mg/L)	Uranium Result (mg/L)
URS-7B1	144439	425	3.15	31.8	0.0035
Duplicate result	144439	----	----	----	0.0035
RPD	144439	----	----	----	1.32%
URS-7B3	144440	422	3.07	33.2	0.0062
Spike result	144440	----	----	----	0.484
Spike added	144440	----	----	----	0.500
Recovery	144440	----	----	----	95.5%
URS-7B5	144441	420	3.13	33.4	0.0034
URS-7B7	144442	424	3.21	33.6	0.0033
URS-8	144443	48.7	<0.25	19.2	0.491
LCSW1	----	19.8	17.5	4.98	0.0971
True Value	----	20.0	20.0	5.00	0.100
Recovery	----	99.0%	87.3%	99.5%	97.1%
LCSW2	----	19.9	17.5	4.92	0.0993
True Value	----	20.0	20.0	5.00	0.100
Recovery	----	99.5%	87.4%	98.3%	99.3%
LCSW3	----	19.7	17.5	4.92	0.0992
True Value	----	20.0	20.0	5.00	0.100
Recovery	----	98.6%	87.7%	98.4%	99.2%
PBW1	----	<0.05	<0.025	<0.025	<0.0001
PBW2	----	<0.05	<0.025	<0.025	<0.0001
PBW3	----	<0.05	<0.025	<0.025	<0.0001

QC Check  
Std. URS-2

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	9.55	10.0	4.50
Calcium	489	500	2.20
Silicon	95.8	100	4.20
Sodium	58.1	50.0	-16.2

QC Check  
Std. URS-3

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	473	500	5.40
Calcium	393	400	1.75
Silicon	49.7	50.0	0.600
Sodium	11.6	10.0	-16.0

QC Check  
Std. URS-4

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	101	100	-1.00
Calcium	584	600	2.67
Silicon	10.1	10.0	-1.00
Sodium	35.4	30.0	-18.0

QC Check  
Std. URS-5

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	50.4	50.0	-0.800
Calcium	439	450	2.44
Silicon	67.4	70.0	3.71
Sodium	6.15	5.00	-23.0

QC Check  
Std. URS-6

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	19.5	20.0	2.50
Calcium	420	430	2.33
Silicon	29.4	30.0	2.00
Sodium	22.6	20.0	-13.0

QC Check  
Std. URS-7

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	0.952	1.00	4.80
Calcium	48.1	50.0	3.80
Silicon	4.91	5.00	1.80
Sodium	0.314	0	####

QC Check  
Std. URS-8

Element of Interest	Analyzed Results (ppm)	Expected Results (ppm)	% Diff.
Uranium	0.491	0.500	1.80
Calcium	48.7	50.0	2.60
Silicon	19.2	20.0	4.00
Sodium	0.025	0	####

6/26/00  
Data - cont'd Summary of Uranophane Solubility  
(375/93) Samples as Analyzed by Div. 01 SWRT:

6/26/00

Uranaphane Solubility Experiment										
	Date/Time	Wt. of bottle before sampling	Wt. of bottle after sampling	Wt. of solution removed	pH of solution removed	Sample Label	Uranium Results (ppm)	Calcium Results (ppm)	Silicon Results (ppm)	Sodium Results (ppm)
URS-4A	4/14/00 - 1040				5.95	UM-4A	22.9	502	29.0	12.6
	4/19/00 - 1040	155.33	150.05	5.28	4.78	URS-4A1	2.38	491	27.4	15.2
	4/26/00 - 1350	150.06	145.03	5.03	4.93	URS-4A2				
	5/03/00 - 1010	145.03	139.98	5.05	-	URS-4A3	2.15	497	29.2	15.1
	5/10/00 - 1015	139.93	134.90	5.03	4.77	URS-4A4				
	5/18/00 - 1350	134.88	129.88	5.00	4.76	URS-4A5	2.11	498	30.1	15.4
	5/25/00 - 1000	129.86	124.66	5.20	4.82	URS-4A6				
	6/01/00 - 0945	124.64	119.61	5.03	4.81	URS-4A7	1.95	504	31.0	15.6
URS-4B	4/14/00 - 1045				5.94	UM-4B	23.1	498	28.8	12.4
	4/19/00 - 1045	155.08	150.17	4.91	4.78	URS-4B1	2.42	498	28.0	15.2
	4/26/00 - 1350	150.17	145.11	5.06	4.82	URS-4B2				
	5/03/00 - 1015	145.11	140.07	5.04	-	URS-4B3	2.25	496	29.4	15.3
	5/10/00 - 1025	140.07	134.99	5.08	4.77	URS-4B4				
	5/18/00 - 1400	134.99	129.95	5.04	4.74	URS-4B5	2.08	500	30.4	15.3
	5/25/00 - 1010	129.95	124.91	5.04	4.77	URS-4B6				
	6/01/00 - 0950	124.90	119.90	5.00	4.80	URS-4B7	1.97	496	31.2	15.2
URS-5A	4/14/00 - 1050				5.94	UM-5A	2.30	455	28.6	1.22
	4/19/00 - 1050	155.16	150.62	4.54	5.25	URS-5A1	0.0792	459	30.7	3.87
	4/26/00 - 1355	150.62	145.57	5.05	5.35	URS-5A2				
	5/03/00 - 1020	145.61	140.60	5.01	-	URS-5A3	0.0074	464	32.6	3.68
	5/10/00 - 1030	140.56	135.52	5.04	6.02	URS-5A4				
	5/18/00 - 1405	135.50	130.43	5.07	6.27	URS-5A5	0.0048	458	32.9	3.71
	5/25/00 - 1015	130.43	125.42	5.01	6.50	URS-5A6				
	6/01/00 - 0955	125.41	120.40	5.01	6.38	URS-5A7	0.0051	458	33.3	3.80
URS-5B	4/14/00 - 1100				5.94	UM-5B	2.28	455	28.8	1.18
	4/19/00 - 1100	155.31	150.30	5.01	5.20	URS-5B1	0.0829	461	31.0	3.78
	4/26/00 - 1400	150.30	145.24	5.06	5.37	URS-5B2				
	5/03/00 - 1025	145.25	140.19	5.06	-	URS-5B3	0.0078	460	32.4	3.66
	5/10/00 - 1030	140.18	135.15	5.03	6.11	URS-5B4				
	5/18/00 - 1410	135.15	130.11	5.04	6.26	URS-5B5	0.0036	454	32.7	3.73
	5/25/00 - 1000	130.12	125.12	5.00	6.39	URS-5B6				
	6/01/00 - 1000	125.12	120.14	4.98	6.48	URS-5B7	0.0032	457	33.2	3.68
URS-6A	4/14/00 - 1300				5.97	UM-6A	0.197	882	29.1	0.606
	4/19/00 - 1300	154.37	149.45	4.92	6.35	URS-6A1	0.003	889	32.3	3.50
	4/26/00 - 1405	149.44	144.40	5.04	6.32	URS-6A2				
	5/03/00 - 1025	144.41	139.37	5.04	-	URS-6A3	0.0026	874	32.9	3.47
	5/10/00 - 1035	139.35	134.31	5.04	6.35	URS-6A4				
	5/18/00 - 1415	134.29	129.26	5.03	6.37	URS-6A5	0.0026	863	32.7	3.49
	5/25/00 - 1020	129.25	124.18	5.07	6.35	URS-6A6				
	6/01/00 - 1000	124.16	119.16	5.00	6.44	URS-6A7	0.0034	872	33.1	3.57
URS-6B	4/14/00 - 1305				5.97	UM-6B	0.194	878	28.9	0.537
	4/19/00 - 1305	154.61	149.67	4.94	6.33	URS-6B1	0.0028	869	31.6	3.60
	4/26/00 - 1410	149.67	144.64	5.03	6.35	URS-6B2				
	5/03/00 - 1030	144.66	139.64	5.02	-	URS-6B3	0.471	866	32.6	3.80
	5/10/00 - 1040	139.63	134.59	5.04	6.34	URS-6B4				
	5/18/00 - 1415	134.58	129.55	5.03	6.57	URS-6B5	0.0018	852	32.4	3.53
	5/25/00 - 1025	129.56	124.52	5.04	6.42	URS-6B6				
	6/01/00 - 1005	124.51	119.50	5.01	6.41	URS-6B7	0.003	859	32.3	3.62
URS-7A	4/14/00 - 1310				5.92	UM-7A	0.0254	422	28.8	0.477
	4/19/00 - 1310	155.25	150.31	4.94	6.40	URS-7A1	0.0028	423	32.0	3.09
	4/26/00 - 1420	150.31	145.26	5.05	6.46	URS-7A2				
	5/03/00 - 1035	145.26	140.24	5.02	-	URS-7A3	0.0022	421	33.3	3.17
	5/10/00 - 1040	140.24	135.21	5.03	6.36	URS-7A4				
	5/18/00 - 1420	135.20	130.16	5.04	6.29	URS-7A5	0.0016	417	33.3	3.06
	5/25/00 - 1030	130.17	125.15	5.02	6.21	URS-7A6				
	6/01/00 - 1010	125.14	120.12	5.02	6.40	URS-7A7	0.0054	422	33.4	3.09
URS-7B	4/14/00 - 1315				5.92	UM-7B	0.0232	422	28.8	0.495
	4/19/00 - 1315	155.25	150.27	4.98	6.41	URS-7B1	0.0035	425	31.8	3.15
	4/26/00 - 1425	150.28	145.25	5.03	6.47	URS-7B2				
	5/03/00 - 1040	145.25	140.26	4.99	-	URS-7B3	0.0062	422	33.2	3.07
	5/10/00 - 1045	140.26	135.24	5.02	6.41	URS-7B4				
	5/18/00 - 1420	135.22	130.21	5.01	6.32	URS-7B5	0.0034	420	33.4	3.13
	5/25/00 - 1030	130.23	125.20	5.03	6.41	URS-7B6				
	6/01/00 - 1015	125.19	120.16	5.03	6.50	URS-7B7	0.0033	424	33.6	3.21

01/09/01 JP 1040hrs

Dissolution of synthesized uranophane

Obj - Dissolve synthesized uranophane (uranophane Nos 5+6) for chemical analysis

Method - Dissolution in 0.1M HCl

Materials + supplies:

0.1 M HCl - place 4.1 ml of conc HCl (lot 418110) in 500 ml volumetric flask + make up to mark with DI water

Synthesized uranophane Nos 5+6 (325/68-69)

Mettler AE 240 balance

125 ml PP bottles

Stirrer and stir bars

Mettler PM4000 balance

Weighing paper

Procedure -

① Label 3 125 ml PP bottles as follows:

U56-1

U56-2

U56-3

② Place 100 g of 0.1M HCl into each bottle using analytical balance. Record weight of 0.1M HCl added to each bottle.

	Wt of 0.1M HCl
U56-1	100.04 g
U56-2	100.02 g
U56-3	100.04 g

③ Place 0.1 g of uranophane Nos 5+6 into each of the sample bottles. Record weight of solid added

	Wt of uranophane 5+6 added
U56-1	0.1019 g
U56-2	0.1028 g
U56-3	0.1000 g

④ Place a stir bar (Teflon) in each bottle. Place bottles on a stirrer until uranophane is dissolved.

01/09/01 gp 1340 hr.

## Chemistry of <sup>synthetic</sup> Uranophane

Obj - determine the chemical content of synthetic uranophane (U, SiO<sub>2</sub>, Ca, Na).

Method - analyse samples of dissolved synthetic uranophane (Nos 5+6) by atomic absorption (Na+Ca) and spectrophotometry (Si).

### Equipment & materials.

- Perkin Elmer 3100 Atomic Absorption spectrophotometer
- Samples U56-1, U56-2, and U56-3
- 1% HNO<sub>3</sub> solution
- glassware & plasticware as needed
- Na Hollow cathode lamp
- Ca Hollow cathode lamp
- 1000 ppm Na standard (lot 7440-23-5)
- QC check stds URS-3, URS-5, URS-7
- 1000 ppm U standard (lot 7440-61-1)
- 1000 ppm Ca standard (lot 986835-2+)
- 1000 ppm Si std (lot 7440-21-3)
- HCl concentrate (lot 418110)
- Milton Roy
- 50 ml nessler tubes
- 5 ml glass cells

### Procedure

#### Na analysis by AA

① Na in samples U56-1, U56-2, and U56-3 was determined using analytical method outlined in the manual entitled "Analytical Methods for Atomic Absorption Spectrophotometry."

② The Perkin Elmer 3100 AA was setup and calibrated for Na analysis using methods outlined in the manual entitled "Running the 3100 Atomic Absorption Spectrometer."

③ Preparation of Na standard for AA calibration.

- a 1 ppm Na standard was prepared by adding 1 ml of the 1000 ppm Na AA standard to a 1000 ml volumetric flask and making up to mark with 1% HNO<sub>3</sub> soln. ~~DI water~~  
gp 1/9/01

④ QC check standards <sup>for Na</sup> were prepared as follows.

URS-3 was diluted by 20 - 1 ml added to 10 ml vol flask and made up to mark with 1% HNO<sub>3</sub> soln.  
URS-5 was diluted by 10 - 1 ml added to 10 ml vol flask and made up to mark with 1% HNO<sub>3</sub> soln.  
URS-7 was used as is (no dilution).



⑤ Samples U56-1, U56-2, and U56-3 were prepared for analysis by taking ~10 ml aliquots and placing in PP sample cups.

⑥ Results of Na analysis are shown below.

Label	Na (ppm)	Dilution Factor
U56-1	.191	-
U56-2	.149	-
U56-3	.148	-
URS-3	.678	20X
URS-5	.737	10X
URS-7	0	

## Ca Analysis by AA

① Calibration stds were prepared in matrices which were similar to those expected for the dissolved monophase samples. The solns had the following characteristics:

0.1 M HCl  
 ~70 ppm Si  
 ~600 ppm U  
 100, 75, 50, and 25 ppm Ca

Calibration standards were prepared and labeled as follows:

Label  
 100 ppm Ca - Into a 100 ml volumetric flask the following amounts of reagents were added - .82 ml conc HCl, 7 ml Si std (1000 ppm), 60 ml U std (1000 ppm) and 10 ml Ca std (1000 ppm). DI water was added up to mark.  
 75 ppm Ca - Same as above except 7.5 ml of Ca std was added.  
 50 ppm Ca - Same as above except 5.0 ml of Ca std was added.  
 25 ppm Ca - Same as above except 2.5 ml of Ca std was added.

② Sample and standards were prepared for analysis by diluting by 25 in 25 ml volumetric flasks. For example, 1 ml of sample and standards were placed in 25 ml volumetric flask and made up to mark with DI water.

③ The Perkin Elmer 3100 was setup and calibrated for Ca analysis using the diluted calibration standards & methods outlined in the manual entitled "Running the 3100 Atomic Absorption Spectrometer".

④ Results of Ca analysis for the sample are shown below:

label	Ca ppm	Dilute	Ca in soln ppm
U56-1	1.87	25x	46.75
U56-2	1.91	25x	47.75
U56-3	1.844	25x	46.1

Si analysis by spectrophotometry

① Si in sample was measured using Technical Operating Procedure TOP-14.

② Reagents used in the analysis are:

10 ppm Si standard - placed 10 ml of 1000 ppm Si std in a 1000 ml volumetric flask and made up to mark with DI water. Labeled plastic 1L bottle as 10 ppm Si standard and transferred soln from volumetric flask to the labeled bottle.

1+1 HCl - placed 50 ml conc HCl and 50 ml DI water in a 250 ml plastic bottle and labeled 1+1 HCl.

Oxalic acid soln - see 325/13.

Ammonium molybdate reagent - see 325/13.

③ Results of Si analysis are shown below:

Label	Si (ppm)
USb-1	67.54
USb-2	67.76
USb-3	65.56

2/1/01 JP 1000 hrs.

The solids in sample bottles URS-4B, URS-5B, and URS-7B were removed, dried, and sent to Div 18 for XRD analysis. The dried solids were placed in plastic containers and labeled as follows:

URS-4B-XRD

URS-5B-XRD

URS-7B-XRD.

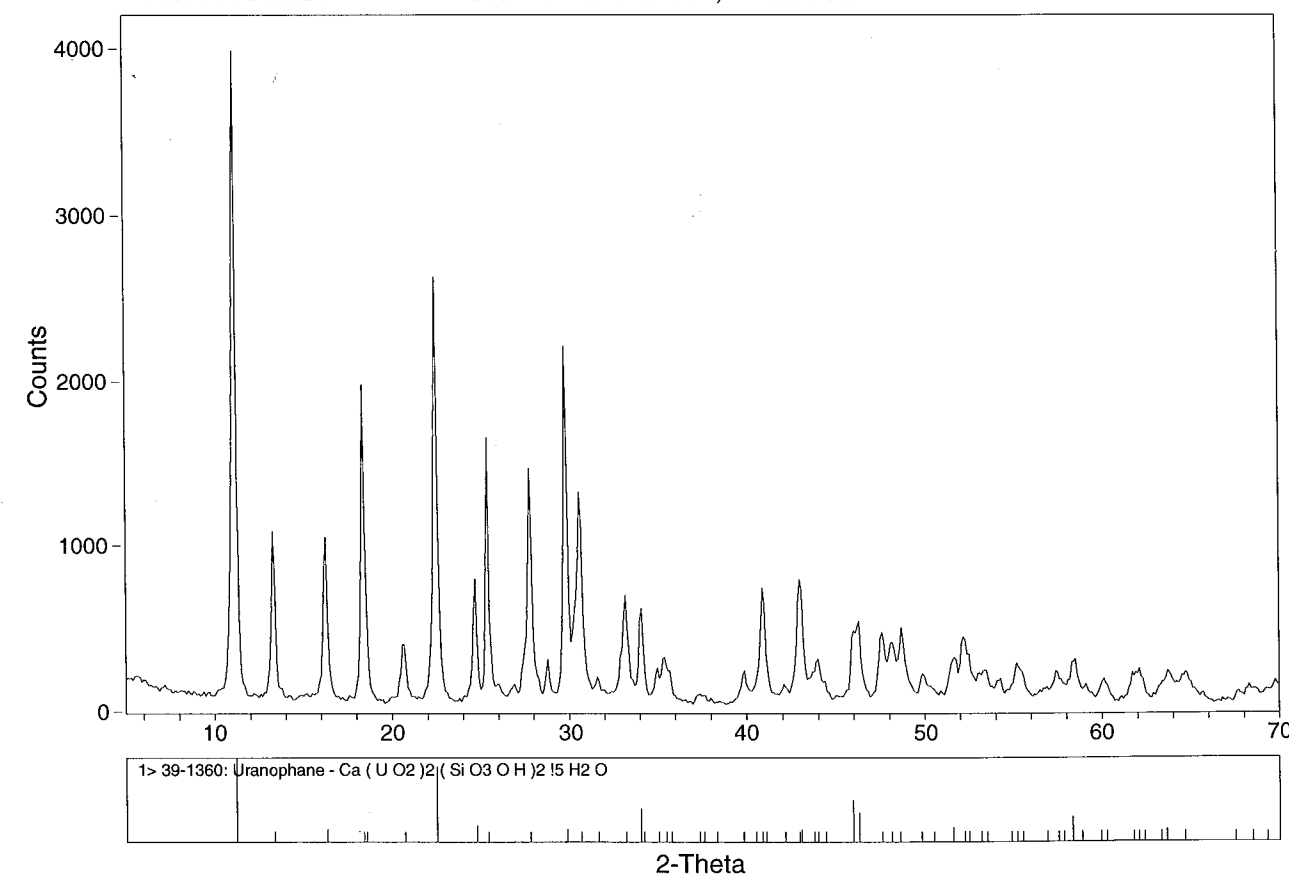
2/8/01 JP 1015 hrs.

Results of XRD analyses:

ID: URS-4B-XRD - 20.01402.561

File: F1701.MDI

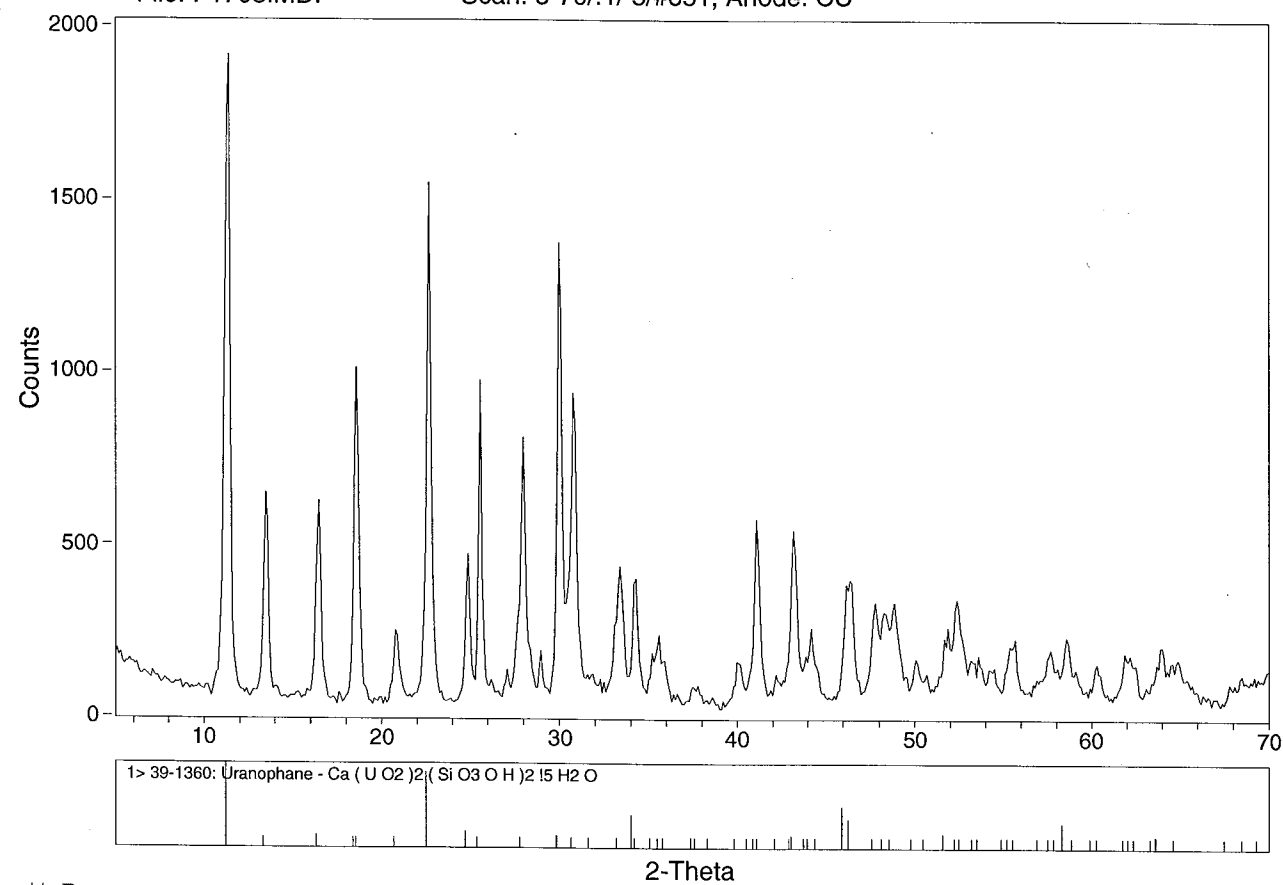
Scan: 5-70/1/ 5/#651, Anode: CU



ID: URS-5B-XRD - 20.01402.561

File: F1703.MDI

Scan: 5-70/.1/ 5/#651, Anode: CU

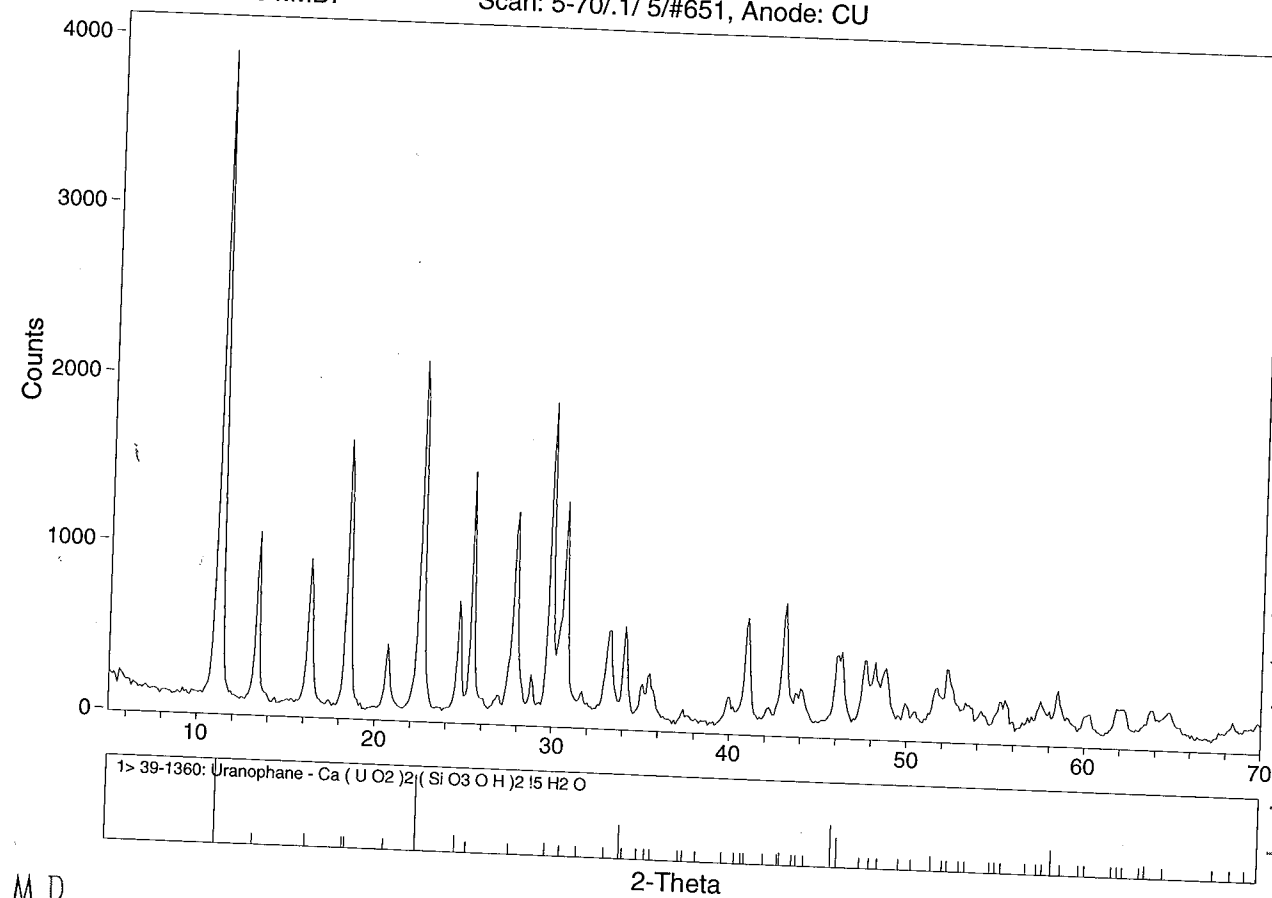


MDI  
MATERIALS DATA, INC.  
Southwest Research Institute - Thu Feb 08 2001 @ 10:29am

ID: URS-7B-XRD - 20.01402.561

File: F1704.MDI

Scan: 5-70/.1/ 5/#651, Anode: CU



MDI  
MATERIALS DATA, INC.  
Southwest Research Institute - Thu Feb 08 2001 @ 10:30am

2/22/01 JF

A new set of uranophane solubility experiments will be conducted. Preparation of matrix solutions were initiated using the procedure on the following page.

2/22/01 JF

Preparation of solutions for uranophane solubility studies

Written by: J. D. Prikryl  
Date: 02/20/01

Objective: Prepare a series of solutions containing U, Ca, and SiO<sub>2</sub> for use in the uranophane solubility experiments. The solutions will have a Ca content of 10<sup>-2</sup> M and a SiO<sub>2</sub> content of 10<sup>-3</sup> M. U content will vary from 10<sup>-5</sup> to 10<sup>-7</sup> M. The pH of the solutions will be approximately 6.0.

Method: Addition of chemical reagents containing Ca and U to an approximate 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Materials and Equipment:

- previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution
- CaCl<sub>2</sub>·2H<sub>2</sub>O; F.W. 147.02; (lot no. 995498)
- UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O; F.W. 502.13; (lot no. 8640 KCAP)
- CaCO<sub>3</sub>; F.W. 100.09; (lot no. 986396)
- Polycarbonate bottles (500 ml)
- Polypropylene bottles (15 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Mettler analytical balance (AE240)
- weighing paper and boats
- Fisher Versabath Model 236

Procedure

1. Transfer about 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution to a 250 ml polycarbonate bottle. Add 0.00502 g of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to the bottle. Label the bottle as 10<sup>-4</sup> m U.
2. Label 6 500 ml polycarbonate bottles as follows:
  - 1.5x10<sup>-5</sup> m U
  - 1.0x10<sup>-5</sup> m U
  - 3.33x10<sup>-6</sup> m U
  - 1.0x10<sup>-6</sup> m U
  - 3.33x10<sup>-7</sup> m U
  - 1.0x10<sup>-7</sup> m U
3. Transfer about 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution into each 500 ml bottle.

2/22/01  
1300 hr  
JF

1305 hr  
JF

1400 hr  
JF



1405hr  
JP

4. Carefully add the following quantities of reagents and 10<sup>-4</sup> M U solution to the specified bottle. Use weighing paper and weigh out reagents as accurately as possible. For the 10<sup>-4</sup> M U solution use plastic weighing boats and rinse boats using the 10<sup>-3</sup> M SiO<sub>2</sub> solution. After adding reagents make up to a final weight of 250 g with the 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Bottle Label	CaCl <sub>2</sub> ·2H <sub>2</sub> O	CaCO <sub>3</sub>	10 <sup>-4</sup> M U solution
1.5x10 <sup>-5</sup> m U	0.36695 g	0.00041 g	37.5 g
1.0x10 <sup>-5</sup> m U	0.36712 g	0.00029 g	25 g
3.33x10 <sup>-6</sup> m U	0.36734 g	0.00014 g	7.5 g
1.0x10 <sup>-6</sup> m U	0.36742 g	0.00009 g	2.5 g
3.33x10 <sup>-7</sup> m U	0.36744 g	0.00007 g	0.75 g
1.0x10 <sup>-7</sup> m U	0.36745 g	0.00006 g	0.25 g

1445hr  
JP  
1500hr  
JP

5. Place a teflon stir bar in each bottle and measure and record pH.
6. Place bottles in shaker water bath set at 25 °C. Allow solutions to equilibrate with air by loosely capping bottles.
7. Measure and record pH periodically (e.g., daily) to determine when solutions reach equilibrium with air.
8. When equilibrium is reached record the weight of each U matrix solution bottle.

Bottle	Wt (g)
1.5x10 <sup>-5</sup> m U	331.80
1.0x10 <sup>-5</sup> m U	330.55
3.33x10 <sup>-6</sup> m U	326.91
1.0x10 <sup>-6</sup> m U	329.35
3.33x10 <sup>-7</sup> m U	328.08
1.0x10 <sup>-7</sup> m U	329.49

JP  
3/5/01  
0900hr

9. Take two 5ml sample aliquots from each U matrix solution and place in 15 ml polypropylene bottles for chemical analysis. Use an Oxford pipettor and 5 ml plastic tips to take samples. Preserve the samples by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle. Chemical analysis will show the initial Ca, SiO<sub>2</sub>, and U contents of the solutions before addition of uranophane. Label the samples as shown below.

Label	U matrix solution sampled
UMA-5A and UMA-5B	1.5x10 <sup>-5</sup> m U
UMA-5C and UMA-5D	1.0x10 <sup>-5</sup> m U
UMA-6A and UMA-6B	3.33x10 <sup>-6</sup> m U
UMA-6C and UMA-6D	1.0x10 <sup>-6</sup> m U
UMA-7A and UMA-7B	3.33x10 <sup>-7</sup> m U
UMA-7C and UMA-7D	1.0x10 <sup>-7</sup> m U

JP  
3/5/01  
0905hr

JP  
3/5/01  
0910hr

12. After sampling record the weight of each U matrix solution bottle.

Bottle	Wt (g)
1.5x10 <sup>-5</sup> m U	321.77
1.0x10 <sup>-5</sup> m U	320.46
3.33x10 <sup>-6</sup> m U	316.86
1.0x10 <sup>-6</sup> m U	319.32
3.33x10 <sup>-7</sup> m U	318.04
1.0x10 <sup>-7</sup> m U	319.45

pH measurements of matrix solutions

	1.5x10 <sup>-5</sup> m U	1.0x10 <sup>-5</sup> m U	3.33x10 <sup>-6</sup> m U	1.0x10 <sup>-6</sup> m U	3.33x10 <sup>-7</sup> m U	1.0x10 <sup>-7</sup> m U
2/26/01 JP 1015hr	6.05	6.10	6.04	6.06	6.13	6.01
3/1/01 JP 0730hr	6.15	6.17	6.08	6.11	6.18	6.06
3/5/01 JP 0810hr	5.95 6.04	6.04 6.10	6.02 6.05	6.07	6.14	6.18

2/26/01 JF

log K calculations for uranophane solubility  
experimental data shown on p 104.

Calculations were carried out using an  
Excel spreadsheet entitled log K calc-2000.xls.

The raw experimental data was first corrected  
using the QA sample results shown on  
p103. These corrected data were then  
used in EQ3 speciation calculations to  
determine the log activity of  $\text{Ca}^{++}$ ,  $\text{SiO}_2$ ,  $\text{UO}_2^{++}$ ,  
and  $\text{H}^+$ . The values were entered into  
the spreadsheet and log K was calculated  
using the formula:

$$\log K = \log(a_{\text{Ca}^{++}} \cdot (a_{\text{UO}_2^{++}})^2 \cdot (a_{\text{SiO}_2})^2 \cdot (a_{\text{H}^+})^6)$$

A listing of the spreadsheet is shown on  
the following pages:

**Uranophane solubility experimental results**  
Corrected results based on QA samples and calculation of log K using EQ3 speciation results

Matrix solutions contained 10-2 M Ca, 10-3 M SiO<sub>2</sub>, and U contents of 10-4, 10-5, 10-6, and 10-7 M were prepared. The reagents used included:

The reagents used included:

- CaCl<sub>2</sub>·H<sub>2</sub>O for Ca
- Silicic acid dissolved in water for SiO<sub>2</sub>
- Uranyl nitrate for U

Chemical analyses of matrix solutions are shown below:

Label	U conc of matrix pH	U ppm	Ca ppm	Si ppm	Na ppm	Corrected values based on average error				Values for EQ3 calcs		
						U ppm	Ca ppm	Si ppm	Na ppm	UO2++	SiO2	
UM-4A	1.0x10-4	5.95	22.9	502	29	12.6	23.49133	515.1069	29.65854	10.75555	26.64943	63.44541
UM-4B	1.0x10-4	5.94	23.1	498	28.8	12.4	23.6965	511.0025	29.45399	10.58483	26.88218	63.00785
UM-5A	1.0x10-5	5.94	2.3	455	28.6	1.22	2.359391	466.8798	29.24945	1.04141	2.676581	62.5703
UM-5B	1.0x10-5	5.94	2.28	455	28.6	1.18	2.338875	466.8798	29.45399	1.007266	2.653306	62.00785
UM-6A	1.0x10-6	5.97	0.197	882	29.1	0.606	0.202087	905.0284	29.76081	0.517291	0.229255	63.66418
UM-6B	1.0x10-6	5.97	0.194	878	28.9	0.537	0.19901	900.924	29.55627	0.458391	0.225764	63.22663
UM-7A	1.0x10-7	5.92	0.0254	422	28.8	0.477	0.026056	433.0181	29.45399	0.407174	0.029559	63.00785
UM-7B	1.0x10-7	5.92	0.0232	422	28.8	0.495	0.023799	433.0181	29.45399	0.422539	0.026999	63.00785

### Experimental data and results.

Experiment label	pH of solution removed	Sample Label	Uranium Results (ppm)	Calcium Results (ppm)	Silicon Results (ppm)	Sodium Results (ppm)
URS-4A	4.78	URS-4A1	2.38	491	27.4	15.2
	4.93	URS-4A2				
	4.79	URS-4A3	2.15	497	29.2	15.1
	4.77	URS-4A4				
	4.76	URS-4A5	2.11	498	30.1	15.4
	4.82	URS-4A6				
	4.81	URS-4A7	1.95	504	31	15.6
URS-4B	4.78	URS-4B1	2.42	498	28	15.2
	4.82	URS-4B2				
	4.80	URS-4B3	2.25	496	29.4	15.3
	4.77	URS-4B4				
	4.74	URS-4B5	2.08	500	30.4	15.3
	4.77	URS-4B6				
	4.80	URS-4B7	1.97	496	31.2	15.2
URS-5A	5.25	URS-5A1	0.0792	459	30.7	3.87
	5.35	URS-5A2				
	5.69	URS-5A3	0.0074	464	32.6	3.68
	6.02	URS-5A4				
	6.27	URS-5A5	0.0048	458	32.9	3.71
	6.50	URS-5A6				
	6.38	URS-5A7	0.0051	458	33.3	3.8
URS-5B	5.20	URS-5B1	0.0829	461	31	3.78
	5.37	URS-5B2				
	5.74	URS-5B3	0.0078	460	32.4	3.66
	6.11	URS-5B4				
	6.26	URS-5B5	0.0036	454	32.7	3.73
	6.39	URS-5B6				
	6.48	URS-5B7	0.0032	457	33.2	3.68
URS-6A	6.35	URS-6A1	0.003	889	32.3	3.5
	6.32	URS-6A2				
	6.34	URS-6A3	0.0026	874	32.9	3.47
	6.35	URS-6A4				

Corrected values based on average error

U ppm	Ca ppm	Si ppm	Na ppm
2.441457	503.8197	28.0222	12.97495
0	0	0	0
2.205518	509.9763	29.86308	12.88959
0	0	0	0
2.164485	511.0025	30.78352	13.14567
0	0	0	0
2.000354	517.1591	31.70395	13.3164
0	0	0	0
0	0	0	0
2.48249	511.0025	28.63583	12.97495
0	0	0	0
2.3081	508.9502	30.06762	13.06031
0	0	0	0
2.133711	513.0547	31.09033	13.06031
0	0	0	0
2.02087	508.9502	31.90849	12.97495
0	0	0	0
0	0	0	0
0	0	0	0
0.081245	470.9842	31.39714	3.303491
0	0	0	0
0.007591	476.1147	33.34029	3.141304
0	0	0	0
0.004924	469.9581	33.6471	3.166912
0	0	0	0
0.005232	469.9581	34.05618	3.243738
0	0	0	0
0	0	0	0
0.085041	473.0364	31.70395	3.226655
0	0	0	0
0.008001	472.0103	33.13574	3.124231
0	0	0	0
0.003693	465.8536	33.44256	3.183984
0	0	0	0
0.003283	468.932	33.95391	3.141304
0	0	0	0
0	0	0	0
0	0	0	0
0.003077	912.2112	33.03347	2.987653
0	0	0	0
0.002667	896.8196	33.6471	2.962045
0	0	0	0

Values for EQ3 calcs

UO2++	SiO2
2.769679	59.94497
0	0
2.502021	63.88296
0	0
2.455472	65.85196
0	0
2.269275	67.82095
0	0
0	0
2.816228	61.25763
0	0
2.618394	64.32051
0	0
2.42056	66.50829
0	0
2.292549	68.25851
0	0
0	0
0.092167	67.16462
0	0
0.008612	71.32139
0	0
0.005586	71.97777
0	0
0.005935	72.85283
0	0
0	0
0.096473	67.82095
0	0
0.009077	70.88393
0	0
0.004189	71.54016
0	0
0.003724	72.63403
0	0
0	0
0.003491	70.56505
0	0
0.003026	71.97777
0	0

Log activities from EQ3 speculations			
Ca++	SiO2	UO2++	H+
-2.1891	-3.001	-5.4419	-4.78
-2.185	-2.9734	-5.5131	-4.75
-2.1844	-2.9602	-5.4861	-4.76
-2.1804	-2.9471	-5.5383	-4.81
-2.1844	-2.9916	-5.4364	-4.78
-2.1857	-2.9704	-5.4737	-4.8
-2.1831	-2.9559	-5.4857	-4.74
-2.1857	-2.9448	-5.5286	-4.8
-2.2101	-2.9516	-7.1591	-5.25
-2.2065	-2.9256	-8.7402	-5.65
-2.2108	-2.9217	-9.9581	-6.27
-2.2108	-2.9165	-10.1437	-6.35
-2.2086	-2.9474	-7.0963	-5.2
-2.2093	-2.9282	-8.7964	-5.74
-2.2137	-2.9243	-10.0641	-6.26
-2.2116	-2.9178	-10.5408	-6.46
-1.9945	-2.9297	-10.3178	-6.35
-1.9999	-2.9217	-10.3606	-6.34

UO2++ K	Log(K)
4.03E+09	9.6051
1	0
3.82E+09	9.582
1	0
3.04E+09	9.483
1	0
5.11E+09	9.7088
1	0
1	0
1	0
4.36E+09	9.6396
1	0
5.32E+09	9.7261
1	0
2.36E+09	9.3737
1	0
4.65E+09	9.6679
1	0
1	0
1	0
1.17E+09	9.0683
1	0
4E+08	8.6019
1	0
4.46E+09	9.6496
1	0
8.89E+09	9.9488
1	0
1	0
1	0
8.02E+08	8.904
1	0
6.05E+08	8.7815
1	0
2.34E+09	9.3695
1	0
5.64E+09	9.7512
1	0
1	0
1	0
4.08E+09	9.6103
1	0
2.99E+09	9.4755
1	0

	6.37	URS-6A5	0.0026	863	32.7	3.49	0.002667	885.5324	33.44256	2.979117	0.003026
	6.35	URS-6A6					0	0	0	0	
	6.44	URS-6A7	0.0034	872	33.1	3.57	0.003488	894.7673	33.85164	3.047406	0.003957
URS-6B							0	0	0	0	
							0	0	0	0	
							0	0	0	0	
							0	0	0	0	
	6.33	URS-6B1	0.0028	869	31.6	3.6	0.002872	891.689	32.31758	3.073014	0.003256
	6.35	URS-6B2					0	0	0	0	
	6.35	URS-6B3	0.471	866	32.6	3.8	0.483162	888.6107	33.34029	3.243738	0.548111
	6.34	URS-6B4					0	0	0	0	
6.57	URS-6B5	0.0018	852	32.4	3.53	0.001846	874.2452	33.13574	3.013261	0.002098	
6.42	URS-6B6					0	0	0	0		
6.41	URS-6B7	0.003	859	32.3	3.62	0.003077	881.4279	33.03347	3.090087	0.003497	
URS-7A							0	0	0	0	
							0	0	0	0	
							0	0	0	0	
	6.40	URS-7A1	0.0028	423	32	3.09	0.002872	434.0443	32.72666	2.637671	0.003256
	6.46	URS-7A2					0	0	0	0	
	6.41	URS-7A3	0.0022	421	33.3	3.17	0.002257	431.992	34.05618	2.70596	0.002596
	6.36	URS-7A4					0	0	0	0	
	6.29	URS-7A5	0.0016	417	33.3	3.06	0.001641	427.8876	34.05618	2.612062	0.001862
6.21	URS-7A6					0	0	0	0		
6.40	URS-7A7	0.0054	422	33.4	3.09	0.005539	433.0181	34.15845	2.637671	0.006284	
URS-7B							0	0	0	0	
							0	0	0	0	
							0	0	0	0	
	6.41	URS-7B1	0.0035	425	31.8	3.15	0.00359	436.0965	32.52212	2.688888	0.004073
	6.47	URS-7B2					0	0	0	0	
	6.44	URS-7B3	0.0062	422	33.2	3.07	0.00636	433.0181	33.95391	2.620598	0.007215
	6.41	URS-7B4					0	0	0	0	
	6.32	URS-7B5	0.0034	420	33.4	3.13	0.003488	430.9659	34.15845	2.671815	0.003957
6.41	URS-7B6					0	0	0	0		
6.50	URS-7B7	0.0033	424	33.6	3.21	0.003385	435.0704	34.36299	2.740105	0.00384	

-2.004	-2.9244	-10.4182	-6.37
-2.0006	-2.9191	-10.4373	-6.44
-2.0017	-2.9392	-10.3093	-6.33
-2.0029	-2.9257	-8.1682	-6.35
-2.0081	-2.9284	-10.9673	-6.57
-2.0055	-2.9297	-10.4335	-6.41
-2.2373	-2.9338	-10.4425	-6.4
-2.2389	-2.9185	-10.5665	-6.41
-2.2421	-2.9184	-10.4733	-6.29
-2.2381	-2.9152	-10.1575	-6.4
-2.2357	-2.9365	-10.365	-6.41
-2.2381	-2.9178	-10.1753	-6.44
-2.2397	-2.9151	-10.2036	-6.32
-2.2365	-2.9126	-10.566	-6.5

3.39E+09	9.5308 *
1	0
8.45E+09	9.9266 *
1	0
1	0
3.03E+09	9.4813 *
1	0
8.12E+13	13.9093
1	0
4.17E+09	9.6205 *
1	0
5.35E+09	9.7281 *
1	0
1	0
2.57E+09	9.4101 *
1	0
1.8E+09	9.2551 *
1	0
5.23E+08	8.7185
1	0
1.04E+10	10.0185 *
1	0
1	0
4.18E+09	9.8213 *
1	0
1.64E+10	10.2157 *
1	0
2.77E+09	9.4429
1	0
6.4E+09	9.8063 *

### QA Standard analyses

Standard Label	U ppm Measured	U ppm Expected	U error	Ca ppm Measured	Ca ppm Expected	Ca error	Si ppm Measured	Si ppm Expected	Si error	Na ppm Measured	Na ppm Expected	Na error
URS-2	9.55	10	1.04712	499	500	1.022495	95.8	100	1.043841	58.1	50	0.860585
URS-3	473	500	1.057082	393	400	1.017812	49.7	50	1.006036	11.6	10	0.862069
URS-4	101	100	0.990099	584	600	1.027397	10.1	10	0.990099	35.4	30	0.847458
URS-5	50.4	50	0.992063	439	450	1.025057	67.4	70	1.038576	6.15	5	0.813008
URS-6	19.5	20	1.025641	420	430	1.02381	29.4	30	1.020408	22.6	20	0.884956
URS-7	0.952	1	1.05042	48.1	50	1.039501	4.91	5	1.01833	0.314	0	
URS-8	0.491	0.5	1.01833	48.7	50	1.026694	19.2	20	1.041667	0.025	0	
Sample Error												
Average			1.025822			1.026109			1.022708			0.853615
Median			1.025641			1.025057			1.020408			0.860585

Average log K and standard deviation for solutions with pHs greater than 6.

2/24/01 History of excel file hogk calc. 2000.xls is shown above

JP 3/5/01 0900hr

Uranophane solubility experiment URSA

Written by: J. D. Prikryl  
Date: 02/20/01

Objective: Determine the solubility of synthesized uranophane.

Method: Monitor the reaction of synthesized uranophane with solutions containing U, Ca, and SiO<sub>2</sub>.

Materials and Equipment:

- Synthesized uranophane Na<sub>5</sub>+Na<sub>6</sub> 325/69
- Previously prepared U matrix solutions with U concentrations from 10<sup>-5</sup> to 10<sup>-7</sup>
- Polycarbonate bottles (250 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Fisher Versabath Model 236
- Mettler analytical balance (AE240 and PM4600)
- Pipets and pipet tips

Procedure

1. Label 12 250 ml polycarbonate bottles as follows and record weights.

3/5/01  
JP  
0915hr

Label	Weight (g)
URSA-5A	51.62
URSA-5B	51.46
URSA-5C	51.44
URSA-5D	51.34
URSA-6A	51.34
URSA-6B	51.30
URSA-6C	51.31
URSA-6D	51.45
URSA-7A	51.48
URSA-7B	51.59
URSA-7C	51.46
URSA-7D	51.32

2. To each sample bottle add 0.5±0.0010 g of synthesized uranophane. Record the weight of uranophane added to each bottle.

3/5/01  
JP  
0920hr

Bottle	Wt of uranophane (g)
URSA-5A	.5004
URSA-5B	.5002
URSA-5C	.5002
URSA-5D	.5005
URSA-6A	.5000
URSA-6B	.5002
URSA-6C	.5007
URSA-6D	.5007
URSA-7A	.5006
URSA-7B	.5005
URSA-7C	.5003
URSA-7D	.5005

3. To each bottle add 100±0.05 g of the specified U matrix solution shown in the table below. Record the weight of matrix solution added.

3/5/01 JP

Bottle	U matrix solution	Wt of U matrix solution (g)
1004hr URSA-5A	1.5x10 <sup>-5</sup> m U	100.07
1006hr URSA-5B	1.5x10 <sup>-5</sup> m U	100.00
1009hr URSA-5C	1.0x10 <sup>-5</sup> m U	100.07
1010hr URSA-5D	1.0x10 <sup>-5</sup> m U	100.06
1012hr URSA-6A	3.33x10 <sup>-6</sup> m U	100.04
1013hr URSA-6B	3.33x10 <sup>-6</sup> m U	100.06
1015hr URSA-6C	1.0x10 <sup>-6</sup> m U	100.05
1017hr URSA-6D	1.0x10 <sup>-6</sup> m U	100.03
1018hr URSA-7A	3.33x10 <sup>-7</sup> m U	100.03
1019hr URSA-7B	3.33x10 <sup>-7</sup> m U	100.05
1020hr URSA-7C	1.0x10 <sup>-7</sup> m U	100.04
1022hr URSA-7D	1.0x10 <sup>-7</sup> m U	100.04

4. Record the initial sample weights (bottle + synthesized uranophane + U matrix solution).

3/5/01  
JP  
1025hr

Sample	Initial wt (g)
URSA-5A	152.18
URSA-5B	151.96
URSA-5C	151.99
URSA-5D	151.86
URSA-6A	151.84
URSA-6B	151.80
URSA-6C	151.82
URSA-6D	151.94
URSA-7A	152.01
URSA-7B	152.11
URSA-7C	152.00
URSA-7D	151.86

5. Cover bottle openings with parafilm with punched holes and place in a shaking water bath set at 25°C.

3/5/01  
JP  
1045hr

6. rotary shaker at room temp.  
JP 3/5/01 1050hr

6. At 1 week intervals record the weight of each bottle and then remove 5 ml aliquots from each bottle. Transfer the samples to 15 ml polypropylene bottles for pH measurement and analysis of Ca, SiO<sub>2</sub>, and U. Record the weights of the bottles after sampling. Below are tables into which data can be recorded. Preserve samples by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle.

URSA-5A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12 0904	151.06	146.03	5.03	5.40 20.1°C	URSA-5A-1
3/19 0819	144.89	139.84	5.05	5.40 18.6°C	URSA-5A-2
3/26 0750	138.31	133.78	5.03	5.39 19.1°C	URSA-5A-3
4/2 0724	132.78	127.86	4.92	5.41 21.2°C	URSA-5A-4
4/9 0814	127.11	122.04	5.09	5.40 21.6°C	URSA-5A-5
4/16 1759	121.16	116.18	4.98	5.40 22.5°C	URSA-5A-6
4/23 1811	115.27	110.33	4.94	5.41 22.0°C	URSA-5A-7
4/30 1722	109.20	104.18	5.02	5.42 21.9°C	URSA-5A-8

URSA-5B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12 0906	150.86	145.85	5.01	5.38 20.1°C	URSA-5B-1
3/19 0820	144.70	139.66	5.04	5.40 18.6°C	URSA-5B-2
3/26 0753	138.67	133.66	5.01	5.40 19.1°C	URSA-5B-3
4/2 0727	132.63	127.59	5.04	5.40 21.2°C	URSA-5B-4
4/9 0815	126.77	121.75	5.02	5.39 21.6°C	URSA-5B-5
4/16 1801	120.89	115.94	4.95	5.39 22.5°C	URSA-5B-6
4/23 1812	114.96	109.96	5.00	5.40 22.0°C	URSA-5B-7
4/30 1724	108.77	103.76	5.01	5.41 21.9°C	URSA-5B-8

URSA-5C

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12 0907	150.78	145.76	5.02	5.48 20.1°C	URSA-5C-1
3/19 0822	144.40	139.39	5.01	5.46 18.6°C	URSA-5C-2
3/26 0755	138.13	133.13	5.00	5.45 19.1°C	URSA-5C-3
4/2 0729	131.84	126.80	5.04	5.48 21.2°C	URSA-5C-4
4/9 0816	125.76	120.72	5.04	5.47 21.6°C	URSA-5C-5
4/16 1802	118.88	114.08	4.80	5.46 22.5°C	URSA-5C-6
4/23 1813	112.77	107.80	4.97	5.44 22.0°C	URSA-5C-7
4/30 1726	106.37	101.42	4.95	5.46 21.9°C	URSA-5C-8

URSA-5D

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 909	150.50	145.47	5.03	5.45 20.1°C	URSA-5D-1
3/19 0824	143.89	138.88	5.01	5.48 18.6°C	URSA-5D-2
3/26 0756	137.36	132.35	5.01	5.45 19.1°C	URSA-5D-3
4/2 0730	130.88	125.85	5.03	5.47 21.2°C	URSA-5D-4
4/9 0817	124.70	119.70	5.00	5.45 21.6°C	URSA-5D-5
4/16 1804	118.46	113.47	4.99	5.46 22.5°C	URSA-5D-6
4/23 1814	112.13	107.15	4.98	5.46 22.0°C	URSA-5D-7
4/30 1727	105.64	100.64	5.00	5.45 21.8°C	URSA-5D-8

URSA-6A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 911	150.80	145.79	5.01	5.53 20.1°C	URSA-6A-1
3/19 0825	144.51	139.50	5.01	5.57 18.6°C	URSA-6A-2
3/26 0757	138.31	133.30	5.01	5.54 19.1°C	URSA-6A-3
4/2 0731	132.11	127.09	5.02	5.54 21.2°C	URSA-6A-4
4/9 0818	126.17	121.20	4.97	5.57 21.6°C	URSA-6A-5
4/16 1805	120.19	115.18	5.01	5.55 22.5°C	URSA-6A-6
4/23 1815	114.10	109.12	4.98	5.65 22.0°C	URSA-6A-7
4/30 1728	107.83	102.85	4.98	5.83 21.8°C	URSA-6A-8

URSA-6B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 913	148.93	143.93	5.00	5.53 20.1°C	URSA-6B-1
3/19 0826	141.06	136.05	5.01	5.56 18.6°C	URSA-6B-2
3/26 0758	134.06	129.05	5.01	5.52 19.1°C	URSA-6B-3
4/2 0733	126.89	121.87	5.02	5.55 21.2°C	URSA-6B-4
4/9 0820	120.20	115.16	5.04	5.55 21.6°C	URSA-6B-5
4/16 1806	112.99	108.01	4.98	5.57 22.5°C	URSA-6B-6
4/23 1816	105.55	100.56	4.99	5.69 22.0°C	URSA-6B-7
4/30 1729	98.10	93.14	4.96	5.76 21.8°C	URSA-6B-8

URSA-6C

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 914	149.83	144.81	5.02	5.60 20.1°C	URSA-6C-1
3/19 0827	142.68	137.69	4.99	5.70 18.6°C	URSA-6C-2
3/26 0759	135.85	130.87	4.98	5.14 19.1°C	URSA-6C-3
4/2 0734	129.11	124.09	5.02	6.52 21.2°C	URSA-6C-4
4/9 0821	122.79	117.77	5.02	6.57 21.6°C	URSA-6C-5
4/16 1807	116.28	111.32	4.96	6.71 22.5°C	URSA-6C-6
4/23 1817	109.58	104.59	4.99	7.68 22.0°C	URSA-6C-7
4/30 1730	102.23	97.25	4.98	6.07 21.8°C	URSA-6C-8



URSA-6D

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 915	150.69	145.71	4.98	5.57 20.1°C	URSA-6D-1
3/19 0829	144.21	139.21	5.00	5.70 18.6°C	URSA-6D-2
3/26 0800	130.87 137.10	132.89	5.01	6.08 19.1°C	URSA-6D-3
4/2 0735	131.56	126.56	5.00	6.52 21.3°C	URSA-6D-4
4/9 0822	125.47	120.47	5.00	6.64 21.6°C	URSA-6D-5
4/16 1808	119.36	114.38	4.98	6.76 22.5°C	URSA-6D-6
4/23 1818	113.01	108.04	4.97	6.73 22.0°C	URSA-6D-7
4/30 1731	106.68	101.68	5.00	6.71 21.7°C	URSA-6D-8

URSA-7A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 917	151.00	146.00	5.00	5.65 20.2°C	URSA-7A-1
3/19 0830	144.72	139.73	4.99	5.76 18.6°C	URSA-7A-2
3/26 0803	138.51	133.51	5.00	6.07 19.1°C	URSA-7A-3
4/2 0737	132.25	127.26	4.99	6.46 21.3°C	URSA-7A-4
4/9 0824	126.13	121.13	5.00	6.65 21.6°C	URSA-7A-5
4/16 1809	119.78	114.81	4.97	6.80 22.5°C	URSA-7A-6
4/23 1819	113.49	108.49	4.95	6.75 22.0°C	URSA-7A-7
4/30 1732	106.72	101.81	4.91	6.80 21.7°C	URSA-7A-8

URSA-7B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 918	150.98	146.02	4.96	5.63 20.2°C	URSA-7B-1
3/19 0831	144.65	139.67	4.98	5.72 18.7°C	URSA-7B-2
3/26 0804	138.35	133.39	4.96	6.13 19.1°C	URSA-7B-3
4/2 0738	132.04	127.04	5.00	6.48 21.3°C	URSA-7B-4
4/9 0825	126.00	121.02	4.98	6.65 21.6°C	URSA-7B-5
4/16 1810	119.82	114.84	4.98	6.77 22.5°C	URSA-7B-6
4/23 1820	113.63	108.66	4.97	6.75 22.0°C	URSA-7B-7
4/30 1733	107.23	102.24	4.99	6.82 21.7°C	URSA-7B-8

URSA-7C

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 919	150.54	145.54	5.00	5.67 20.2°C	URSA-7C-1
3/19 0832	144.06	139.07	4.99	5.83 18.7°C	URSA-7C-2
3/26 0805	137.55	132.56	4.99	6.13 19.2°C	URSA-7C-3
4/2 0739	131.04	126.04	5.00	6.42 21.3°C	URSA-7C-4
4/9 0826	124.83	119.84	4.99	6.58 21.6°C	URSA-7C-5
4/16 1811	118.49	113.49	5.00	6.70 22.5°C	URSA-7C-6
4/23 1821	111.94	106.95	4.99	6.70 22.0°C	URSA-7C-7
4/30 1734	104.93	99.94	4.99	6.85 21.7°C	URSA-7C-8

URSA-7D

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/01 0920	150.52	145.53	4.99	5.68 20.2°C	URSA-7D-1
3/19 0834	144.11	139.06	5.05	5.87 18.7°C	URSA-7D-2
3/26 0806	137.91	132.92	4.99	6.07 19.2°C	URSA-7D-3
4/2 0740	131.71	126.71	5.00	6.40 21.3°C	URSA-7D-4
4/9 0827	125.69	120.69	5.00	6.57 21.6°C	URSA-7D-5
4/16 1812	119.45	114.57	4.88	6.72 22.5°C	URSA-7D-6
4/23 1822	113.47	108.49	4.98	6.75 22.0°C	URSA-7D-7
4/30 1735	107.16	102.15	5.01	6.84 21.7°C	URSA-7D-8

3/5/01 JP  
12.53 hr  
1

The U matry solutions remaining in the 500 ml polycarbonate bottles were transferred to 30 ml PP bottles and labeled accordingly as shown below.

label	Sample labels for Cl <sup>-</sup> analysis
$1.5 \times 10^{-5}$ M U	UMA-5E
$1.0 \times 10^{-5}$ M U	UMA-5F
$3.33 \times 10^{-6}$ M U	UMA-6E
$1.0 \times 10^{-6}$ M U	UMA-6F
$3.33 \times 10^{-7}$ M U	UMA-7E
$1.0 \times 10^{-7}$ M U	UMA-7F

About 5 ml of each U matry solw was transferred to 15 ml PP bottles for Cl<sup>-</sup> analysis. These samples were labeled as shown above.

4/3/01 JP

### Preparation of QA standards for uranophane solubility experiments.

Obj - prepare a series of standards containing Ca, Si, Cl, Na, & U with concentrations similar to those of solution sampled in the uranophane solubility experiments. These will be used for QA purposes.

### Method - Dilution of certified standard solutions.

#### Materials

- Glassware & plasticware as needed
- Calcium standard, 1000 ppm, Spex Cert. prep (lot # 7-114CA)
- Silicon standard, 1000 ppm, Spex Cert. prep (lot # 7-136SI)
- Uranium standard, 1000 ppm, Spex Cert. prep (lot # 7-141U)
- Sodium standard, 1000 ppm, Spex Cert. prep (lot # 7-87NA)
- HCl - lot 956110
- 125 ml PP bottles
- 50 ml volumetric flasks
- pipets, pipettors, & plastic pipettor tips as needed
- DI H<sub>2</sub>O.
- 15 ml PP bottles

### Procedure

① Standards will be prepared with total volume of 50 ml. The following solution volumes were prepared for use in making the QA standards.

1000 ppm Ca solution - used Ca std as is.

500 ppm Si solution - add 25 ml of Si std to a 50 ml volumetric flask + make up to mark with H<sub>2</sub>O.

50 ppm Na solution - add 5 ml of Na std to 100 ml volumetric flask + make up to mark with H<sub>2</sub>O.

10 ppm U solution - add 10 ml of U std to 1000 ml volumetric flask + make up to mark with H<sub>2</sub>O.

1 ppm U solution - add 1 ml of U std to 1000 ml volumetric flask + make up to mark with H<sub>2</sub>O.

0.2 M HCl solution - add 16.4 ml concentrated HCl to 1000 ml volumetric flask + make up to mark with H<sub>2</sub>O.

② Take 10 50 ml volumetric flasks and label as follows.

URSA-4A-1

URSA-4A-2

URSA-4A-3

URSA-4A-4

URSA-4A-5

URSA-4B-1

URSA-4B-2

URSA-4B-3

URSA-4B-4

URSA-4B-5

③ Using the table below add the volumes of solution specified to the flasks specified.

Std label	Ca soln 1000ppm	Si soln 500ppm	Na soln 50ppm	U soln 10ppm	0.2M HCl soln
URSA-4A-1	20 ml	3 ml	2 ml	5 ml	5 ml
URSA-4A-2	22 ml	2.8 ml	0.2 ml	0.5 ml	5 ml
URSA-4A-3	19 ml	3.2 ml	0.5 ml	2.5 ml	5 ml
URSA-4A-4	21 ml	3.4 ml	0.1 ml	1.0 ml	5 ml
URSA-4A-5	22.5 ml	2.9 ml	1 ml	10 ml	5 ml
				U soln 1ppm	
URSA-4B-1	20 ml	3.3 ml	5 ml	0.5 ml	5 ml
URSA-4B-2	20.5 ml	3.0 ml	0.3 ml	0.1 ml	5 ml
URSA-4B-3	19.5 ml	2.8 ml	0.2 ml	2.5 ml	5 ml
URSA-4B-4	22 ml	3.1 ml	0.5 ml	0.25 ml	5 ml
URSA-4B-5	23 ml	3.5 ml	1.0 ml	1.0 ml	5 ml

After adding the above volumes make up to mark with DI H<sub>2</sub>O.

④ Transfer solution in the 50 ml volumetric flasks to 125 ml PP bottles with identical labels to the volumetric flasks.

⑤ The table below shows the concentration of elements in each standard.

Std label	Ca ppm	Si ppm	Na ppm	U ppm	Cl ppm
URSA-4A-1	400	30	2.0	1.0	709
URSA-4A-2	440	28	0.2	0.1	709
URSA-4A-3	380	32	0.5	0.5	709
URSA-4A-4	420	34	0.1	0.2	709
URSA-4A-5	450	29	1.0	2.0	709
URSA-4B-1	400	33	5.0	0.01	709
URSA-4B-2	410	30	0.3	0.002	709
URSA-4B-3	390	28	0.2	0.05	709
URSA-4B-4	440	31	0.5	0.005	709
URSA-4B-5	460	35	1.0	0.02	709

⑥ Transfer 5 ml of each standard to 15 ml labeled polypropylene bottles for ICP analysis of cations. Label the bottles with the same label as the standard container.

4/10/01 JF

Preparation of QA standards for determining  
chemistry of synthetic macrophage

Obj - prepare a series of standards  
with U, Ca, Si, & Na concentrations  
similar to solution prepared by  
dissolving 0.1 g synthetic macrophage  
in 100 ml of 0.1 M HCl.

Method - Dilute of certified standard  
solutions.

#### Materials

- conc HCl - lot 965110
- glassware & plasticware as needed
- Calcium standard, 1000 ppm, Spex Certiprep (lot # 7-11424)
- Silicon standard, 1000 ppm, Spex Certiprep (lot # 7-13651)
- Uranium standard, 1000 ppm, Spex Certiprep (lot # 7-1411)
- Sodium standard, 1000 ppm, Spex Certiprep (lot # 7-87Na)
- DI H<sub>2</sub>O
- 100 ml volumetric flasks
- 15 ml PP bottles
- 125 ml PP bottles

#### Procedure

① Standards will be prepared with total  
volume of 100 ml. The following solution  
and volumes were prepared for use in  
making the QA standards

100 ppm Na std soln - add 10 ml of Na  
std to a 100 ml volumetric flask &  
make up to mark with DI H<sub>2</sub>O

1.0 M HCl soln - add 8.2 ml conc HCl to 100 ml  
volumetric flask & make up to mark with DI H<sub>2</sub>O.

② Take 3 100 ml volumetric flasks and  
label as follows.

U56-4

U56-5

U56-6

③ Using the table below add the volume of  
solution specified to the flask specified

Std label	Ca soln 1000ppm	Si soln 1000ppm	Na soln 100ppm	U soln 1000ppm	1.0 M HCl soln
U56-4	5.0	6.5	0.1	50	10
U56-5	4.5	6.0	0.5	55	10
U56-6	4.0	7.0	1.0	60	10

④ After adding the above volume make up  
to mark with DI H<sub>2</sub>O

⑤ Transfer solns in 100 ml volumetric flasks to 125 ml PP bottles with identical labels to the flasks.

⑥ The table below shows the contents of elements in each standard.

Std label	Ca(ppm)	Si(ppm)	Na(ppm)	U(ppm)
US6-4	50	65	0.1	500
US6-5	45	60	0.5	550
US6-6	40	70	1.0	600

⑦ Transfer 5 ml aliquot of each standard to a 15 ml PP bottle and label for ICP analysis of cations.

4/10/01 JP

Chemical analysis of synthetic uranophane by ICP.

5 ml  
Two aliquots of samples US6-1, US6-2, and US6-3 (see p 106-107) were transferred to 15 ml PP bottles for ICP analysis in Div 01. The 15 ml PP bottles were labeled as follows.

US6-1A

US6-1B

US6-2A

US6-2B

US6-3A

US6-3B

These samples along with QA standards US6-4, US6-5, and US6-6 were taken to Div 01 for ICP analysis.

The Div 01 Sample List / Chain of Custody Form for these samples is shown on the following page.



Client Name/Address

Jim Pinkryl  
CWRRA/Div 20  
Bldg 57

SAMPLE LIST/CHAIN OF CUSTODY

Southwest Research Institute  
Chemistry and Chemical Engineering Division  
8220 Culebra Road  
San Antonio, Texas 78239-5166

Requested Turnaround:

☐ 1 Week  
☒ 2 Weeks (Normal)  
☐ 3 Weeks  
☐ Other

Client Purchase Order/Other ID

Site/Zone ID

Sample ID

Sample Collection Date (mm/dd/yy)

Sample Collection Time (mm/dd/yy)

Matrix Type

Sample Type

# of Containers

ICP of Major Cations including Si and U

\*Note for calibration purposes. These samples contain the following cations:  
U - 500-600 ppm  
Ca - 40-50 ppm  
Si - 60-70 ppm  
Na - 10-10 ppm  
All other cations should be at or below 10 ppm.

REMARKS

U56-1A	4/10/01	1500 hrs	W	DM	1	X		
U56-1B					1	X		
U56-2A					1	X		
U56-2B					1	X		
U56-3A					1	X		
U56-3B					1	X		
U56-4					1	X		
U56-5					1	X		
U56-6					1	X		

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

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

Relinquished by Sampler (Signature): *James D. Bell*

Received by (Signature): *Bradley Marking*

Relinquished by (Signature):

Comments:

SWRI Project No.

20.0402.561

Relinquished by SWRI (Signature): *Mike Romano*

Samples Disposed by:

SWRI Contact: *Mike Romano*

Preservation:  
a = HCl to pH <2  
b = HNO<sub>3</sub> to pH <2  
c = H<sub>2</sub>SO<sub>4</sub> to pH <2  
d = NaOH to pH >12  
e = Other (Specify)

Project is Nuclear Safety related 10 CFR 50 Part 21 Appendix B Question Per: Jim Pinkryl x5667

SWRI Project No. 20.0402.561

Relinquished by SWRI (Signature): *Mike Romano*

Samples Disposed by:

Date/TIME: 04-11-01 @ 1120

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

4/10/01 JP

The following samples were taken to Div 01 for analysis by ion chromatography.

- UMA-5E
- UMA-5F
- UMA-6E
- UMA-6F
- UMA-7E
- UMA-7F

These samples are aliquots of matrix solution prepared for the ionophore solubility experiment (see p 131).

The Div 01 Sample List/Chain of Custody Form for these samples is shown on the next page.

4/24/01 JP 1000 hrs

Uranophane solubility samples were taken to Div 01 for ICP analysis of major cations including U and Si.

The samples sent to Div 01 are listed on the Div 01 Sample List / Chain of Custody Forms which are shown on the following pages. Samples include urine/plasma solubility samples taken over the last 7 week period (see p 128-131), matrix solutions (see p 120) and standards (see p 132-135).

Client Name/Address Jim Pukyl CNWRA/Div 20 BIO 57		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166										Requested Turnaround: <input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> 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 of major cations including U and Si						Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Other (Specify)						
URSA-4A-1	4/3/01	1900	W	DM	1	✓						Project is nuclear safety related 10 CFR 50 Part 21						
URSA-4A-2					1	✓						Appendix B						
URSA-4A-3					1	✓												
URSA-4A-4					1	✓												
URSA-4A-5					1	✓						Quote POC						
URSA-4B-1					1	✓						Jim Pukyl x5667						
URSA-4B-2					1	✓												
URSA-4B-3					1	✓												
URSA-4B-4					1	✓												
URSA-4B-5					1	✓												
Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water							Relinquished by (Signature):											
Sample Types: DM - Dissolved Metals; ER - Equipment Rinseate; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; ES - Environmental Samples; FD - Field Duplicate							Received by (Signature):											
Relinquished by Sampler (Signature):							Relinquished by (Signature):											
Received by (Signature):							Comments:											

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

Client Name/Address Jim Pukyl CNWRA/Div 20 BIO 57		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166										Requested Turnaround: <input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> 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 of major cations including U and Si						Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Other (Specify)						
URSA-5C-1	3/12/01		W	DM	1	✓						Project is nuclear safety related 10 CFR 50 Part 21						
URSA-5C-2	3/13/01				1	✓						Appendix B						
URSA-5C-3	3/26/01				1	✓												
URSA-5C-4	4/2/01				1	✓												
URSA-5C-5	4/9/01				1	✓						Quote POC						
URSA-5C-6	4/11/01				1	✓						Jim Pukyl x5667						
URSA-5C-7	4/23/01				1	✓												
URSA-5D-6	4/11/01				1	✓												
URSA-5D-7	4/23/01				1	✓												
Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water							Relinquished by (Signature):											
Sample Types: DM - Dissolved Metals; ER - Equipment Rinseate; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; ES - Environmental Samples; FD - Field Duplicate							Received by (Signature):											
Relinquished by Sampler (Signature):							Relinquished by (Signature):											
Received by (Signature):							Comments:											

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

Client Name/Address: <b>Jim Prikeyl</b> <b>CNWRADiv 20</b> <b>BIDg 57</b>	<h2>SAMPLE LIST/CHAIN OF CUSTODY</h2> <p><b>Southwest Research Institute</b>          Chemistry and Chemical Engineering Division          8220 Culbreth Road          San Antonio, Texas 78238-5166</p>	Requested Turnaround: <input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> Other: _____
Client Purchase Order/Other ID:	Site/Zone ID:	
Analyzes Requested		SWRI Contact:
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time (mm/dd/yyyy)
Matrix Type	Sample Type	# of Containers
K.P.S. major extract including standards		
URSA-GC-1	3/12/01	W DM 1 ✓
URSA-GC-2	3/19/01	✓
URSA-GC-3	3/26/01	✓
URSA-GC-4	4/2/01	✓
URSA-GC-5	4/4/01	✓
URSA-GC-6	4/16/01	✓
URSA-GC-7	4/23/01	✓
URSA-GD-6	4/16/01	✓
URSA-GD-7	4/23/01	✓
Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water Sample Types: DM - Dissolved Metals; EM - Equipment Rinseate; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; ES - Environmental Samples; PD - Field Duplicate		
Relinquished by (Signature): 		
Received by (Signature): 		
Relinquished by Sampler (Signature): 		
Comments: Project is nuclear safety related UCFR50 Part 21 Appendix B Quote Poc Jim Prikeyl x 566		



Client Name/Address: Jim Pirkyl  
CNWRA/Div 20  
BIO 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

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP of major cations including U and Si
URSA-7A-1	3/12/01		W	DM	1	✓
URSA-7A-2	3/15/01				1	✓
URSA-7A-3	3/24/01				1	✓
URSA-7A-4	4/12/01				1	✓
URSA-7A-5	4/16/01				1	✓
URSA-7A-6	4/16/01				1	✓
URSA-7A-7	4/23/01				1	✓
URSA-7B-6	4/11/01				1	✓
URSA-7B-7	4/23/01				1	✓

Relinquished by (Signature):  
Received by (Signature):  
Relinquished by (Signature):  
Comments:

Preservation:  
a = HCl to pH <2  
b = HNO<sub>3</sub> to pH <2  
c = H<sub>2</sub>SO<sub>4</sub> to pH <2  
d = NaOH to pH >12  
e = Other (Specify)

Project is nuclear  
sample collected  
10 CFR 50 Part 21  
Appendix B  
Question POC  
- Jim Pirkyl x 5067

Div 01 COC Form 01-01-001, Rev 1/99

Client Name/Address: Jim Pirkyl  
CNWRA/Div 20  
BIO 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

Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time (mm/dd/yy)	Matrix Type	Sample Type	# of Containers	ICP of major cations including U and Si
URSA-7C-1	3/12/01		W	DM	1	✓
URSA-7C-2	3/15/01				1	✓
URSA-7C-3	3/24/01				1	✓
URSA-7C-4	4/12/01				1	✓
URSA-7C-5	4/16/01				1	✓
URSA-7C-6	4/16/01				1	✓
URSA-7C-7	4/23/01				1	✓
URSA-7D-6	4/11/01				1	✓
URSA-7D-7	4/23/01				1	✓

Relinquished by (Signature):  
Received by (Signature):  
Relinquished by (Signature):  
Comments:

Preservation:  
a = HCl to pH <2  
b = HNO<sub>3</sub> to pH <2  
c = H<sub>2</sub>SO<sub>4</sub> to pH <2  
d = NaOH to pH >12  
e = Other (Specify)

Project is nuclear  
sample collected  
10 CFR 50 Part 21  
Appendix B  
Question POC  
- Jim Pirkyl x 5067

Div 01 COC Form 01-01-001, Rev 1/99



Page 7 of 7

Results of chemical analysis of synthesized uranophane & synthesized uranophane (see p139-140)

5/4/01 JP

Results of chemical analysis of synthesized uranophane and QA samples by ICP in Div 01  
All concentrations reported as mg/L

Sample ID	Aluminum	Antimony	Arsenic	Barium	Beryllium	Bismuth	Boron	Cadmium	Calcium	Chromium	Cobalt	Copper	Iron	Lanthanum	Lead	Lithium	Magnesium	Manganese	Molybdenum
U56-1A	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	47.9	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.092
U56-1A (Duplicate)	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	48.0	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.099
U56-2A	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	48.0	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.092
U56-2B	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	48.7	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.094
U56-3A	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	47.0	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.095
U56-3B	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	47.6	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.099
U56-4	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	51.2	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	0.094
U56-5	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	45.6	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	<0.025
U56-6	<0.5	<0.1	<0.05	<0.025	<0.025	<0.05	<0.25	<0.025	40.7	<0.05	<0.025	<0.1	<1.25	<0.05	<0.05	<0.025	<1	<0.025	<0.025
Reporting limit	0.5	0.1	0.05	0.025	0.025	0.05	0.25	0.025	0.5	0.05	0.025	0.1	1.25	0.05	0.05	0.025	1	0.025	0.025

Nickel	Palladium	Phosphorus	Potassium	Selenium	Silicon	Silver	Sodium	Strontium	Sulfur	Thallium	Thorium	Tin	Titanium	Tungsten	Uranium	Vanadium	Yttrium	Zinc	Zirconium
<0.05	<0.05	<0.25	<0.5	<0.05	70.8	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	537	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	71.2	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	542	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	70.8	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	542	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	71.0	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	540	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	69.5	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	541	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	66.7	<0.05	<0.5	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	528	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	62.7	<0.05	0.637	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	471	<0.05	<0.025	<0.025	<0.125
<0.05	<0.05	<0.25	<0.5	<0.05	72.2	<0.05	1.26	<0.025	<0.25	<0.1	<0.25	<0.05	<0.05	<0.1	519	<0.05	<0.025	<0.025	<0.125
0.05	0.05	0.25	0.5	0.05	0.25	0.05	0.5	0.025	0.25	0.1	0.25	0.05	0.05	0.1	0.5	0.05	0.025	0.025	0.125

5/4/01 JP

Results of anion analysis of matrix solutions used in uranophane solubility experiments (see p 141-142)

Results of anion analysis of matrix solutions prepared for uranophane solubility experiments. Analyses were done by ion chromatography in Div 01  
All concentrations reported in mg/L

Sample ID  
UMA-5E  
UMA-5E (Duplicate)  
UMA-5F  
UMA-6E  
UMA-6F  
UMA-7E  
UMA-7F

Reporting limit

Bromide	Chloride	Fluoride	Nitrate-N	Nitrite-N	Phosphate-P	Sulfate
<0.1	690	<0.1	0.497	<0.2	<0.1	0.126
<0.1	694	<0.1	0.503	<0.2	<0.1	0.117
<0.1	692	<0.1	0.367	<0.2	<0.1	<0.1
<0.1	684	<0.1	0.186	<0.2	<0.1	<0.1
<0.1	704	<0.1	0.166	<0.2	<0.1	0.489
<0.1	713	<0.1	0.118	<0.2	<0.1	0.118
<0.1	692	<0.1	0.146	<0.2	<0.1	<0.1
0.1	0.1	0.1	0.1	0.2	0.1	0.1

Results of chemical analysis of unexposed solubility sample, matrix solution, + QA samples are shown on the following page.

Sample ID  
Matrix solutions

URSA-7D-6	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-7D-7	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
<b>QA sàmples</b>							
URSA-4A-1	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4A-2	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4A-3	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4A-4	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4A-5	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4B-1	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4B-2	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4B-3	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4B-4	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1
URSA-4B-5	<0.25	<0.05	<0.025	<0.025	<0.025	<0.05	<0.1

0.25	0.05	0.025	0.025	0.025	0.05	0.1
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<0.025	420	<0.025	<0.025	<0.025	<0.15	<0.025	<0.025	<0.025	<0.25	<0.025	0.196	0.030	<0.05	<0.1
<0.025	428	<0.025	<0.025	<0.025	<0.15	<0.025	<0.025	<0.025	<0.25	<0.025	0.212	0.029	<0.05	<0.1
<0.025	404	<0.025	<0.025	<0.025	<0.15	0.064	0.040	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	469	<0.025	<0.025	<0.025	<0.15	0.080	0.043	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	384	<0.025	<0.025	<0.025	<0.15	0.060	0.036	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	421	<0.025	<0.025	<0.025	<0.15	0.070	0.037	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	455	<0.025	<0.025	<0.025	<0.15	0.077	0.047	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	404	<0.025	<0.025	<0.025	<0.15	0.067	0.037	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	415	<0.025	<0.025	<0.025	<0.15	0.067	0.045	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	395	<0.025	<0.025	<0.025	<0.15	0.068	0.044	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	439	<0.025	<0.025	<0.025	<0.15	0.076	0.039	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
<0.025	461	<0.025	<0.025	<0.025	<0.15	0.080	0.048	<0.025	<0.25	<0.025	<0.025	<0.025	<0.05	<0.1
0.025	0.25	0.025	0.025	0.025	0.15	0.025	0.025	0.025	0.25	0.025	0.025	0.025	0.05	0.1

Potassium	Selenium	Silicon	Silver	Sodium	Strontium	Sulfur	Thallium	Thorium	Tin	Titanium	Tungsten	Uranium	Vanadium	Yttrium	Zinc	Zirconium
<0.5	<0.025	31.3	<0.025	<0.25	0.217	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	2.33	<0.025	<0.025	0.129	<0.025
<0.5	<0.025	31.4	<0.025	<0.25	0.217	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	2.49	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.0	<0.025	<0.25	0.216	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	2.41	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.2	<0.025	<0.25	0.218	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.762	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.4	<0.025	<0.25	0.218	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.778	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.1	<0.025	<0.25	0.220	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.250	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.4	<0.025	<0.25	0.222	0.142	<0.05	<0.05	<0.025	<0.025	<0.05	0.265	<0.025	<0.025	0.175	<0.025
<0.5	<0.025	31.5	<0.025	<0.25	0.221	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0760	<0.025	<0.025	<0.025	0.040
<0.5	<0.025	31.3	<0.025	0.280	0.221	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0778	<0.025	0.025	<0.025	<0.025
<0.5	<0.025	31.3	<0.025	0.220	0.220	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0246	<0.025	<0.025	<0.025	<0.025
<0.5	<0.025	31.5	<0.025	<0.25	0.221	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0244	<0.025	<0.025	0.214	<0.025
6.65	<0.025	31.4	<0.025	2.76	0.210	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.158	<0.025	<0.025	<0.025	<0.025
6.55	<0.025	31.4	<0.025	2.75	0.210	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.159	<0.025	<0.025	<0.025	<0.025
4.51	<0.025	32.1	<0.025	2.86	0.213	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.249	<0.025	<0.025	<0.025	<0.025
4.10	<0.025	33.5	<0.025	2.96	0.219	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	1.44	<0.025	<0.025	<0.025	<0.025
3.81	<0.025	34.3	<0.025	2.96	0.222	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0698	<0.025	<0.025	<0.025	<0.025
5.46	<0.025	34.8	<0.025	3.05	0.223	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.773	<0.025	<0.025	<0.025	<0.025
4.99	<0.025	36.1	<0.025	3.12	0.229	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.455	<0.025	<0.025	<0.025	<0.025
2.06	<0.025	37.0	<0.025	3.13	0.233	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.516	<0.025	<0.025	<0.025	<0.025
2.37	<0.025	36.5	<0.025	3.01	0.231	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.410	<0.025	<0.025	<0.025	<0.025
3.58	<0.025	37.4	<0.025	3.07	0.235	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.501	<0.025	<0.025	<0.025	<0.025
4.73	<0.025	31.5	<0.025	2.70	0.213	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.210	<0.025	<0.025	<0.025	<0.025
4.24	<0.025	32.5	<0.025	2.77	0.215	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.600	<0.025	<0.025	<0.025	<0.025
6.43	<0.025	33.3	<0.025	2.78	0.219	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.278	<0.025	<0.025	<0.025	<0.025
5.74	<0.025	34.8	<0.025	3.02	0.227	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.586	<0.025	<0.025	<0.025	<0.025
5.29	<0.025	35.6	<0.025	3.00	0.228	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.524	<0.025	<0.025	<0.025	<0.025
4.04	<0.025	36.5	<0.025	3.03	0.232	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.201	<0.025	<0.025	<0.025	<0.025
14.8	<0.025	40.7	<0.025	3.42	0.256	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.402	<0.025	<0.025	<0.025	<0.025
3.15	<0.025	38.9	<0.025	3.77	0.251	0.135	<0.05	<0.05	<0.025	<0.025	<0.05	0.197	<0.025	<0.025	<0.025	<0.025
7.45	<0.025	39.7	<0.025	3.42	0.256	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.218	<0.025	<0.025	<0.025	<0.025
7.11	<0.025	31.8	<0.025	2.73	0.215	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.120	<0.025	<0.025	<0.025	<0.025
6.89	<0.025	33.1	<0.025	2.83	0.222	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0800	<0.025	<0.025	<0.025	<0.025
8.04	<0.025	34.4	<0.025	3.33	0.231	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.183	<0.025	<0.025	<0.025	<0.025
8.08	<0.025	34.5	<0.025	3.32	0.231	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.183	<0.025	<0.025	<0.025	<0.025
13.7	<0.025	36.0	<0.025	3.44	0.237	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.320	<0.025	<0.025	<0.025	<0.025
19.9	<0.025	37.6	<0.025	3.57	0.241	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.329	<0.025	<0.025	<0.025	<0.025
23.9	<0.025	38.5	<0.025	3.60	0.243	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.705	<0.025	<0.025	<0.025	<0.025
26.7	<0.025	41.1	<0.025	3.89	0.254	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	4.85	<0.025	<0.025	<0.025	<0.025
9.33	<0.025	38.7	<0.025	3.50	0.239	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	1.85	<0.025	<0.025	<0.025	<0.025
10.9	<0.025	39.1	<0.025	3.46	0.240	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.322	<0.025	<0.025	<0.025	<0.025
5.7	<0.025	35.1	<0.025	3.01	0.239	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.264	<0.025	<0.025	<0.025	<0.025
7.01	<0.025	32.8	<0.025	3.23	0.222	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.777	<0.025	<0.025	<0.025	<0.025
5.61	<0.025	33.8	<0.025	3.32	0.225	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.212	<0.025	<0.025	<0.025	<0.025
13.4	<0.025	35.2	<0.025	3.43	0.228	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.210	<0.025	<0.025	<0.025	<0.025
12.6	<0.025	37.0	<0.025	3.44	0.233	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.279	<0.025	<0.025	<0.025	<0.025
11.0	<0.025	38.1	<0.025	3.41	0.234	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	2.11	<0.025	<0.025	<0.025	<0.025
10.9	<0.025	38.7	<0.025	3.58	0.239	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.207	<0.025	<0.025	<0.025	<0.025
12.1	<0.025	37.9	<0.025	3.49	0.238	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.245	<0.025	<0.025	<0.025	<0.025
9.85	<0.025	38.8	<0.025	3.53	0.243	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.220	<0.025	<0.025	<0.025	<0.025
4.24	<0.025	31.8	<0.025	3.20	0.220	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.0667	<0.025	<0.025	<0.025	<0.025
9.05	<0.025	32.4	<0.025	3.26	0.220	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.113	<0.025	<0.025	<0.025	<0.025
6.76	<0.025	34.1	<0.025	3.30	0.229	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.557	<0.025	<0.025	<0.025	<0.025
9.24	<0.025	35.4	<0.025	3.46	0.234	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.184	<0.025	<0.025	<0.025	<0.025
10.6	<0.025	37.2	<0.025	3.30	0.235	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.454	<0.025	<0.025	<0.025	<0.025
10.5	<0.025	37.1	<0.025	3.32	0.236	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.451	<0.025	<0.025	<0.025	<0.025
8.24	<0.025	38.0	<0.025	3.30	0.236	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.504	<0.025	<0.025	<0.025	<0.025
9.24	<0.025	39.0	<0.025	3.44	0.242	<0.1	<0.05	<0.05	<0.025	<0.025	<0.05	0.646	<0.025	<0.025	<0.025	<0.025

Sample ID  
Matrix solutions  
UMA-5C  
UMA-5D  
UMA-5D (duplicate)  
UMA-6A  
UMA-6B  
UMA-6C  
UMA-6D  
UMA-7A  
UMA-7B  
UMA-7C  
UMA-7D

Solubility Samples  
URSA-5C-1  
URSA-5C-1 (duplicate)  
URSA-5C-2  
URSA-5C-3  
URSA-5C-4  
URSA-5C-5  
URSA-5C-6  
URSA-5C-7  
URSA-5D-6  
URSA-5D-7

URSA-6A-1  
URSA-6A-2  
URSA-6A-3  
URSA-6A-4  
URSA-6A-5  
URSA-6A-6  
URSA-6A-7  
URSA-6B-6  
URSA-6B-7

URSA-6C-1  
URSA-6C-2  
URSA-6C-3  
URSA-6C-3 (duplicate)  
URSA-6C-4  
URSA-6C-5  
URSA-6C-6  
URSA-6C-7  
URSA-6D-6  
URSA-6D-7

URSA-7A-1  
URSA-7A-2  
URSA-7A-3  
URSA-7A-4  
URSA-7A-5  
URSA-7A-6  
URSA-7A-7  
URSA-7B-6  
URSA

7/16/01 JP

Determination of activities and reaction quotients from measured solution chemistry for the uranophane solubility experiments

Data & chemical analyses from the uranophane solubility experiments were entered into an EXCEL spreadsheet entitled uran-sol-2001-results.

Data was taken from entries made on pages 126-135. Chemical analyses were taken from entries on pages 154-156.

Chemical analyses for Ca, Si, <sup>Na</sup> and U were corrected based on QA standard analyses.

The log activities of Ca<sup>++</sup>, SiO<sub>2</sub>, and UO<sub>2</sub><sup>++</sup> in experimental solution were determined using the EQ3 speciation code (EQ3-EVT.2b). The EQ3 input file for sample URSA-5C-1 is shown on the following page. Input for the species were taken from the uran-sol-2001-results spreadsheet.

A hard copy of uran-sol-2001-results is shown on p 160-161.

(cont on p 162)

EQ3NR input file name= ursa5c1.3i  
Description= "uranophane solubility experiments"

Version level= 7.2  
Created 06/28/99 Creator= J.D. Prikryl

Purpose: to determine saturation states of minerals for uranophane solubility experiments.

Temperature (C)	25.0	Density(gm/cm3)	1.00000
Total Dissolved Salts		mg/kg	mg/l *not used

-----  
Electrical Balancing on Cl-performed | code selects | not

SPECIES TYPE	BASIS SWITCH/CONSTRAINT	CONC/ETC	UNITS OR
redox		-0.700	LogfO2
H+		5.48	pH
Cl-		706.58	mg/L
Ca++		385.06	mg/L
Na+		1.896	mg/L
SiO2(aq)		66.334	mg/L
NO3-		0.370	mg/L
UO2++		0.1508	mg/L
HCO3-fugacity	CO2(g)	-3.5	Log

-----  
Input Solid Solutions

-----  

none			
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-----  

SUPPRESSED SPECIES	(suppress, replace, augmentk, augmentg)	value
none		

OPTIONS

- SOLID SOLUTIONS -  
\* ignore solid solutions



Sample  
Error  
Average  
Median


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7/16/01 JP  
(cont from p 158)

The log activities of  $\text{Ca}^{++}$ ,  $\text{SiO}_2$ ,  $\text{UO}_2^{++}$ ,  $\text{H}^+$ ,  $(\text{Ca}^{++})/(\text{H}^+)^2$  and  $(\text{UO}_2^{++})/(\text{H}^+)^2$  determined by EQ3 were entered into the uwm-sol-2001 results spreadsheet. The reaction quotient (Q) for uranophane dissolution was then calculated using the following formula

$$Q = (10^{\log[\text{Ca}^{++}]}) (10^{\log[\text{UO}_2^{++}]})^2 (10^{\log[\text{SiO}_2]})^2 (10^{\log[\text{H}^+]})^{-6}$$

3/4/02 JP

Test and development of new method to synthesize uranophane for solubility and coprecipitation experiments

The new method is based on modification made to a procedure for uranophane synthesis by Vochten et al. 1997 which appears in Canadian Mineralogist Vol 35 pp 735-741

In this procedure boltwoodite  $[\text{K}(\text{UO}_2)(\text{SiO}_3\text{OH}) \cdot \text{H}_2\text{O}]$  is initially synthesized and then converted to uranophane by exchanging  $\text{Ca}^{++}$  for  $\text{K}^+$  in the uranyl silicate crystal structure.

### Method

- ① 1.166 g of uranyl nitrate hexahydrate  $[\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$  (lot no 8140 KAP) and 0.166 g of KCl (lot no 006242) was dissolved in 25 ml DI water in a 50 ml plastic beaker.
- ② The pH of the solution was adjusted to ~11.5 with 1M KOH (lot no 015356). A K-U gel formed with the addition of 1M KOH.
- ③ The solution was transferred to a 125 ml teflon liner which was placed in a Parr reaction vessel.

④ About 4 grams of natural quartz fragments which had been ultrasonically cleaned was added to the sol. The quartz fragments were large enough to easily remove from the product.

⑤ Placed the Parr vessel in a Blue M oven at  $185^{\circ}\text{C}$  for about 4 days.

⑥ After 4 days the vessel was removed from the oven and allowed to cool. Quartz fragments were removed and the solution was centrifuged to separate the solid product.

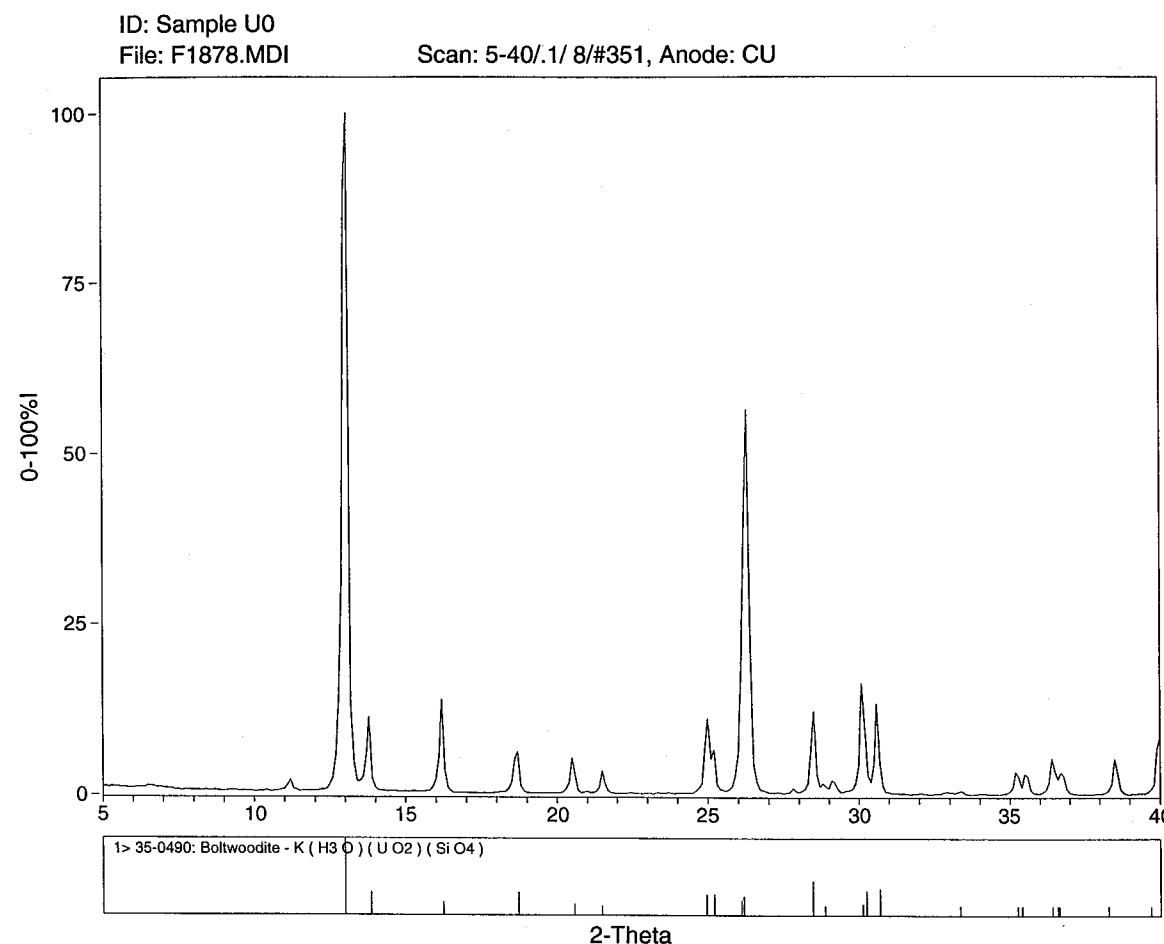
⑦ The solid consisted of pale yellow waxy silicate mineral and a white gel-like substance.

⑧ The gel-like substance was removed by washing the solid 3 times with 5% acetic acid (centrifuge + decant supernatant).

⑨ The waxy solid was vacuum filtered using a  $0.45\ \mu\text{m}$  membrane and washed several times with DI water to remove the acid.

⑩ The solid was allowed to air dry and then a portion was taken and crushed for XRD analysis (Sample U0). The XRD pattern is shown on the following page and indicates that boltwoodite was produced.

3/11/02  
JP Remaining sample was placed in plastic container and labeled Boltwoodite - 1.



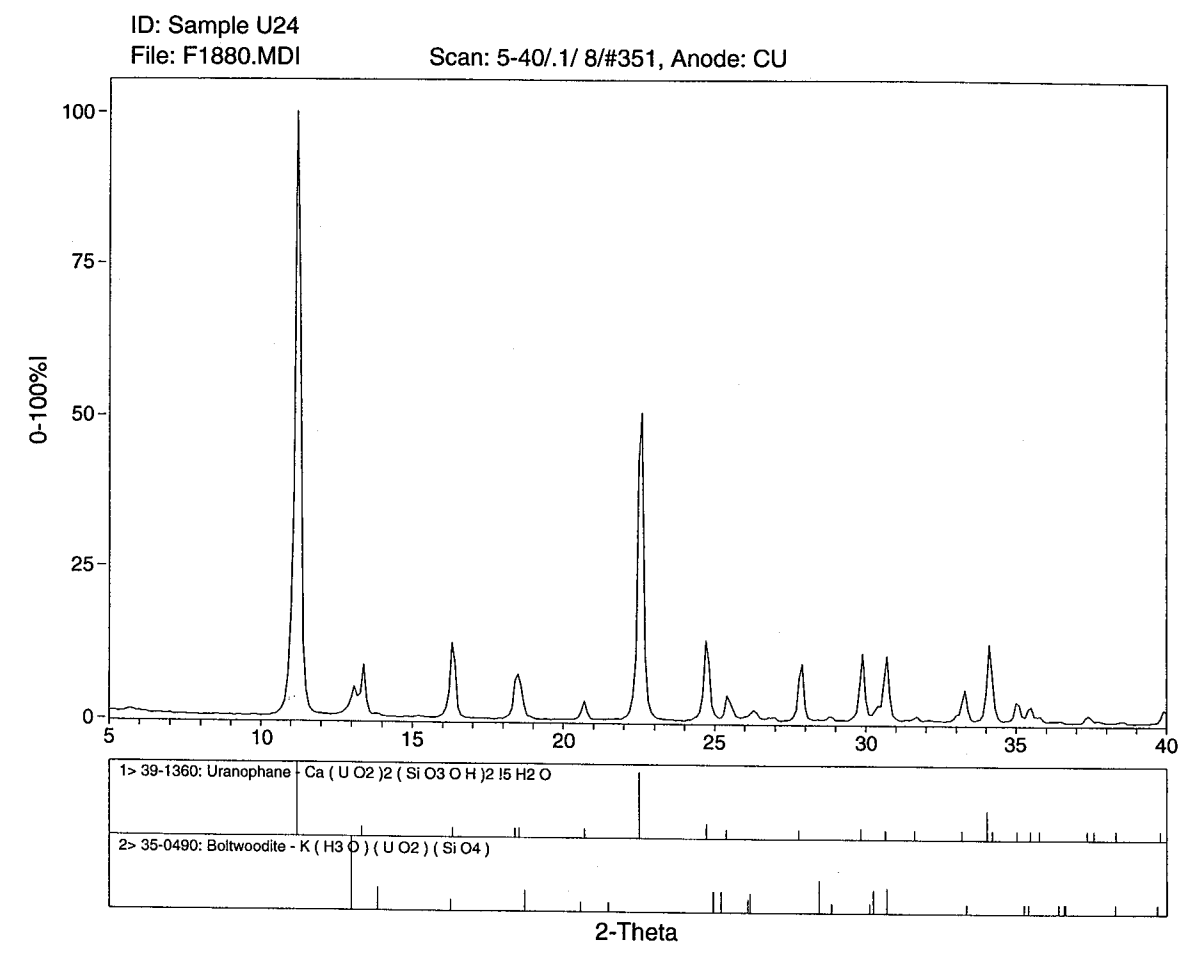
3/11/02  
JP ⑪ To make monophase the synthetic boltwoodite will be placed in the 125 ml Teflon liner and 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  solution will be added. The solution will then be heated to  $185^{\circ}\text{C}$  in the Blue M oven. It is unknown how long it will take to completely convert the boltwoodite to monophase. Therefore, the solution will be removed from the oven

at 24 hr intervals, the vessel will be cooled and a portion of the solid will be sampled after decanting the solution. The removed solid will be vacuum filtered through a 0.45  $\mu$ m membrane, washed with DI water, air dried, and stored for future analysis. 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  will be added to the teflon liner and the vessel will be placed back in the oven for 24 hr at 185°C. This will be repeated for 4 days.

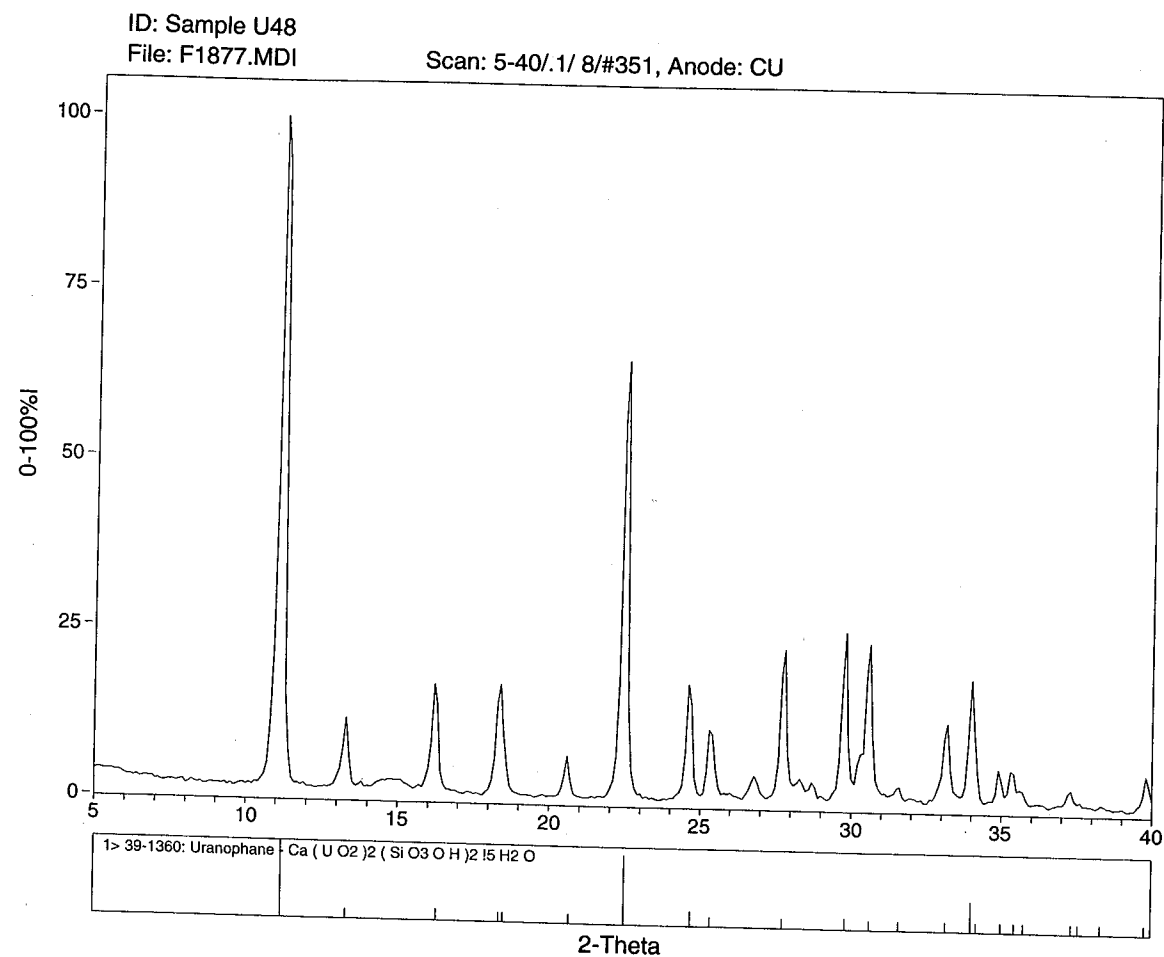
3/15/02 JP (12) After 4 days 4 solid samples had been taken. Samples were labeled as follows.

- U24 - sample taken at 24 hours
- U48 - " " " 48 hours
- U72 - " " " 72 hours
- U96 - " " " 96 hours

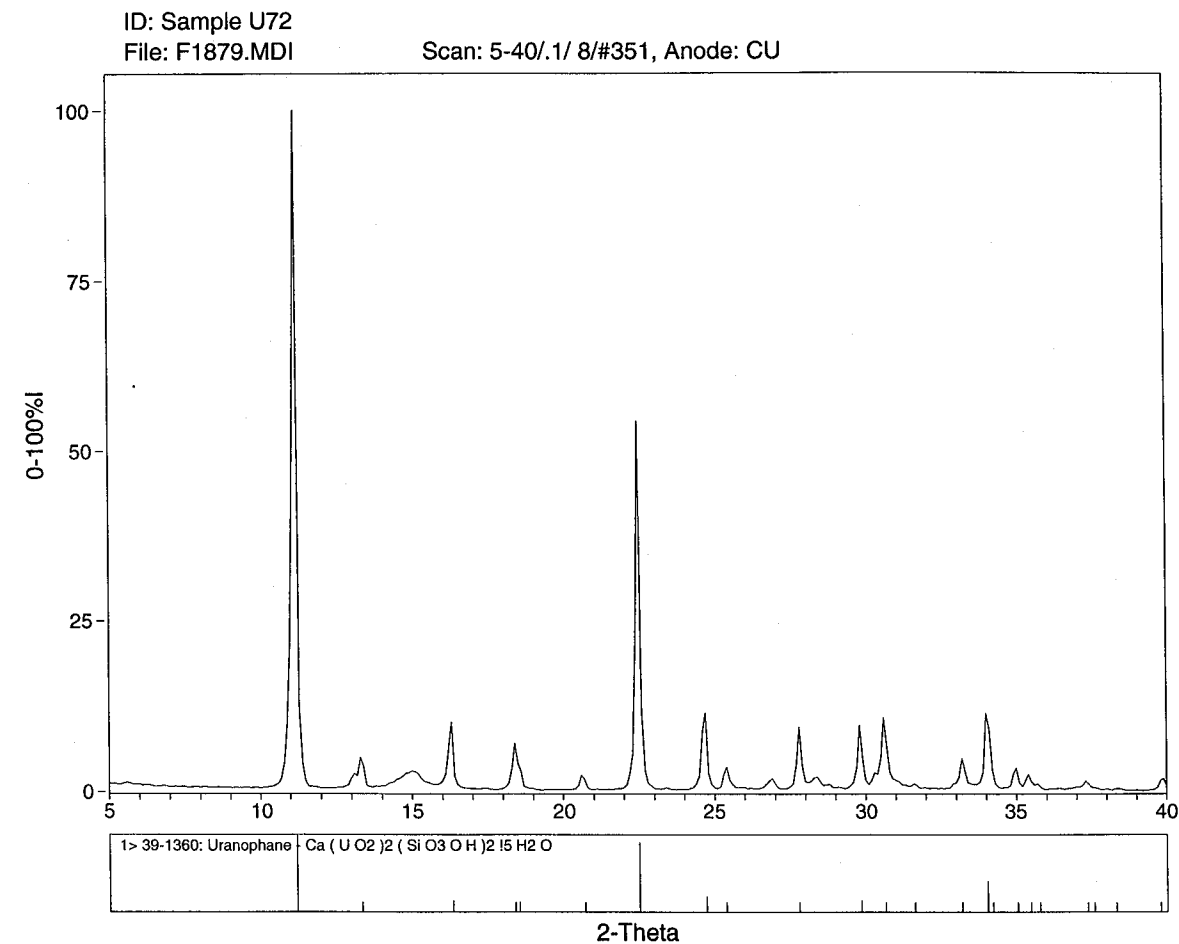
3/20/02 JP (13) Samples were powdered and analyzed by XRD. The XRD patterns are shown on the following pages.



Sample taken at 24 hours. Pattern shows that boltwoodite is still present in a small quantity.

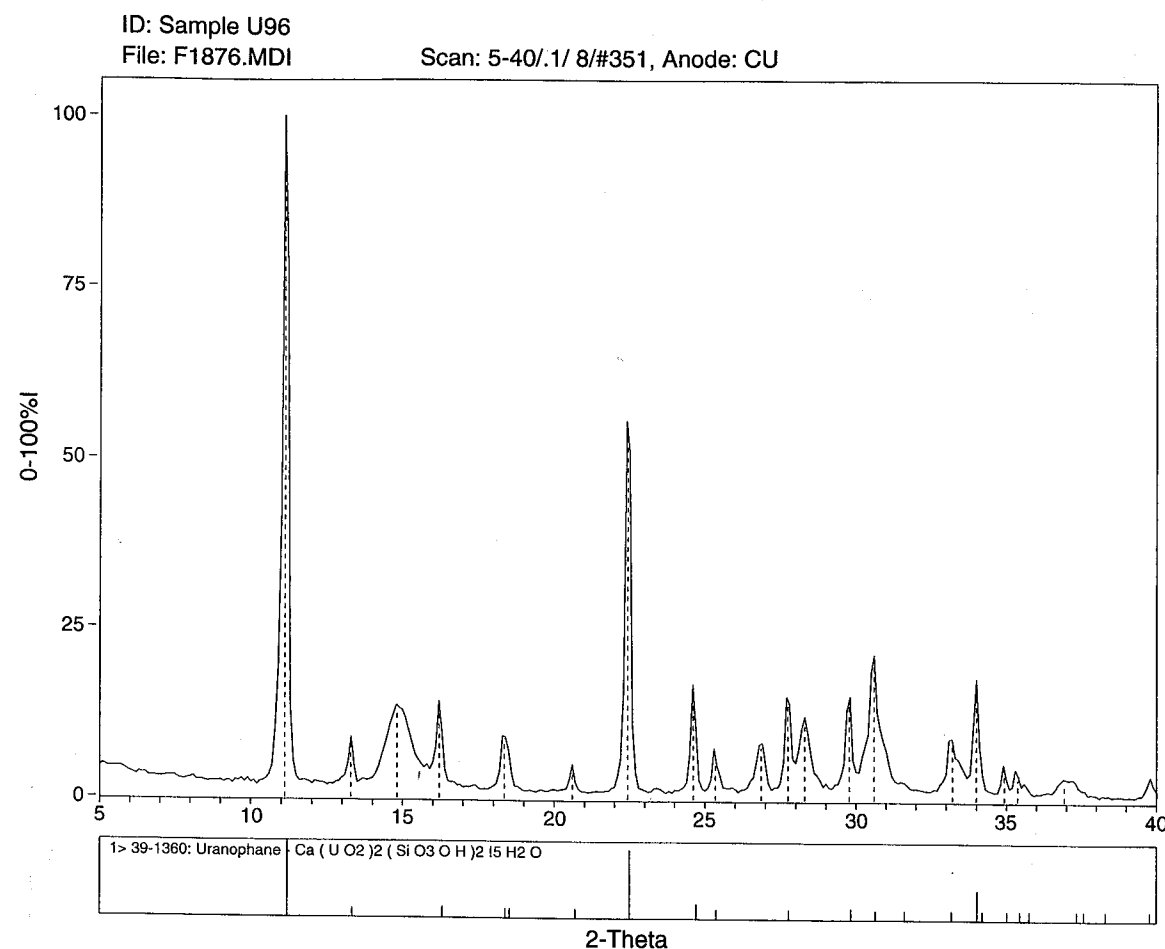


Sample taken at 48 hours. Appears that  
bohtwoodite is no longer present.



Sample at 72 hours. Uranophane is present but  
a broad peak at about 15° 2-Theta has  
appeared. Bohtwoodite is not present.





Sample at 96 hours. The broad peak at  $15^\circ 2\theta$  has increased in intensity. Also some of the uranyl peaks have broadened.

3/21/02 JF

The presence of the broad peak in the XRD pattern at  $15^\circ 2\theta$  at 72 + 96 hours suggests precipitation of a phase other than uranyl. or possibly contamination or degradation of the teflon liner. Leaching of the liner could introduce F into the system which could then be incorporated in the solid. This leaching may be due to the high temperature used in the corrosion.

3/21/02 JF

To test whether F or some other element is being introduced a chemical analysis of this uranyl silicate will be performed.

3/21/02 JF  
1400hr  
Dissolve of U24, U48, U72, and U96 for chemical analysis

Weigh out ~ 0.1 g of the solids and place in 100 volumetric flasks. Sample weights are shown below

Sample	Wt (g)
U24	0.1064
U48	0.1017
U72	0.1031
U96	0.1013
U0	0.1056

3/21/02  
1440h

Add 100 ml of 0.1 N  $\text{HNO}_3$  to volumetric flasks

Once solids have dissolved transfer solution to 125 ml bottles and label with solid name (u0, u24, u48, u72 or u96).

3/21/02 JF  
1500 hr

Send sample to Div 01 for  
chemical analysis. Major + trace cation  
by ICP. Anions by ion chromatography

3/26/02 JF  
1050 hrs

Anions by ion chromatography could not be conducted on the samples because of the  $\text{HNO}_3$  matrix. Therefore only cations by ICP will be analyzed.

3/26/02 JF  
1230 hrs

An attempt will be made by Disol  
to analyze for  $\text{Cl}^-$  by IC.

The Sample List / Chain of Custody  
for the sample sent to Div 01  
is shown on the next page

[illegible]

4/1/02 JF  
0830 hrs.

A new procedure to convert synthesized boltwoodite to manopite was tested. In this test about 1g of synthesized boltwoodite was placed in a teflon lined 125 ml Parr vessel and about 1g of synthesized boltwoodite was placed in a 100 ml PFA teflon vessel. About 50 ml of 2M  $\text{CaCl}_2$  solution was added to each vessel and then the vessels were placed in a Blue M oven set to  $125^\circ\text{C}$ . At several times the vessels were removed from the oven, cooled, and a portion of the solid was removed for XRD analysis after decanting the solution. After sampling another 50 ml of 2M  $\text{CaCl}_2$  was added to each vessel and they were placed back into the oven.

Samples taken from the teflon lined 125 ml Parr vessel were labeled as follows:

U0A - original boltwoodite  
U48A - 48 hrs of reaction  
U96A - 96 " " "  
U168A - 168 " " "  
U336A - 336 " " "

Samples taken from the PFA teflon vessel were labeled:

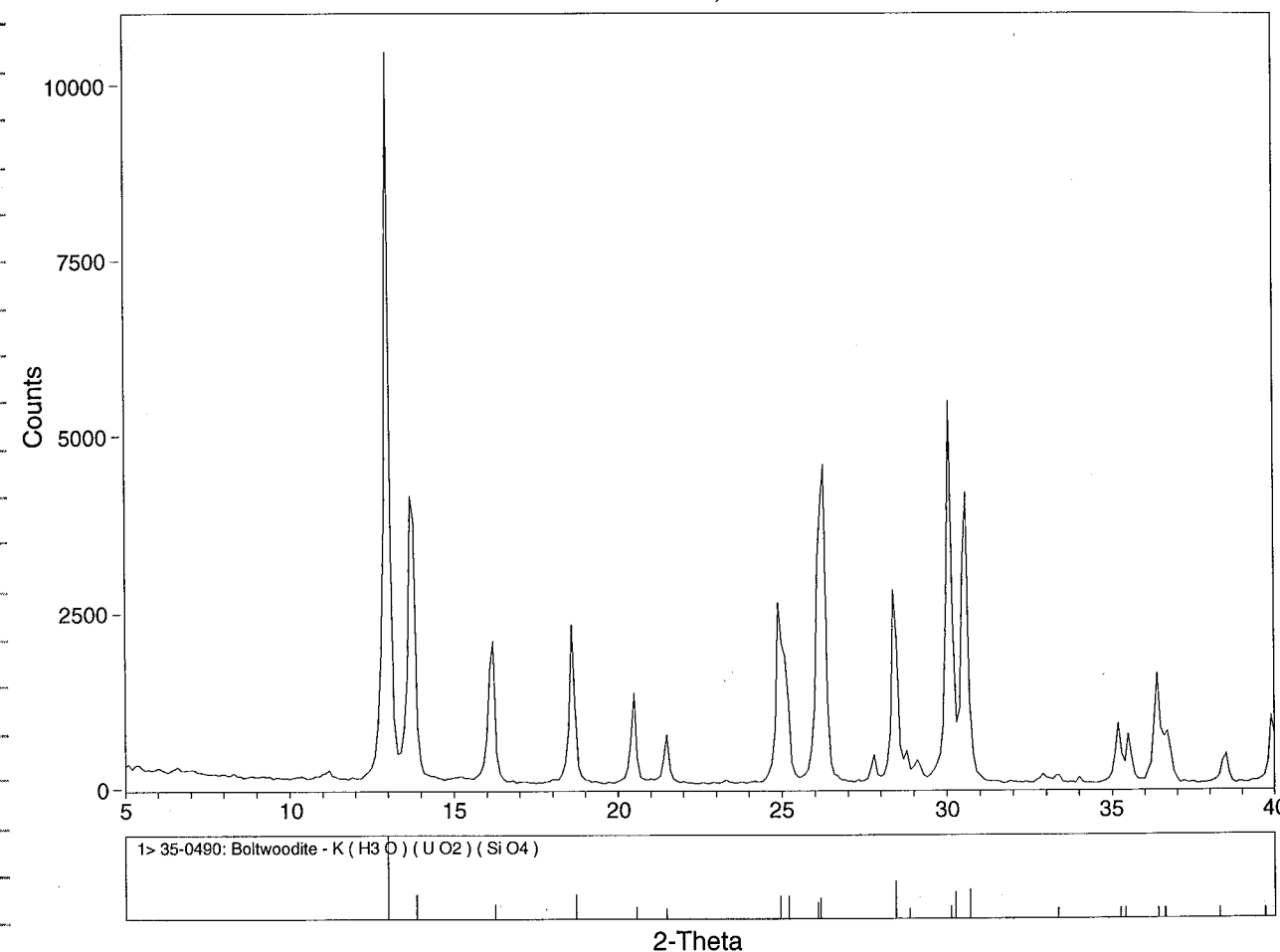
U0B - original boltwoodite  
U48B - 48 hrs of reaction  
U144B - 144 " " "  
U312B - 312 " " "

4/15/02 JF

The samples were powdered and taken to Div 01 for XRD analysis. XRD patterns are shown on the following pages.

ID: U0A - 4/10/02  
File: F1886.MDI

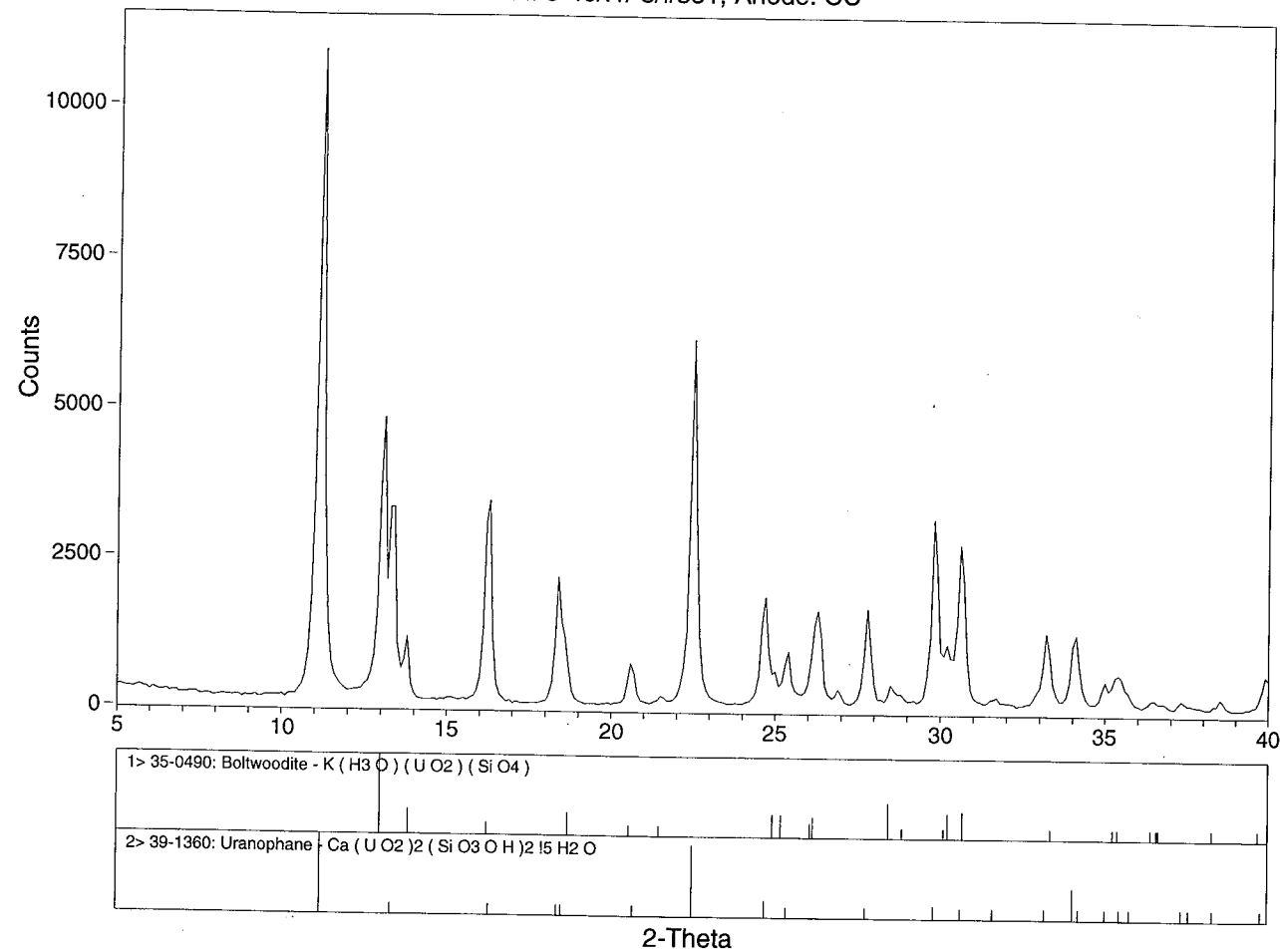
Scan: 5-40/.1/ 8/#351, Anode: CU



U0A - synthesized boltwoodite before reaction with 2M  $\text{CaCl}_2$  soln.

ID: U48A - 4/11/02  
File: F1888.MDI

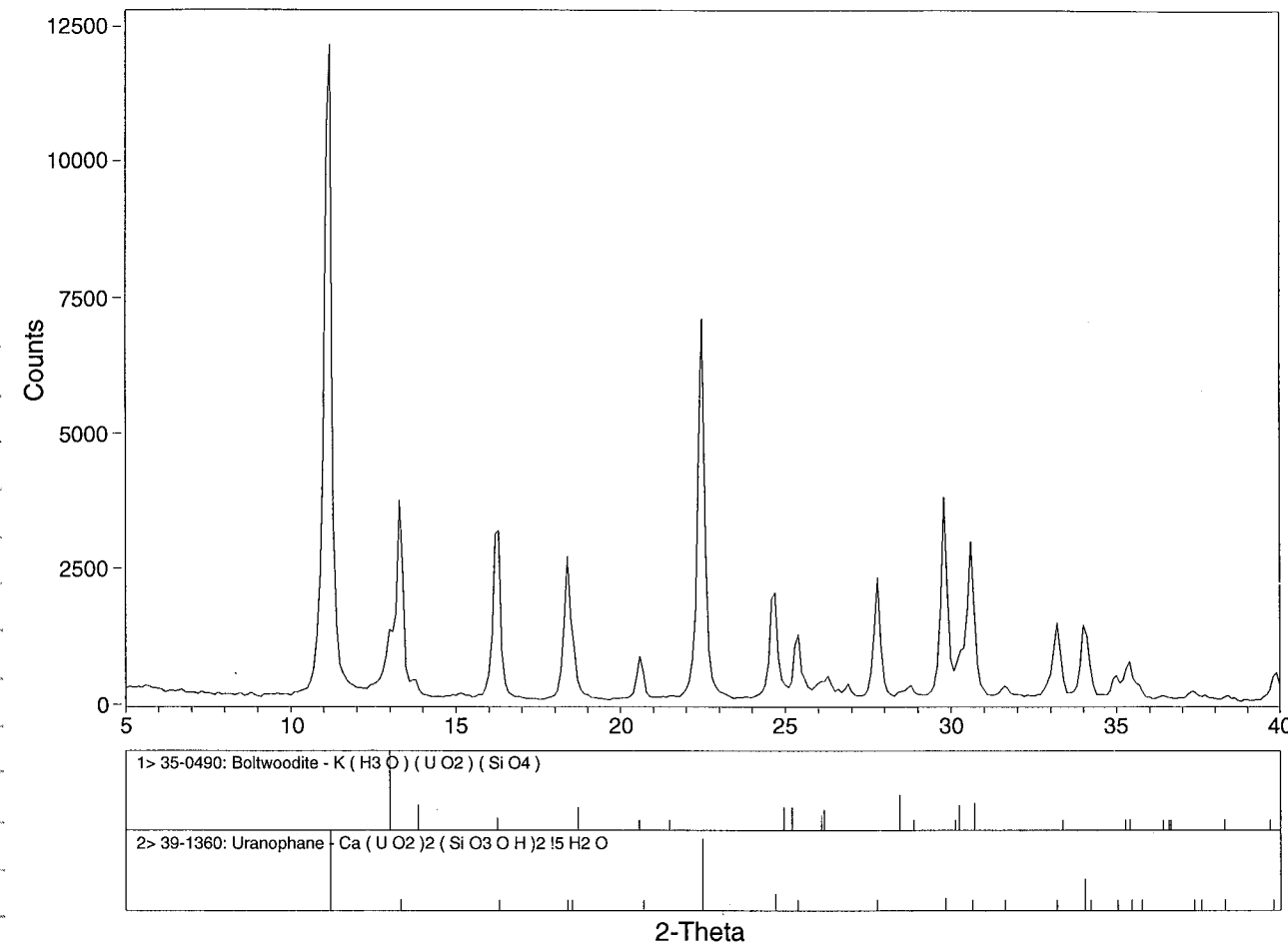
Scan: 5-40/.1/ 8/#351, Anode: CU



U48A - sample after 48 hrs of reaction. Both boltwoodite & uranophane are present.

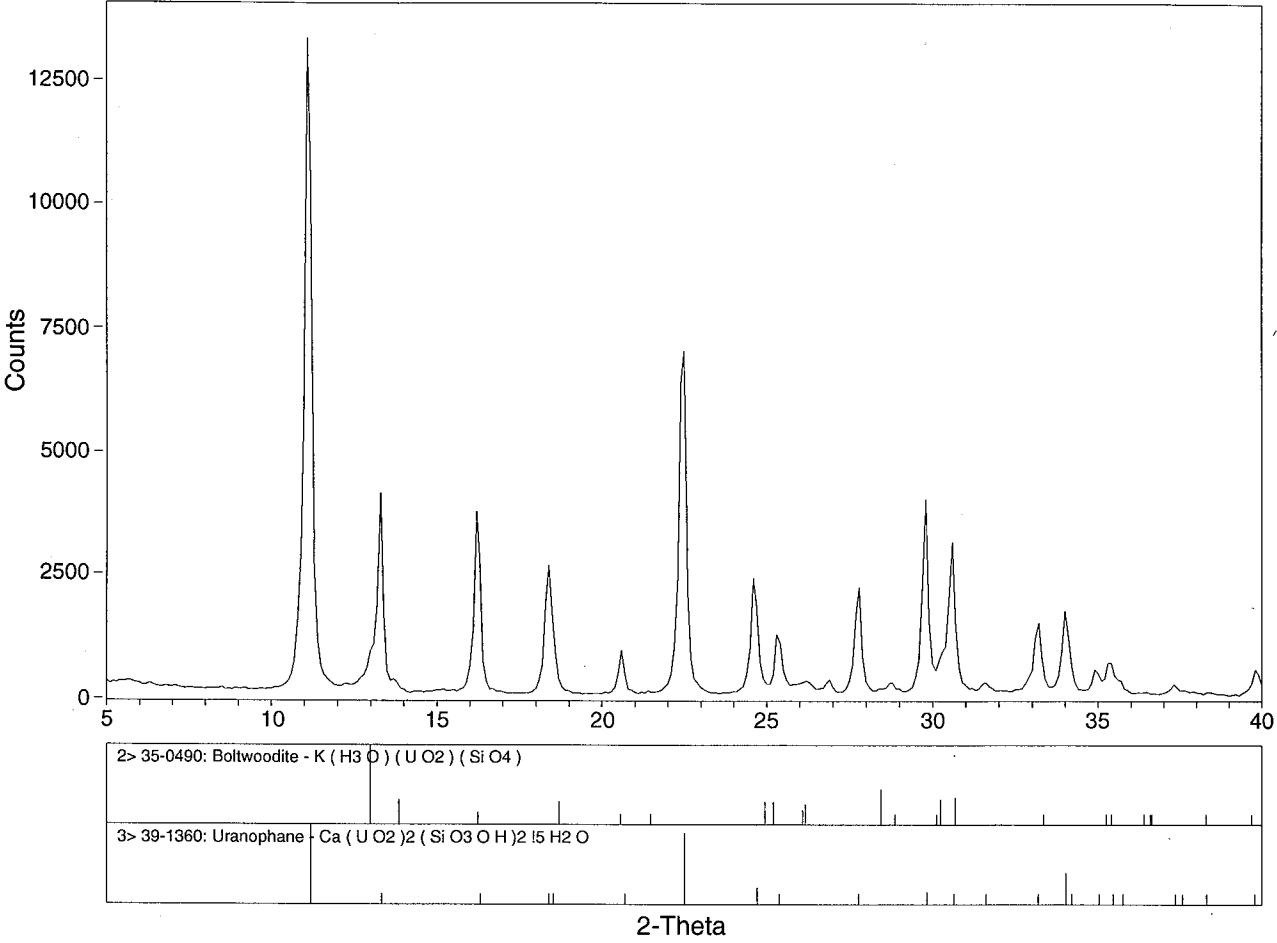
ID: U96A - 4/11/02  
File: F1890.MDI

Scan: 5-40/.1/ 8/#351, Anode: CU



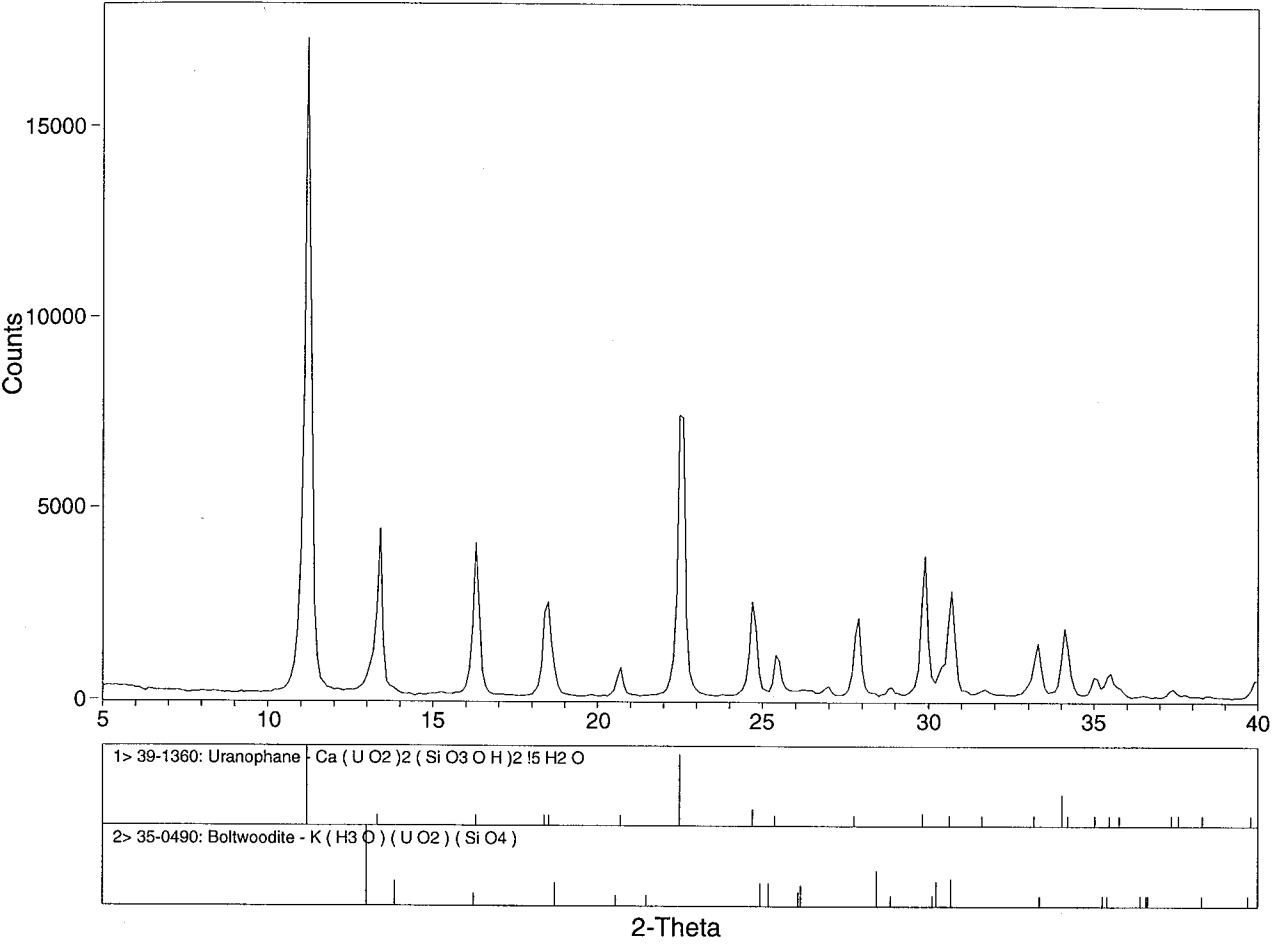
U96A - sample after 96 hrs. of reaction. Some boltwoodite is still present but mostly uranophane.

ID: U168A - 4/11/02  
File: F1892.MDI Scan: 5-40/.1/ 8/#351, Anode: CU



U168A- sample after 168 hrs of reaction. There appears to be a small amount of boltwoodite left.

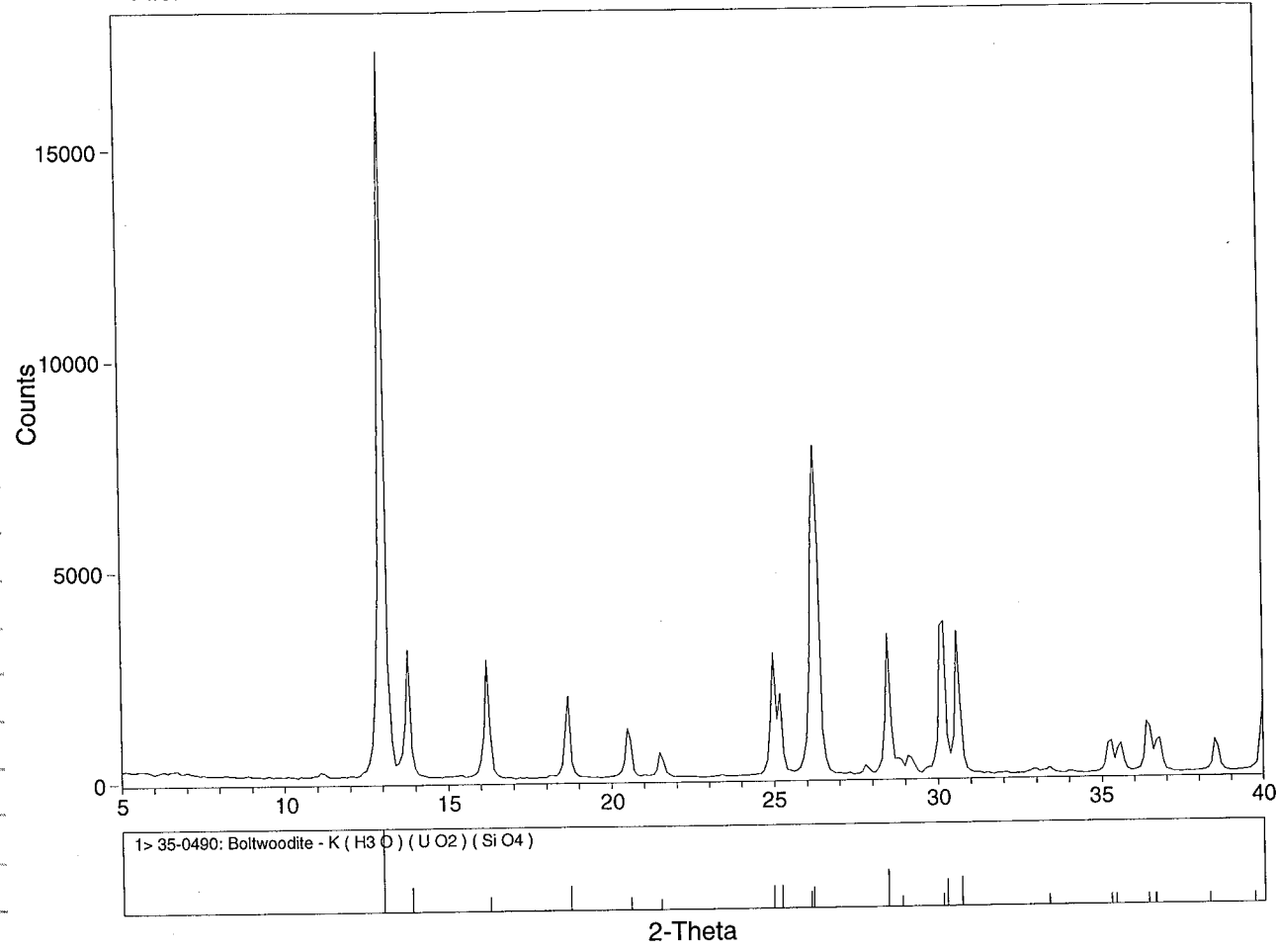
ID: U336A - 4/17/02  
File: F1894.MDI Scan: 5-40/.1/ 8/#351, Anode: CU



U336A- sample after 336 hrs of reaction. The sample appears to have been totally converted to Uranophane.

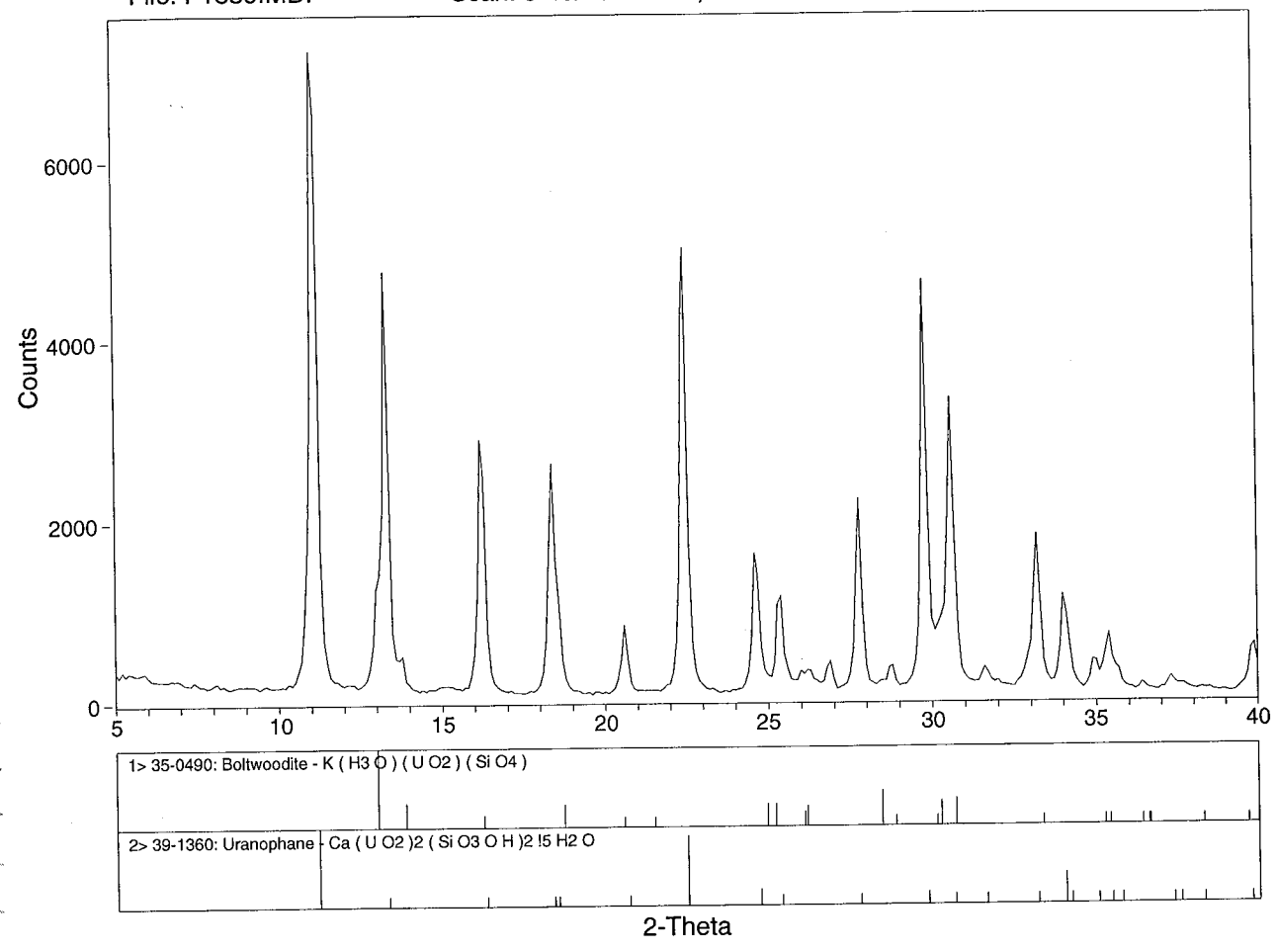


ID: U0B - 4/10/02  
File: F1887.MDI  
Scan: 5-40/.1/ 8/#351, Anode: CU



U0B- synthetic boltwoodite before react with  
2M CaCl<sub>2</sub> solate

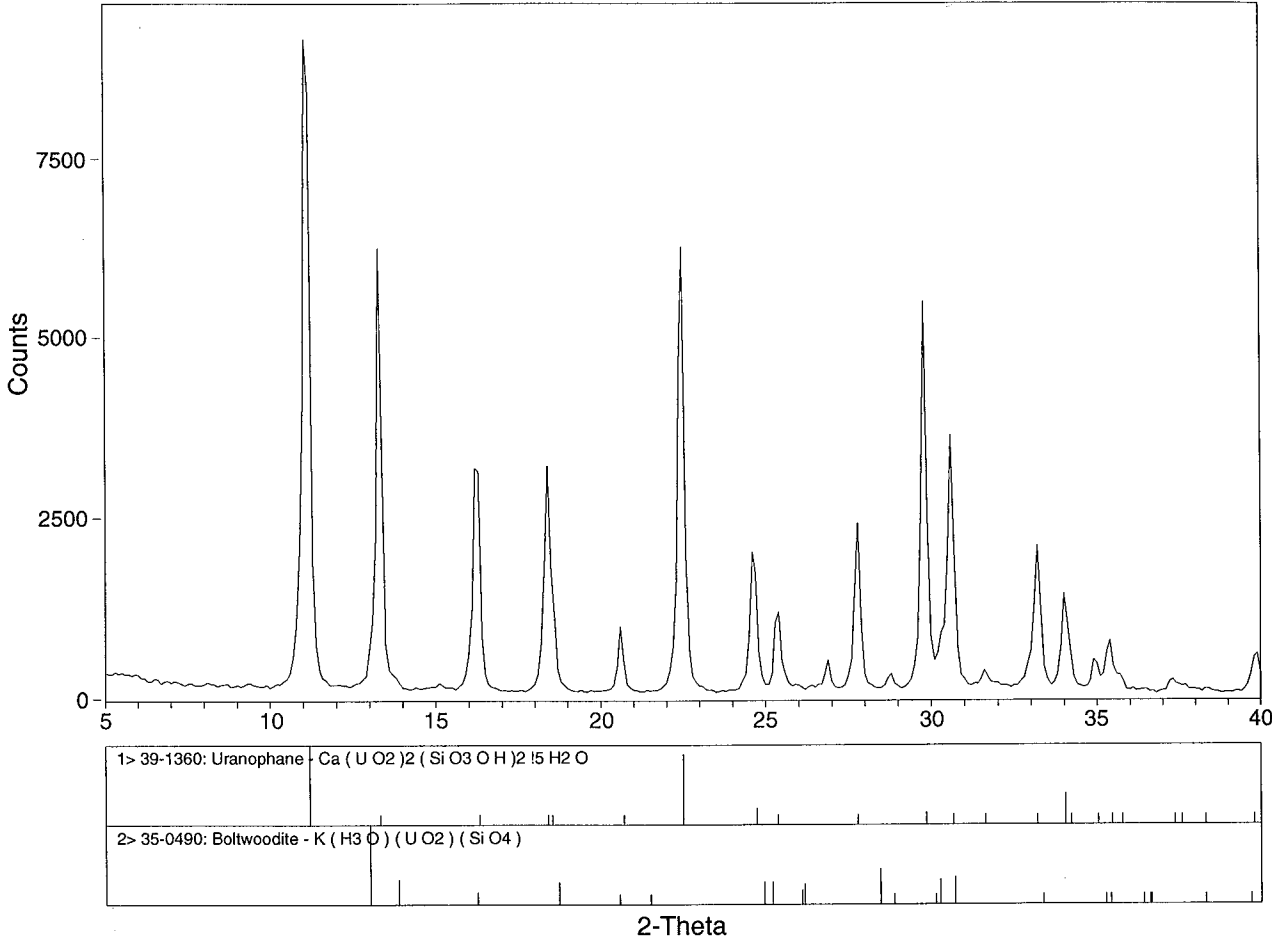
ID: U48B - 4/11/02  
File: F1889.MDI  
Scan: 5-40/.1/ 8/#351, Anode: CU



U48B- sample after 48 hrs of reaction. Both  
boltwoodite + uranophane are present.

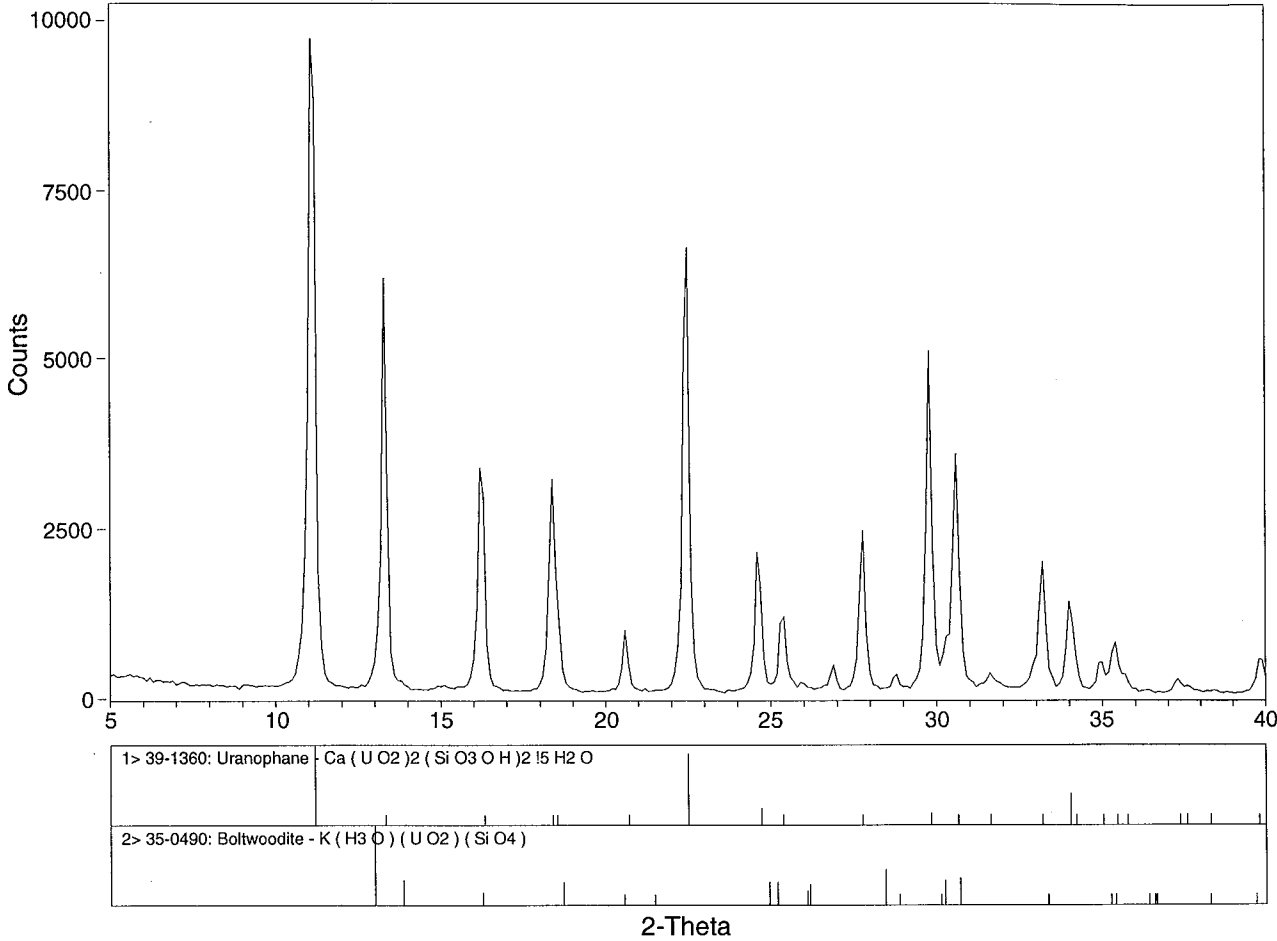
ID: U144B - 4/11/02  
File: F1891.MDI

Scan: 5-40/.1/ 8/#351, Anode: CU



ID: U312B - 4/17/02  
File: F1893.MDI

Scan: 5-40/.1/ 8/#351, Anode: CU



U144B - Sample at 144 hrs of reaction. Mostly uranophane; maybe a small amount of boltwoodite present

U312B - Sample at 312 hrs of reaction. Appears that uranophane conversion is complete.

4/18/02 JP

Dissolution of samples of botwoodite/muscovite  
for chemical analysis

Weigh out approximately 0.1g of the solids  
listed below and place in 100 ml  
volumetric flasks. Sample weights are  
recorded below.

Sample	Weight (g)
U0A	0.1036
U48A	— not sent / analyzed
U96A	— not analyzed
U168A	0.1008
U336A	0.1034
U0B	0.1012
U48B	— not analyzed
U144B	0.1018
U312B	0.1008

Volumetric flasks were filled to mark with  
0.1 M HNO<sub>3</sub>. After solids had completely  
dissolved solute in each volumetric  
flask was transferred to 2 60 ml  
PP bottles and labeled with sample  
number.

One of the 60 ml PP bottles for each  
sample was sent to Div 01 for  
analysis of cation by ICP. The other  
60 ml PP bottle was stored.

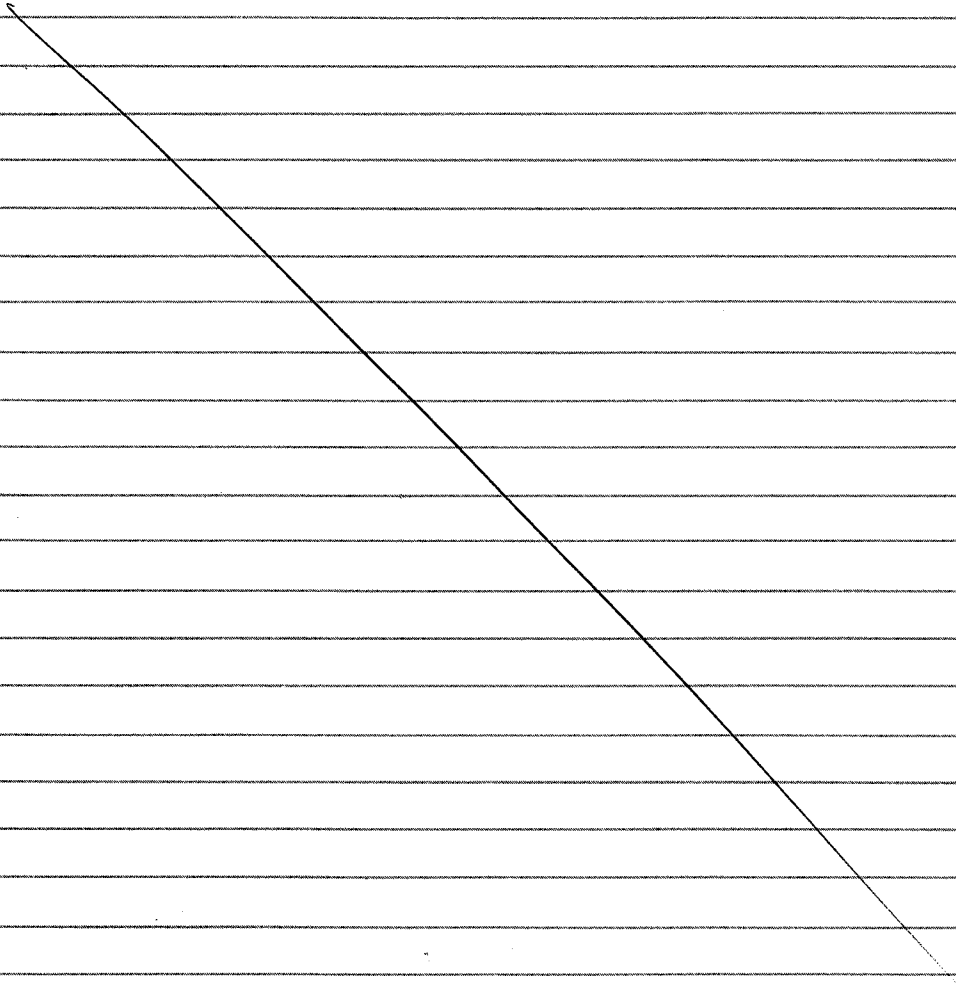
The sample list / chain of custody for the samples  
sent to Div 01 is shown below.

Client Name/Address Jim Prikrny COWRA / Div 20 BIDg 57		SAMPLE LIST/CHAIN OF CUSTODY Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166										Requested Turnaround: <input type="checkbox"/> 1 Week <input checked="" type="checkbox"/> 2 Weeks (Normal) <input type="checkbox"/> 3 Weeks <input type="checkbox"/> 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 - major and minor elements including Uranium											REMARKS Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Other (Specify)
U0A	4/18/02		W	DM	1	✓											Project is nuclear safety related
U0B					1	✓											10 CFR 50 Part 21 Appendix B
U168A					1	✓											
U336A					1	✓											
U144B					1	✓											Question POC
U312B					1	✓											Jim Prikrny x5667 jprikrny@swri.edu
Matrix Types: A - Air; P - Product; S - Soil; T - Tissue; W - Water						Relinquished by (Signature):										SwRI Project No: 20-01402-561	
Sample Types: DM - Dissolved Metals; ER - Equipment Rinse; FB - Field Blank; MSD - Matrix Spike Duplicate; MS - Matrix Spike; TB - Trip Blank; TM - Total Metals; ES - Environmental Samples; FD - Field Duplicate						Received by (Signature):										Received by SwRI Lab (Signature):	
Relinquished by Sampler (Signature): James D. Pugh 4/18/02						Relinquished by (Signature):										Samples Disposed by:	
Received by (Signature):						Comments:										Date/Time:	

7/2/02 JP

Results of chemical analyses of samples submitted to Div 01 on 3/26/02 were received. The samples were portions of synthesized boltwoodite (sample U10) which had been reacted with 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  solution at 185°C for 24 hrs (sample U24), 48 hrs (sample U48), 72 hrs (sample U72) and 96 hrs (sample U96).

Results of the analyses are shown on the following pages.



**SOUTHWEST RESEARCH INSTITUTE**  
**PRELIMINARY SAMPLE ANALYSIS DATA SHEET**

Sample ID  
U10

Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SWRI      Date Received: 03/26/02  
Matrix: Liquid      Project No.: 20.01402.561  
Lab System ID: 200677      Work Order: 22106

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	<0.5	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	75.2	0.1
Selenium	<0.1	0.1
Silicon	69.5	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	591	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3
Chloride	<1.0	1

Synthesized boltwoodite prior to reaction with  $2\text{M CaCl}_2 \cdot \text{H}_2\text{O}$

SOUTHWEST RESEARCH INSTITUTE  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 200678

Client: Division 20

Date Received: 03/26/02

Project No.: 20.01402.571

Work Order: 22106

Sample ID

U48

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	48.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.75	0.1
Selenium	<0.1	0.1
Silicon	68.0	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	577	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3
Chloride	1.86	1

24 hrs of reaction with 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$

SOUTHWEST RESEARCH INSTITUTE  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Lab System ID: 200679

Client: Division 20

Date Received: 03/26/02

Project No.: 20.01402.571

Work Order: 22106

Sample ID

U48

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	50.4	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	60.1	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	558	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3
Chloride	4.12	1

48 hrs of reaction with 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$



SOUTHWEST RESEARCH INSTITUTE  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 200680

Client: Division 20  
Date Received: 03/26/02  
Project No.: 20.01402.571  
Work Order: 22106

Sample ID  
U72

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	48.6	0.05
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	55.2	0.025
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	586	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3
Chloride	1.55	1

72 hrs of vent with 2M CaCl<sub>2</sub> · 2H<sub>2</sub>O

SOUTHWEST RESEARCH INSTITUTE  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 200681

Client: Division 20  
Date Received: 03/26/02  
Project No.: 20.01402.571  
Work Order: 22106

Sample ID  
U96

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	64.9	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	46.5	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	602	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3
Chloride	2.05	1

96 hrs of vent with 2M CaCl<sub>2</sub> · 2H<sub>2</sub>O.

7/2/02 JP

The table below shows variation in elemental concentration of Ca, K, Si, Cl, U for the sample analyzed shown on the previous pages with time.

(in ppm)						
Sample	Reaction Time	Ca	K	Si	U	Cl
UO	0	<0.5	75.2	69.5	591	<1
U24	24 hr	48.1	1.75	68.0	577	1.86
U48	48 hr	50.4	<1	60.1	558	4.12
U72	72 hr	48.6	<1	55.2	586	1.55
U96	96 hr	64.9	<1	46.5	602	2.05

The variation in elemental concentration shown above along with XRD analysis of these samples shown on pages 167-170 indicate that a phase other than monophase was formed during the reaction of synthetic botwoodite with 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  at  $185^\circ\text{C}$ . The general increase in Ca + U and decrease in Si with time suggest that the phase was a Ca uranyl hydrate. The presence of Cl in the reacted solids indicate that the phase may have contained Cl also.

7/3/02 JP

Results of chemical analysis of samples submitted to Div 01 on 4/18/02 (p185) were received. The samples were portions of synthetic botwoodite (samples UOA and UOB) which had been reacted with 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  solution at  $125^\circ\text{C}$  for 144 hr (U144B), 312 hr (U312B), 168 hr (U168A) and 336 hr (U336A).

Results of the analysis are shown on the following pages.

**SOUTHWEST RESEARCH INSTITUTE**  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203979

Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.571  
Work Order: 22287

Sample ID  
UOA

Analysis	Sample Result (mg/l.)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	<0.5	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	0.054	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	73.9	0.1
Selenium	<0.1	0.1
Silicon	67.6	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	572	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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**SOUTHWEST RESEARCH INSTITUTE**  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203976

Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.571  
Work Order: 22287

Sample ID  
U168A

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	44.6	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.80	0.1
Selenium	<0.1	0.1
Silicon	65.7	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	557	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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**SOUTHWEST RESEARCH INSTITUTE**  
**PRELIMINARY SAMPLE ANALYSIS DATA SHEET**

Sample ID  
U336A

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203978

Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.571  
Work Order: 22287

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	0.201	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.4	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.23	0.1
Selenium	<0.1	0.1
Silicon	65.9	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	561	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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**SOUTHWEST RESEARCH INSTITUTE**  
**PRELIMINARY SAMPLE ANALYSIS DATA SHEET**

Sample ID  
UOB

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203980

Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.571  
Work Order: 22287

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	<0.5	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	0.039	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	72.9	0.1
Selenium	<0.1	0.1
Silicon	65.3	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	564	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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**SOUTHWEST RESEARCH INSTITUTE**  
**PRELIMINARY SAMPLE ANALYSIS DATA SHEET**

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203975

Sample ID  
U144B  
Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.561  
Work Order: 22287

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	44.7	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	63.6	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	543	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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**SOUTHWEST RESEARCH INSTITUTE**  
**PRELIMINARY SAMPLE ANALYSIS DATA SHEET**

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 203977

Sample ID  
U312B  
Client: Division 20  
Date Received: 04/18/02  
Project No.: 20.01402.571  
Work Order: 22287

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1	1
Antimony	<0.1	0.1
Arsenic	<0.05	0.05
Barium	0.036	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	45.9	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	65.1	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	548	10
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.3	0.3

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7/3/02 JP

The table belows shows variations in concentrations of Ca, K, Si, and U for the samples shown in the previous pages.

Sample	React Time (hrs)	(in ppm)			
		Ca	K	Si	U
U0A	0	<0.5	73.9	67.6	572
U168A	168	44.6	2.80	65.7	557
U336A	336	46.4	1.23	65.9	561
U0B	0	<0.5	72.9	65.3	564
U144B	144	44.7	<1	63.6	543
U312B	312	45.9	<1	65.1	548

The elemental concentrations shown above along with XRD data in the samples (p 175-183) indicate that boltwoodite was successfully converted to uranophane. The A series samples which were reacted in a Parr vessel with teflon liner still appear to contain some K. The B series samples which were reacted in a PFA Teflon vessel contain < 1 ppm K.

7/3/02 JP

Based on results of the previous tests conducted to develop a procedure for synthesizing uranophane (p 163-200), the following procedure was designed.

## BOLTWOODITE/URANOPHANE SYNTHESIS

Written by J. D. Prikryl  
Date - July 3, 2002

- OBJECTIVE:** to synthesize uranophane  $[\text{Ca}(\text{UO}_2)_2\text{Si}_2\text{O}_7 \cdot 6\text{H}_2\text{O}]$  for use in solubility and coprecipitation experiments
- METHOD:** boltwoodite  $[\text{K}(\text{UO}_2)(\text{SiO}_3\text{OH}) \cdot \text{H}_2\text{O}]$  is initially synthesized and then converted to uranophane by exchanging  $\text{Ca}^{2+}$  for  $\text{K}^+$  in the uranyl silicate crystalline structure
- EQUIPMENT:** Parr pressure reactor with 2L reaction vessel and 2L teflon liner  
Parr 4843 Controller  
ORION pH/mV/ISE/ $^{\circ}\text{C}$  meter  
Ross combination pH electrode  
Analytical balance (Mettler PM 4600)  
Corning stir plate  
Blue M oven  
Fisher FS-28 ultrasonic cleaner  
CEM composite vessels with 100 ml teflon PFA liners  
Spectra Mesh nylon with 210 micron openings
- SUPPLIES:** pH buffer 7, Fisher certified, lot no. 012717-24  
pH buffer 10, Fisher certified, lot no. 012607-24  
ultrapure water  
glassware and plasticware as needed
- REAGENTS:** Uranyl nitrate hexahydrate  $[\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ , lot no. D19L14  
KCl (potassium chloride), lot no. 006242  
KOH (potassium hydroxide), lot no. 015356  
1M KOH; made by adding 28.05g of KOH to 500 ml of ultrapure water  
crystalline quartz fragments (purchased from Wards Geology)  
acetic acid, lot no. 012236  
5% acetic acid; made by adding 50 ml of acetic acid to 950 ml ultrapure water  
Calcium chloride dihydrate  $(\text{CaCl}_2 \cdot \text{H}_2\text{O})$ , lot no. 015282  
2M  $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ ; made by adding 147.20 g of  $\text{CaCl}_2 \cdot \text{H}_2\text{O}$  to 1L ultrapure water

## PROCEDURE:

The following method for uranophane synthesis is based on modifications to a procedure for uranophane synthesis from Vochten et al., 1997, which appeared in Canadian Mineralogist, Vol 35, pp. 735-741.

1. Dissolve 9.28 g of uranyl nitrate and 1.328 g of KCl in 200 ml of ultrapure water.
2. Adjust the pH of the resulting solution with 1M KOH to about 11.5. A K-U gel will form; this gel is highly reactive and will be consumed in the subsequent reaction.
3. Transfer the mixture to a 2L teflon liner used with the Parr reaction vessel.
4. Add about 32 grams of natural quartz fragments that have been cleaned ultrasonically to the teflon liner. Make sure the quartz fragments are large enough to be removed easily from the product.
5. Heat the Parr reaction vessel at 185°C for about 4 days.
6. Allow the vessel to cool; remove the quartz fragments by filtering the mixture through a nylon mesh with 210 micron openings; collect the filtered mixture which will consist of pale yellow needles of boltwoodite and a gel-like white precipitate.
7. Remove the gel-like precipitate by washing the solid 3 times with 5% acetic acid (centrifuge and decant supernatant). Vacuum filter the solid using a 0.45 micron membrane and wash several times with D.I. water to remove acid. Air dry the solid and store in a glass beaker labeled BOLTWOODITE.-2.
8. To make uranophane, place about 1 g of the synthetic boltwoodite in 100 ml PFA vessels (6 vessels were used) and add about 50 ml of 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  solution to each vessel.
9. Place the PFA vessels in a 125°C oven for 2 weeks. Every 2 or 3 days during this 2 week period, remove the vessels, decant the supernatant, add about 50 ml of 2M  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  solution to the vessels, and place back into the 125°C oven.
10. After the 2 week period, remove the vessels, vacuum filter the solid using a 0.45 micron membrane and wash several times with D.I. water, and allow the solid to air dry and store in a glass beaker labeled URANOPHANE.-7.

Preparation of solutions for uranophane solubility experiment URSB

Written by: J. D. Prikryl  
Date: 07/10/02

Objective: Prepare 2 solutions containing U, Ca, and SiO<sub>2</sub> for use in the uranophane solubility experiment URSB. The solutions will have a Ca content of 10<sup>-2</sup> M and a SiO<sub>2</sub> content of 10<sup>-3</sup> M. U content will be 10<sup>-7</sup> M in one solution and U will be absent in the other solution. The pH of the solutions will be approximately 6.0.

Method: Addition of chemical reagents containing Ca and U to an approximate 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Materials and Equipment:

- previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution (see p. 73-74)
- CaCl<sub>2</sub>·2H<sub>2</sub>O; F.W. 147.02; (lot no. 015282)
- UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O; F.W. 502.13; (lot no. D19L14)
- CaCO<sub>3</sub>; F.W. 100.09; (lot no. 986396)
- Polycarbonate bottles; various volumes
- Polypropylene bottles (15 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Mettler analytical balance (AE240)
- weighing paper and boats
- Fisher Versabath Model 236

Procedure

- 7/11/02  
0800hr  
JP
1. Transfer 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution to a 250 ml polycarbonate bottle. Add 0.00502 g of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to the bottle. Label the bottle as 10<sup>-4</sup> m U.
  2. Label 2 500 ml polycarbonate bottles as follows:  
  
1.0x10<sup>-7</sup> m U  
0.0 m U
  3. Transfer about 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution into each 500 ml bottle.
  4. Carefully add the following quantities of reagents and 10<sup>-4</sup> M U solution to the specified bottle. Use weighing paper and weigh out reagents as accurately as possible. For the 10<sup>-4</sup> M U solution use plastic weighing boats and rinse boats using the 10<sup>-3</sup> M SiO<sub>2</sub> solution. After adding reagents make up to a final weight of 500 g with the 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Bottle Label	CaCl <sub>2</sub> ·2H <sub>2</sub> O	CaCO <sub>3</sub>	10 <sup>-4</sup> M U solution
1.0x10 <sup>-7</sup> m U	0.73490 g	0.00013 g	0.5 g
0.0 m U	0.73491 g	0.00013 g	0.0 g

5. Place bottles in shaker water bath set at 25 °C. Allow solutions to equilibrate with air by loosely capping bottles.

6. Every 3 or 4 days take 5 ml aliquots from each bottle and use to measure and record pH to determine when solutions reach equilibrium with air.

7. When equilibrium is reached (i.e., when pH is stable) record the weight of each U matrix solution bottle.

JP  
8/8/02  
0820hr

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	557.39
0.0 m U	558.34

8. Take four 5ml sample aliquots from each U matrix solution and place in 15 ml polypropylene bottles for chemical analysis (2 samples for cation analysis by ICP and 2 samples for Cl analysis by ion chromatography). Use an Oxford pipettor and 5 ml plastic tips to take samples. Preserve the samples for cation analysis by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle. Chemical analysis will show the initial Cl, Ca, SiO<sub>2</sub>, and U contents of the solutions before addition of uranophane. Label the samples as shown below.

JP  
8/8/02  
0825hr

Label	Analysis type	U matrix solution sampled
UMB-7A and UMB-7B	Cations (ICP)	1.0x10 <sup>-7</sup> m U
UMB-7C and UMB-7D	Anions (IC)	1.0x10 <sup>-7</sup> m U
UMB-0A and UMB-0B	Cations (ICP)	0.0 m U
UMB-0C and UMB-0D	Anions (IC)	0.0 m U

9. After sampling record the weight of each U matrix solution bottle.

JP  
8/8/02  
0830hr

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	532.13
0.0 m U	538.23

pH measurements

Sample Matrix

Date / Time	0.0 m U	1.0x10 <sup>-7</sup> m U
7/12/02 0910	5.44	5.48
7/15/02 0800	5.90	5.91
7/19/02 0830	5.56	5.60
7/22/02 0845	5.62	5.62
7/24/02 0725	6.04	5.98
7/29/02 0730	6.01	5.98
8/8/02 0800	5.95	5.93

7-11-02

CONT

BAW

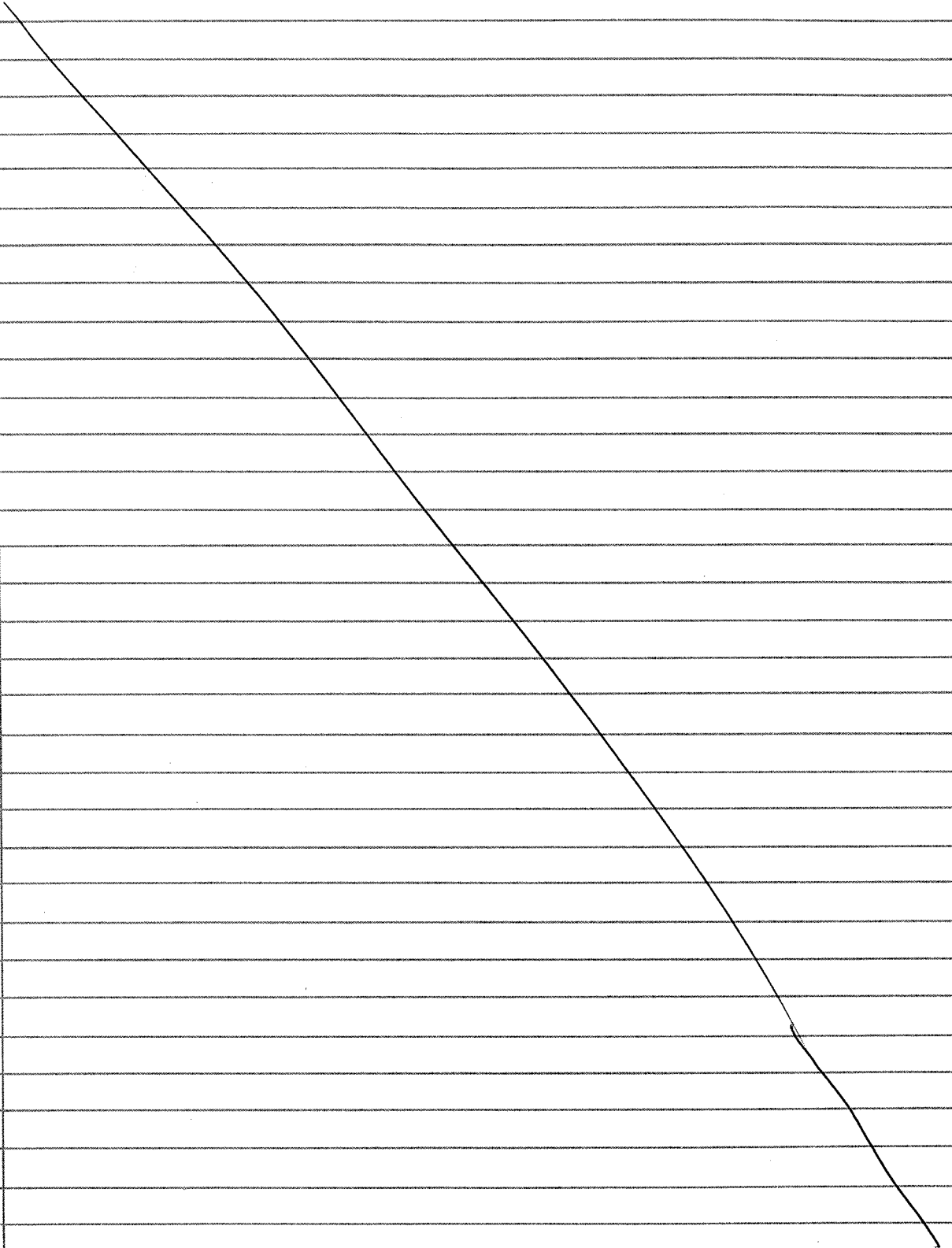
Sample of Silica Soln for ICP DVOI Analysis

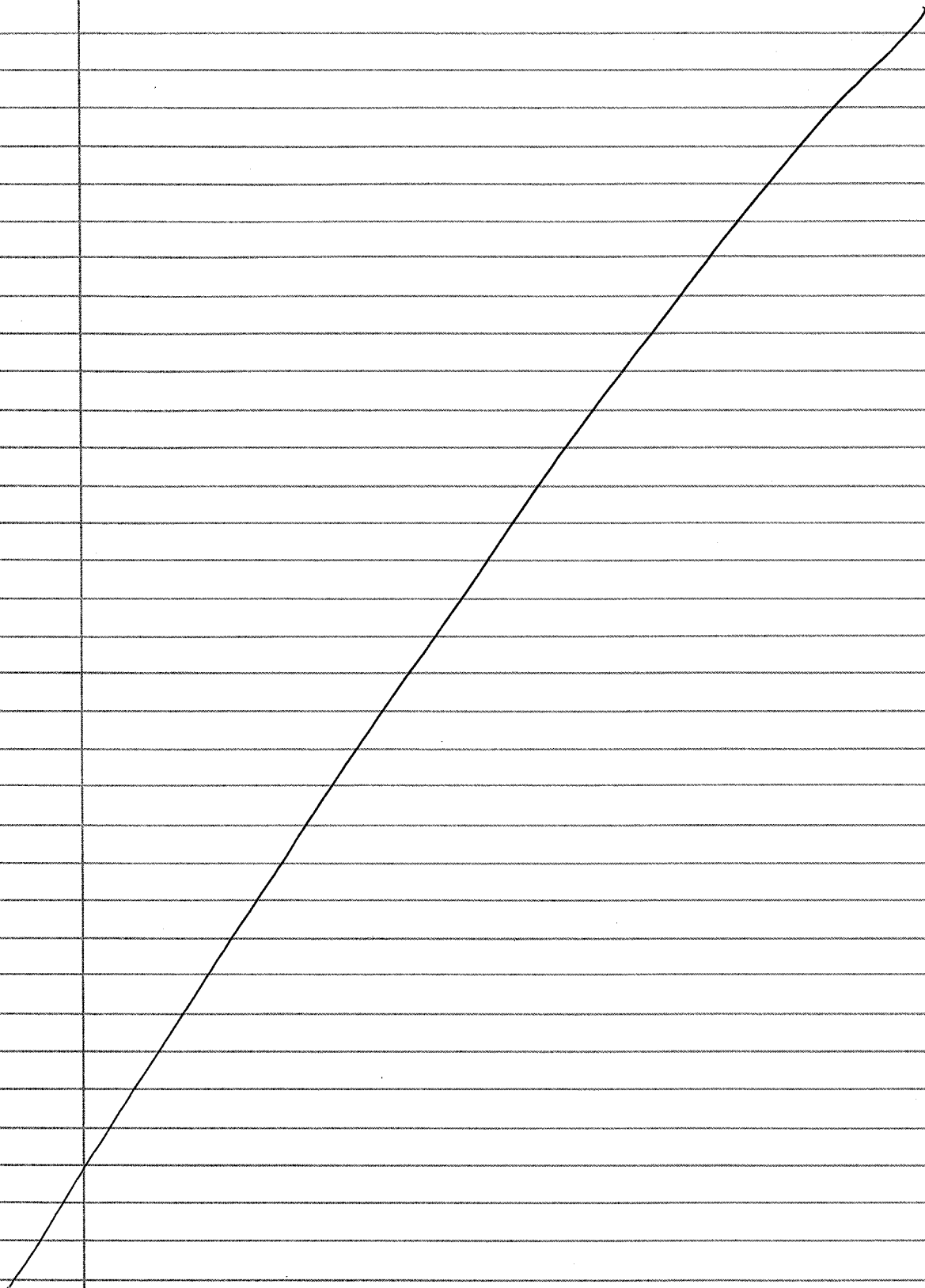
Took three aliquots of  $10^{-5}$  M silica soln (JP 12-22-00, filtered 1/18/00 in carboy) for cation analysis by ICP from DVOI. Each aliquot was  $\sim 20$  mL (10 mL disposable pipet) and transferred to a 30 mL pp bottle. No acid/preservation. Carboy thoroughly shaken before aliquots removed. Labeled SiA, SiB, + SiC.

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# Uranophane solubility experiment URSB

Written by: J. D. Prikryl

Date: 07/10/02

Objective: Determine the solubility of synthesized uranophane.

Method: Monitor the reaction of synthesized uranophane with solutions containing U, Ca, and  $\text{SiO}_2$ .

Materials and Equipment:

- Synthesized uranophane (URANOPHANE-7)
- Previously prepared U matrix solutions with U concentrations from of 0.0 and  $10^{-7}$
- Polycarbonate bottles (250 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Fisher Versabath Model 236
- Mettler analytical balance (AE240 and PM4600)
- Pipets and pipet tips
- Syringe filter; 0.2  $\mu\text{m}$  pore size; Whatman PTFE

## Procedure

1. Label 4 250 ml polycarbonate bottles as follows and record weights.

JP 8/8/02  
0830 hrs.

Label	Weight (g)
URSB-0A	51.50
URSB-0B	51.36
URSB-7A	51.49
URSB-7B	51.38

2. To each sample bottle add  $1.0 \pm 0.0010$  g of synthesized uranophane. Record the weight of uranophane added to each bottle.

JP 8/8/02  
0840 hrs.

Bottle	Wt of uranophane (g)
URSB-0A	1.0004
URSB-0B	1.0008
URSB-7A	0.9999
URSB-7B	1.0007

3. To each bottle add  $200 \pm 0.05$  g of the specified U matrix solution shown in the table below. Record the weight of matrix solution added.

8/8/02  
0850 hrs.

Bottle	U matrix solution	Wt of U matrix solution (g)
URSB-0A	0.0 m U	200.05
URSB-0B	0.0 m U	200.03
URSB-7A	$1.0 \times 10^{-7}$ m U	200.01
URSB-7B	$1.0 \times 10^{-7}$ m U	200.01

4. Record the initial sample weights (bottle + synthesized uranophane + U matrix solution).

JP  
8/2/02  
0910 hr.

Sample	Initial wt (g)
URSB-0A	252.55
URSB-0B	252.39
URSB-7A	252.50
URSB-7B	252.38

5. Cover bottle openings with parafilm with punched holes and place in a shaking water bath set at 25 °C.

6. At 1 week intervals record the weight of each bottle and then remove two 5 ml aliquots from each bottle. Transfer the samples to 15 ml polypropylene bottles for chemical analysis (1 bottle for cation analysis by ICP and the other for pH measurement and Cl analysis by IC). Record the weights of the bottles after sampling. Below are tables into which data can be recorded. Preserve samples for cation analysis by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle.

\* Sample aliquots were filtered thru 0.2 µm filter.

URSB-0A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
8/15/02 0800	250.83	240.85	9.98	7.96	CAT-0A-1	AN-0A-1
8/22/02 0822	238.95	228.96	10.49	8.54	CAT-0A-2	AN-0A-2
8/29/02 0845	226.36	215.97	10.39	7.99	CAT-0A-3	AN-0A-3
9/5/02 1304	214.19	203.92	10.27	8.44	CAT-0A-4	AN-0A-4
9/12/02 0931	201.85	191.48	10.37	8.61	CAT-0A-5	AN-0A-5
9/19/02 1014	189.81	179.72	10.09	5.76	CAT-0A-6	AN-0A-6
9/25/02 0945	178.07	167.85	10.22	5.79	CAT-0A-7	AN-0A-7
10/1/02 0937	166.46	156.10	10.36	5.78	CAT-0A-8	AN-0A-8
10/9/02 1007	154.36	143.79	10.57	5.75	CAT-0A-9	AN-0A-9
10/17/02 0920	141.68	131.13	10.55	5.78	CAT-0A-10	AN-0A-10

URSB-0B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
8/15/02 0807	250.63	240.32	10.31	8.12	CAT-0B-1	AN-0B-1
8/22/02 0826	238.89	228.53	10.36	8.56	CAT-0B-2	AN-0B-2
8/29/02 0850	226.86	216.67	10.19	8.03	CAT-0B-3	AN-0B-3
9/5/02 1311	215.05	204.92	10.13	8.45	CAT-0B-4	AN-0B-4
9/12/02 0939	203.33	193.02	10.31	8.62	CAT-0B-5	AN-0B-5
9/19/02 1022	191.35	181.26	10.09	5.65	CAT-0B-6	AN-0B-6
9/25/02 0949	179.45	169.33	10.12	5.63	CAT-0B-7	AN-0B-7
10/1/02 0941	167.90	157.43	10.47	5.70	CAT-0B-8	AN-0B-8
10/9/02 1014	155.59	145.28	10.31	5.64	CAT-0B-9	AN-0B-9
10/17/02 0924	143.32	132.94	10.38	5.62	CAT-0B-10	AN-0B-10

URSB-7A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
8/15/02 0816	250.87	240.65	10.22	8.04	CAT-7A-1	AN-7A-1
8/22/02 0830	238.89	228.52	10.37	8.56	CAT-7A-2	AN-7A-2
8/29/02 0854	225.84	215.71	10.13	8.02	CAT-7A-3	AN-7A-3
9/5/02 1317	213.50	203.41	10.09	8.24	CAT-7A-4	AN-7A-4
9/12/02 0944	201.36	191.02	10.34	8.11	CAT-7A-5	AN-7A-5
9/19/02 1026	188.51	178.47	10.04	5.59	CAT-7A-6	AN-7A-6
9/25/02 0952	176.53	166.47	10.06	5.59	CAT-7A-7	AN-7A-7
10/1/02 0945	164.66	154.42	10.24	5.70	CAT-7A-8	AN-7A-8
10/9/02 1018	151.78	141.59	10.19	5.55	CAT-7A-9	AN-7A-9
10/17/02 0928	139.05	128.65	10.40	5.54	CAT-7A-10	AN-7A-10

URSB-7B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
8/15/02 0822	251.22	240.78	10.44	8.10	CAT-7B-1	AN-7B-1
8/22/02 0835	239.49	229.27	10.22	8.58	CAT-7B-2	AN-7B-2
8/29/02 0858	228.03	217.82	10.21	8.06	CAT-7B-3	AN-7B-3
9/5/02 1321	216.46	206.22	10.24	8.48	CAT-7B-4	AN-7B-4
9/12/02 0950	204.95	194.61	10.34	8.66	CAT-7B-5	AN-7B-5
9/19/02 1030	193.23	183.20	10.03	5.72	CAT-7B-6	AN-7B-6
9/25/02 0956	181.87	171.79	10.08	5.74	CAT-7B-7	AN-7B-7
10/1/02 0948	170.44	160.14	10.30	5.75	CAT-7B-8	AN-7B-8
10/9/02 1021	158.76	148.43	10.33	5.69	CAT-7B-9	AN-7B-9
10/17/02 0931	146.62	136.32	10.30	5.70	CAT-7B-10	AN-7B-10

8/14/02 JP

Results of chemical analyses of silica solutions (p206)  
sent to Div 01 are shown below.

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 209036

Sample ID  
Si A  
Client: Division 20  
Date Received: 07/11/02  
Project No.: 20.01402.561  
Work Order: 22803

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.050	0.050
Antimony	<0.010	0.010
Arsenic	<0.005	0.005
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.010	0.010
Boron	<0.050	0.050
Cadmium	<0.005	0.005
Calcium	0.050	0.040
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.025	0.025
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.050	0.050
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.020	0.020
Potassium	<0.100	0.100
Selenium	<0.005	0.005
Silicon	34.1	0.020
Silver	<0.005	0.005
Sodium	0.109	0.050
Strontium	<0.005	0.005
Sulfur	0.033	0.020
Thallium	<0.010	0.010
Thorium	<0.010	0.010
Tin	<0.005	0.005
Titanium	<0.005	0.005
Tungsten	<0.010	0.010
Uranium	<0.100	0.100
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 209037

Client: Division 20  
Date Received: 07/11/02  
Project No.: 20.01402.561  
Work Order: 22803

Sample ID  
Si B

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.050	0.050
Antimony	<0.010	0.010
Arsenic	<0.005	0.005
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.010	0.010
Boron	<0.050	0.050
Cadmium	<0.005	0.005
Calcium	0.047	0.040
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.025	0.025
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.050	0.050
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.020	0.020
Potassium	<0.100	0.100
Selenium	<0.005	0.005
Silicon	33.8	0.020
Silver	<0.005	0.005
Sodium	0.092	0.050
Strontium	<0.005	0.005
Sulfur	0.140	0.020
Thallium	<0.010	0.010
Thorium	<0.010	0.010
Tin	<0.005	0.005
Titanium	<0.005	0.005
Tungsten	<0.010	0.010
Uranium	<0.100	0.100
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

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SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Lab System ID: 209038

Sample ID  
Si C

Client: Division 20  
Date Received: 07/11/02  
Project No.: 20.01402.561  
Work Order: 22803

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<0.050	0.050
Antimony	<0.010	0.010
Arsenic	<0.005	0.005
Barium	<0.005	0.005
Beryllium	<0.005	0.005
Bismuth	<0.010	0.010
Boron	<0.050	0.050
Cadmium	<0.005	0.005
Calcium	0.059	0.040
Chromium	<0.005	0.005
Cobalt	<0.005	0.005
Copper	<0.005	0.005
Iron	<0.025	0.025
Lanthanum	<0.005	0.005
Lead	<0.005	0.005
Lithium	<0.005	0.005
Magnesium	<0.050	0.050
Manganese	<0.005	0.005
Molybdenum	<0.005	0.005
Nickel	<0.005	0.005
Palladium	<0.005	0.005
Phosphorus	<0.020	0.020
Potassium	<0.100	0.100
Selenium	<0.005	0.005
Silicon	33.9	0.020
Silver	<0.005	0.005
Sodium	0.099	0.050
Strontium	<0.005	0.005
Sulfur	0.034	0.020
Thallium	<0.010	0.010
Thorium	<0.010	0.010
Tin	<0.005	0.005
Titanium	<0.005	0.005
Tungsten	<0.010	0.010
Uranium	<0.100	0.100
Vanadium	<0.005	0.005
Yttrium	<0.005	0.005
Zinc	<0.005	0.005
Zirconium	<0.005	0.005

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Preparation of solutions for uranophane solubility experiment URSC

Written by: J. D. Prikryl  
Date: 08/15/02

Objective: Prepare 2 solutions containing U, Ca, and SiO<sub>2</sub> for use in the uranophane solubility experiment URSC. The solutions will have a Ca content of 10<sup>-2</sup> M and a SiO<sub>2</sub> content of 10<sup>-3</sup> M. U content will be 10<sup>-7</sup> M in one solution and U will be absent in the other solution. The pH of the solutions will be approximately 6.0.

Method: Addition of chemical reagents containing Ca and U to an approximate 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Materials and Equipment:

- previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution (see p. 73-74)
- CaCl<sub>2</sub>·2H<sub>2</sub>O; F.W. 147.02; (lot no. 015282)
- UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O; F.W. 502.13; (lot no. D19214)
- CaCO<sub>3</sub>; F.W. 100.09; (lot no. 986396)
- Polycarbonate bottles; various volumes
- Polypropylene bottles (15 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Mettler analytical balance (AE240)
- weighing paper and boats
- Fisher Versabath Model 236

Procedure

8/15/02  
1000hr  
1. Transfer 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution to a 250 ml polycarbonate bottle. Add 0.00502 g of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to the bottle. Label the bottle as 10<sup>-4</sup> m U.

2. Label 2 500 ml polycarbonate bottles as follows:

1.0x10<sup>-7</sup> m U  
0.0 m U

3. Transfer about 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution into each 500 ml bottle.

8/15/02  
1005hr

8/15/02  
1020hr  
4. Carefully add the following quantities of reagents and 10<sup>-4</sup> M U solution to the specified bottle. Use weighing paper and weigh out reagents as accurately as possible. For the 10<sup>-4</sup> M U solution use plastic weighing boats and rinse boats using the 10<sup>-3</sup> M SiO<sub>2</sub> solution. After adding reagents make up to a final weight of 500 g with the 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Bottle Label	CaCl <sub>2</sub> ·2H <sub>2</sub> O	CaCO <sub>3</sub>	10 <sup>-4</sup> M U solution
1.0x10 <sup>-7</sup> m U	0.73490 g	0.00013 g	0.5 g
0.0 m U	0.73491 g	0.00013 g	0.0 g

8/15/02  
1030hr  
5. Place bottles in shaker water bath set at 25 °C. Allow solutions to equilibrate with air by loosely capping bottles.

6. Every 3 or 4 days take 5 ml aliquots from each bottle and use to measure and record pH to determine when solutions reach equilibrium with air.
7. When equilibrium is reached (i.e., when pH is stable) record the weight of each U matrix solution bottle.

8/29/02  
0940hr

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	584.55 582.30
0.0 m U	584.55

8. Take four 5ml sample aliquots from each U matrix solution and place in 15 ml polypropylene bottles for chemical analysis (2 samples for cation analysis by ICP and 2 samples for Cl analysis by ion chromatography). Use an Oxford pipettor and 5 ml plastic tips to take samples. Preserve the samples for cation analysis by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle. Chemical analysis will show the initial Cl, Ca, SiO<sub>2</sub>, and U contents of the solutions before addition of uranophane. Label the samples as shown below.

8/29/02  
0944hr

Label	Analysis type	U matrix solution sampled
UMC-7A and UMC-7B	Cations (ICP)	1.0x10 <sup>-7</sup> m U
UMC-7C and UMC-7D	Anions (IC)	1.0x10 <sup>-7</sup> m U
UMC-0A and UMC-0B	Cations (ICP)	0.0 m U
UMC-0C and UMC-0D	Anions (IC)	0.0 m U

9. After sampling record the weight of each U matrix solution bottle.

8/29/02  
0950hr

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	562.34
0.0 m U	564.57

pH measurements

Sample Matrix

Date/Time	0.0 m U	1.0x10 <sup>-7</sup> m U
8/19/02 0900hr	6.38	6.24
8/21/02 0830hr	5.99	6.00
8/22/02 0935hr	6.00	5.94
8/24/02 0805hr	6.05	5.94
8/29/02 0930hr	6.02	5.98

Uranophane solubility experiment URSC

Written by: J. D. Prikryl  
Date: 08/21/02

Objective: Determine the solubility of synthesized uranophane.

Method: Monitor the reaction of synthesized uranophane with solutions containing U, Ca, and SiO<sub>2</sub>.

Materials and Equipment:

- Synthesized uranophane (URANOPHANE-7)
- Previously prepared U matrix solutions with U concentrations from of 0.0 and 10<sup>-7</sup>
- Polycarbonate bottles (250 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Fisher Versabath Model 236
- Mettler analytical balance (AE240 and PM4600)
- Pipets and pipet tips
- Syringe filter, 0.2 µm pore size, Whatman PTFE

Procedure

1. Label 4 250 ml polycarbonate bottles as follows and record weights.

8/29/02 JP  
1000hr

Label	Weight (g)
URSC-0A	51.48
URSC-0B	51.48
URSC-7A	51.32
URSC-7B	51.49

2. To each sample bottle add 0.5±0.0010 g of synthesized uranophane. Record the weight of uranophane added to each bottle.

8/29/02 JP  
1005hr

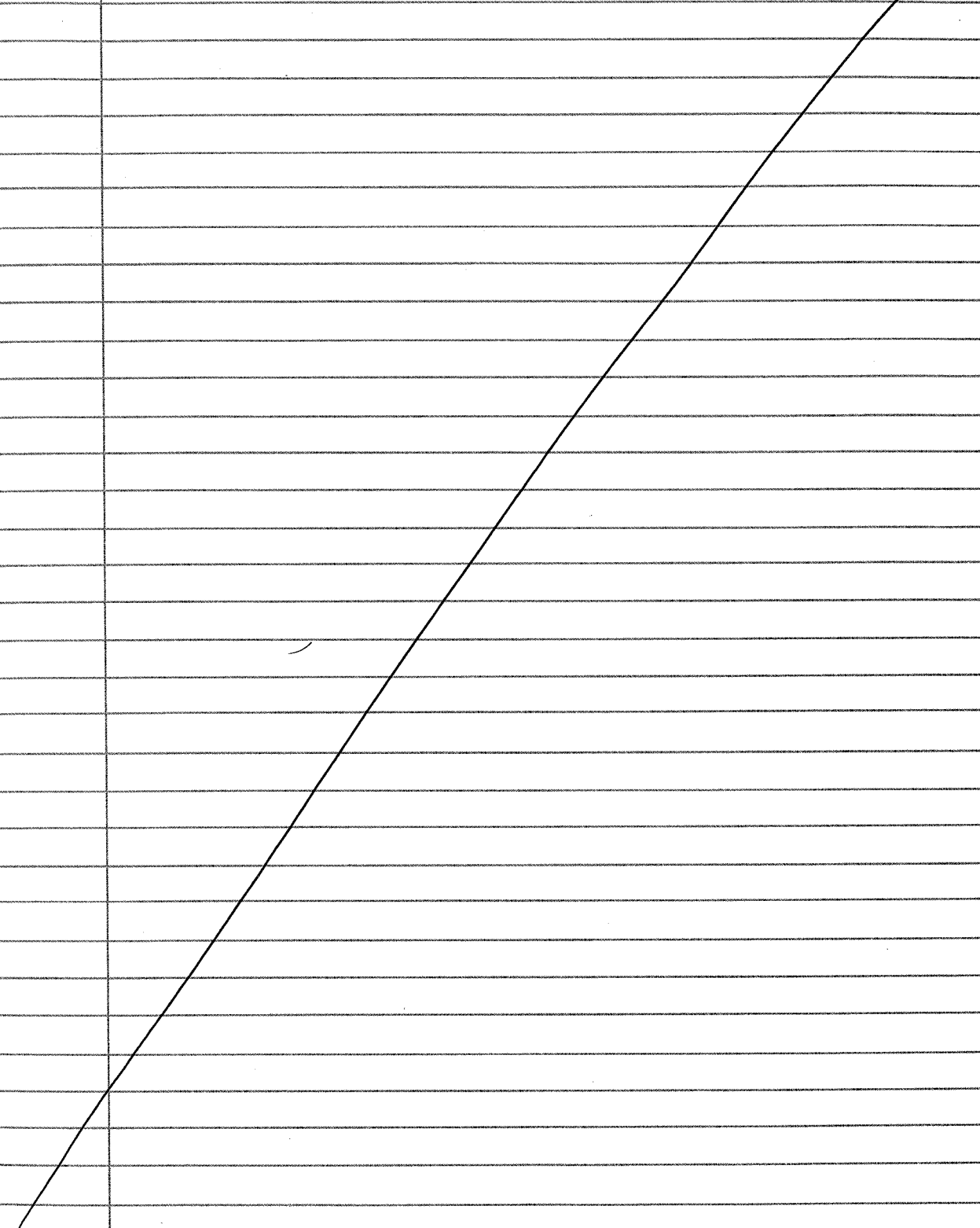
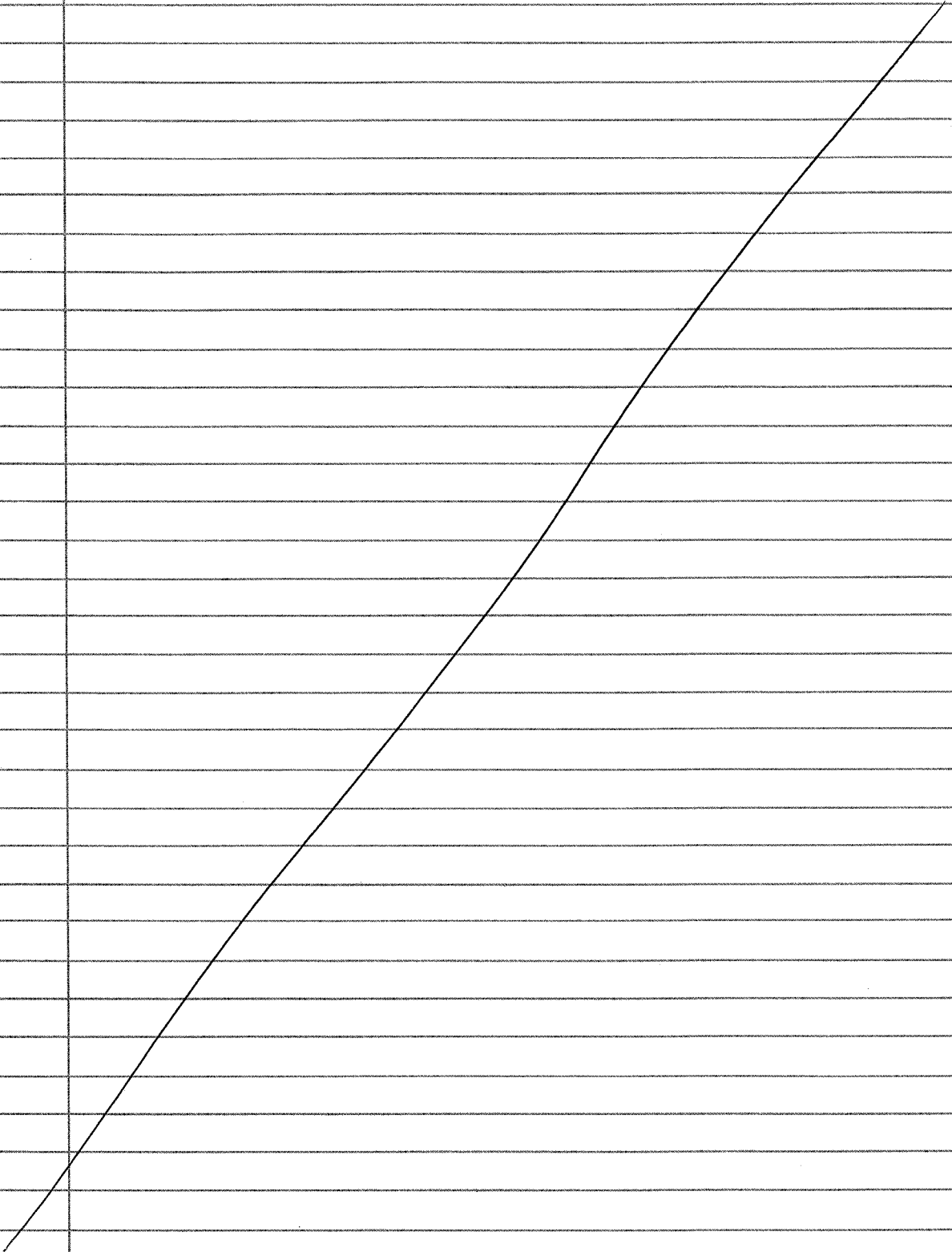
Bottle	Wt of uranophane (g)
URSC-0A	0.4995
URSC-0B	0.5009
URSC-7A	0.5003
URSC-7B	0.5005

3. To each bottle add 200±0.05 g of the specified U matrix solution shown in the table below. Record the weight of matrix solution added.

8/29/02 JP  
1015hr

Bottle	U matrix solution	Wt of U matrix solution (g)
URSC-0A	0.0 m U	200.04
URSC-0B	0.0 m U	200.03
URSC-7A	1.0x10 <sup>-7</sup> m U	200.02
URSC-7B	1.0x10 <sup>-7</sup> m U	200.06

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4. Record the initial sample weights (bottle + synthesized uranophane + U matrix solution).

8/29/02  
1020hr

Sample	Initial wt (g)
URSC-0A	252.01
URSC-0B	252.01
URSC-7A	251.83
URSC-7B	252.04

5. Cover bottle openings with parafilm with punched holes and place in a shaking water bath set at 25 °C.

6. At 1 week intervals record the weight of each bottle and then remove two 5 ml aliquots from each bottle. Transfer the samples to 15 ml polypropylene bottles for chemical analysis (1 bottle for cation analysis by ICP and the other for pH measurement and Cl analysis by IC). Record the weights of the bottles after sampling. Below are tables into which data can be recorded. Preserve samples for cation analysis by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle.

Sample aliquots were filtered thru 0.2 µm filters.

URSC-0A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
9/5/02 1329	250.40	240.22	10.18	8.05	CATC-0A-1	ANC-0A-1
9/12/02 1001	238.22	227.88	10.34	8.55	CATC-0A-2	ANC-0A-2
9/19/02 1036	226.23	216.08	10.15	5.93	CATC-0A-3	ANC-0A-3
9/25/02 1000	214.41	204.24	10.17	5.91	CATC-0A-4	ANC-0A-4
10/1/02 0952	202.62	192.35	10.27	5.91	CATC-0A-5	ANC-0A-5
10/9/02 1025	190.64	180.42	10.22	5.91	CATC-0A-6	ANC-0A-6
10/17/02 0935	178.16	167.89	10.27	5.84	CATC-0A-7	ANC-0A-7
10/24/02 0959	166.38	156.11	10.27	5.88	CATC-0A-8	ANC-0A-8
10/31/02 0911	154.19	143.99	10.20	5.98	CATC-0A-9	ANC-0A-9
11/7/02 1239	141.34	131.12	10.22	5.97	CATC-0A-10	ANC-0A-10

URSC-0B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
9/5/02 1334	250.21	240.03	10.18	8.45	CATC-0B-1	ANC-0B-1
9/12/02 1006	238.16	227.97	10.19	8.55	CATC-0B-2	ANC-0B-2
9/19/02 1040	226.00	215.85	10.15	5.93	CATC-0B-3	ANC-0B-3
9/25/02 1003	214.19	204.03	10.16	5.90	CATC-0B-4	ANC-0B-4
10/1/02 0955	202.34	192.09	10.25	5.87	CATC-0B-5	ANC-0B-5
10/9/02 1030	190.19	179.98	10.21	5.91	CATC-0B-6	ANC-0B-6
10/17/02 0939	177.93	167.69	10.24	5.88	CATC-0B-7	ANC-0B-7
10/24/02 1003	165.80	155.61	10.19	5.90	CATC-0B-8	ANC-0B-8
10/31/02 0914	153.50	143.15	10.35	5.94	CATC-0B-9	ANC-0B-9
11/7/02 1242	140.84	130.47	10.37	5.97	CATC-0B-10	ANC-0B-10

URSC-7A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
9/5/02 1338	250.06	239.86	10.20	8.50	CATC-7A-1	ANC-7A-1
9/12/02 1010	238.14	227.79	10.35	8.60	CATC-7A-2	ANC-7A-2
9/19/02 1044	225.84	215.81	10.03	5.85	CATC-7A-3	ANC-7A-3
9/25/02 1006	213.91	203.92	9.99	5.80	CATC-7A-4	ANC-7A-4
10/1/02 0958	202.27	192.10	10.17	5.79	CATC-7A-5	ANC-7A-5
10/9/02 1032	190.00	179.86	10.14	5.87	CATC-7A-6	ANC-7A-6
10/17/02 0943	177.28	167.00	10.28	5.79	CATC-7A-7	ANC-7A-7
10/24/02 1006	165.21	155.07	10.14	5.83	CATC-7A-8	ANC-7A-8
10/31/02 0917	153.25	143.04	10.21	5.82	CATC-7A-9	ANC-7A-9
11/7/02 1245	140.17	130.13	10.04	5.86	CATC-7A-10	ANC-7A-10

URSC-7B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label Cations	Sample Label Anions
9/5/02 1344	250.32	240.24	10.08	8.45	CATC-7B-1	ANC-7B-1
9/12/02 1015	238.41	228.16	10.25	8.16	CATC-7B-2	ANC-7B-2
9/19/02 1047	226.42	216.37	10.05	5.79	CATC-7B-3	ANC-7B-3
9/25/02 1009	214.30	204.26	10.04	5.81	CATC-7B-4	ANC-7B-4
10/1/02 1000	202.87	192.67	10.20	5.81	CATC-7B-5	ANC-7B-5
10/9/02 1035	190.92	180.72	10.20	5.87	CATC-7B-6	ANC-7B-6
10/17/02 0945	178.61	168.28	10.33	5.78	CATC-7B-7	ANC-7B-7
10/24/02 1010	166.73	156.42	10.31	5.87	CATC-7B-8	ANC-7B-8
10/31/02 0920	154.98	144.74	10.24	5.89	CATC-7B-9	ANC-7B-9
11/7/02 1247	142.84	132.52	10.32	5.90	CATC-7B-10	ANC-7B-10

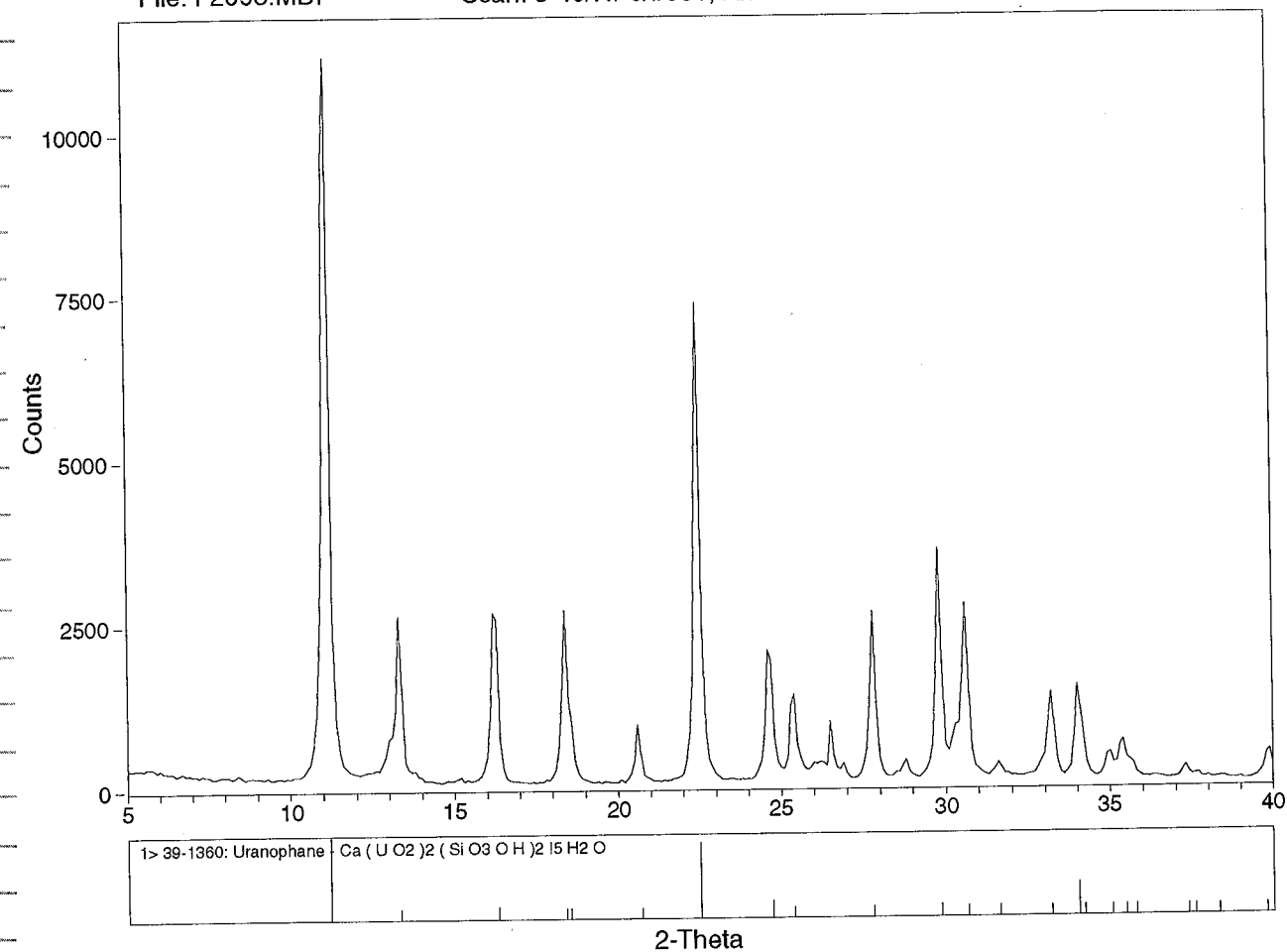
8/29/02 JP

A portion of URANOPHANE-7 was analyzed by XRD in DTD 18. The XRD pattern is shown below.

ID: UR-7

File: F2093.MDI

Scan: 5-40/.1/ 8/#351, Anode: CU





9/17/02 JF 1030hr.

# Dissolution and chemical analysis of URANOPHANE-7

Obj - Determine chemistry and stoichiometry of  
synthesized uranophane (URANOPHANE-7)

Method - Dissolve and analyze cations by  
ICP.

## Materials + Equipment

- URANOPHANE-7 (synthesized uranophane)
- volumetric flasks (50 + 100 ml)
- Mettler AE240 balance
- conc  $\text{HNO}_3$  (lot # 012229)
- DI water
- weighing paper
- PP bottles (various volumes)
- volumetric flask (25 ml)
- Eppendorf pipettes + tips
- Fixed volume glass pipettes
- Spex Centiprep U standard (lot # 8-1234) (1000 ppm)
- Spex Centiprep 1000 ppm Si standard (lot # 8-91 Si)
- Spex Centiprep 1000 ppm Ca standard (lot # 8-140 CA)
- Spex Centiprep 1000 ppm K standard (lot # 8-33 K-Y)
- Spex Centiprep 1000 ppm Na standard (lot # 8-66 NA)

## Procedure

- Label 2 50 ml and 4 100 ml volumetric  
flasks as below

Label	Volume of flask
UR7A	50
UR7B	50
UR7C	100
UR7D	100
UR7E	100
UR7F	100

- Place ~ 0.05 g URANOPHANE-7 in the  
50 ml volumetric flasks or ~ 0.1 g  
URANOPHANE-7 in the 100 ml volumetric  
flasks. Record wts.

Sample	Wt URANOPHANE-7 added
UR7A	0.0510 g
UR7B	0.0501 g
UR7C	0.1010 g
UR7D	0.1018 g
UR7E	0.1009 g
UR7F	0.1013 g

- Add 0.3 ml conc  $\text{HNO}_3$  to the 2 50 ml  
flasks or 0.6 ml conc  $\text{HNO}_3$  to the  
100 ml volumetric flasks.

- Make flasks up to mark with  
DI water + allow URANOPHANE-7 to  
dissolve.

- ⑤ Transfer solution in volumetric flasks to 125 ml PP bottles and label PP bottles same as flasks. (UR7A-UR7B-UR7C-UR7D-UR7E + UR7F).

- ⑥ For each sample, transfer approximately 15 to 20 ml of solute to 2 30 ml PP bottles and label as below.

UR7A-1

UR7A-2

UR7B-1

UR7B-2

UR7C-1

UR7C-2

UR7D-1

UR7D-2

UR7E-1

UR7E-2

UR7F-1

UR7F-2

These samples were sent to Div 01 for cation analysis by ICP.

- ⑦ Prepare standards for quality control of ICP analysis of dissolved URANOPHANE-7. Solutions with known concentrations of U, Si, Ca, K, + Na were prepared.

- ⑧ label 5 25ml volumetric flasks as below.

UR7G

UR7H

UR7I

UR7J

UR7K

- ⑨ Transfer the volume U, Si, Ca, K, or Na standard solution shown in the Tables below to the specified flask.

	U (ml)	Si (ml)	Ca (ml)	K (ml)	Na (ml)
label	1000 ppm	1000 ppm	1000 ppm	20 ppm	20 ppm
UR7G	13.75	1.5	1.0	2.5	2.5
UR7H	14.00	1.6	1.05	1.0	1.0
UR7I	13.50	1.7	1.1	0.5	0.5
UR7J	14.25	1.55	1.15	4.0	5.0
UR7K	14.50	1.65	1.2	5.0	2.0

- ⑩ The table below shows the ppm value of U, Si, Ca, K, + Na for each standard solution.

Label	U (ppm)	Si (ppm)	Ca (ppm)	K (ppm)	Na (ppm)
UR7G	550	60	40	1.0	1.0
UR7H	560	64	42	0.4	0.4
UR7I	540	68	44	0.2	0.2
UR7J	570	62	46	3.2	2.0
UR7K	580	66	48	2.0	0.8

Div 01 COC Form 01-01-001, Rev 4/02

SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client Purchase Order/Other ID				Site/Zone ID	
Analyses Requested				SwRI Contact	
Jim Pickrel CNWRA - Div 20 Bldg 57				Mike Demann	
Sample ID	Sample Collection Date (mm/dd/yy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
UR7F-1	9/17/02	11:00	W		1
UR7F-2					1
UR7G-1					1
UR7G-2					1
UR7H-1					1
UR7I-1					1
UR7I-2					1
UR7J-1					1
UR7K-1					1
UR7K-2					1
Matrix Types: A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe					
Sample Types: D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank					
Relinquished by (Print/Signature) <i>Bigal Wevling / Brad Wevling</i> Date <i>9/24/02</i> Time <i>0928</i> Received by (Print/Signature) <i>[Signature]</i> Date <i>9/24/02</i> Time <i>0928</i> Relinquished by (Print/Signature) <i>[Signature]</i> Date <i>9/24/02</i> Time <i>0928</i> Received by (Print/Signature) <i>[Signature]</i> Date <i>9/24/02</i> Time <i>0928</i> Relinquished by (Print/Signature) <i>[Signature]</i> Date <i>9/24/02</i> Time <i>0928</i> Received by (Print/Signature) <i>[Signature]</i> Date <i>9/24/02</i> Time <i>0928</i>					
Temp: <i>22.0°C</i> Therm #: <i>020</i> Comments: <i>Rel'd. intact</i> <i>325/2284230</i>					
REMARKS Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify) For Jim Pickrel None 5167 Sat 5184 Nuclear safety related - use appropriate QA procedures.					

Div 01 COC Form 01-01-001, Rev 4/02

Page 2 of 2

10/18/02 JP

Recovery of solids from solubility experiment URSB

obj - recover solids remaining at end of solubility experiment URSB (see p 209-211)

method - recover solids by filtration.

Materials and equipment

- Samples URSB-0A, URSB-0B, URSB-7A, + URSB-7B
- 0.45 µm melrose filter
- Vacuum pump
- Fishhawk filter holder
- Erlenmeyer flasks
- Plastic sample container

Procedure.

10/18/02 1. For each sample from solubility  
 14:30hr experiment URSB, capture solids by  
 JP filtering remaining solid/solute mixture  
 through a 0.45 µm melrose filter

10/18/02 2. Allow captured solid to air  
 1520hr JP dry on the filter (~48 hr)

11/21/02 ③ Remove solids from filter and place  
 in plastic bottles labeled as follows.

URSB-0A-S  
 URSB-0B-S  
 URSB-7A-S  
 URSB-7B-S

11/7/02 JP

Recovery of solids from solubility  
 experiment URSC

Obj - recover solids remaining at end of  
 experiment URSC

Method - Filtration

Materials & Equipment

- Syplex URSC-0A, URSC-0B, URSC-7A, URSC-7B
- 0.45  $\mu$ m membrane filter
- Vacuum pump
- Filter holder
- Erlenmeyer flask
- Plastic sample containers

Procedure

11/7/02 ① For each sample from solubility  
 1530h experiment URSC, capture solids by  
 JP filtering remaining solid/solvent mixture  
 through a 0.45  $\mu$ m membrane

11/7/02 ② Allow captured solid to air  
 1600h dry on the filter for at  
 JP least 48 hrs

③ Remove solid from filter + place  
in plastic container labeled as  
follows:

URSC-0A-S

URSC-0B-S

URSC-7A-S

URSC-7B-S



11/12/02 gpp

Results of ICP analyses of URANO PHANE-7  
and QA standards are shown on the  
following pages. See p 226-230 for sample identifications

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7A-1

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 09/24/02

Matrix: Water

Project No.: 20.06002.01.071

Lab System ID: 213278

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.128	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.9	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.39	1
Selenium	<0.1	0.1
Silicon	65.5	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	547	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7A-2

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 09/24/02

Matrix: Water

Project No.: 20.06002.01.071

Lab System ID: 213279

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.128	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.6	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.39	1
Selenium	<0.1	0.1
Silicon	65.5	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	559	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213280

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7B-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.122	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.2	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.24	1
Selenium	<0.1	0.1
Silicon	64.8	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	513	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213281

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7B-2

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.122	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.3	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.30	1
Selenium	<0.1	0.1
Silicon	64.4	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	534	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213282

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.125	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.3	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.36	1
Selenium	<0.1	0.1
Silicon	64.8	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	514	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213283

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.126	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.7	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	2.42	1
Selenium	<0.1	0.1
Silicon	65.3	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	543	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213284

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Sample ID  
UR7D-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.197	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.8	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.79	1
Selenium	<0.1	0.1
Silicon	66.1	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	551	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213285

Sample ID  
UR7D-2  
Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.197	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.7	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	3.55	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.76	1
Selenium	<0.1	0.1
Silicon	65.9	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	0.359	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	553	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	0.710	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213286

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Sample ID  
UR7E-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.186	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.77	1
Selenium	<0.1	0.1
Silicon	65.3	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	549	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213287

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Sample ID  
UR7E-2

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.186	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.3	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.75	1
Selenium	<0.1	0.1
Silicon	65.1	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	548	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213288

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Sample ID  
UR7F-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.186	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.0	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.69	1
Selenium	<0.1	0.1
Silicon	65.0	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	590	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7F-2

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213289

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	0.187	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	47.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.74	1
Selenium	<0.1	0.1
Silicon	65.4	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	551	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5



***SOUTHWEST RESEARCH INSTITUTE***  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7G-1

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwR1

Date Received: 09/24/02

Matrix: Water

Project No.: 20.06002.01.071

Lab System ID: 213290

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	42.2	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	59.6	0.25
Silver	<0.075	0.075
Sodium	1.59	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	547	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

***SOUTHWEST RESEARCH INSTITUTE***  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7G-2

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwR1

Date Received: 09/24/02

Matrix: Water

Project No.: 20.06002.01.071

Lab System ID: 213291

Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	42.2	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	59.4	0.25
Silver	<0.075	0.075
Sodium	1.59	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	545	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213292

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7H-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	44.0	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	63.2	0.25
Silver	<0.075	0.075
Sodium	1.03	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	554	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213293

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7I-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	67.0	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	536	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

***SOUTHWEST RESEARCH INSTITUTE***  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7I-2

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213294

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	46.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	<1	1
Selenium	<0.1	0.1
Silicon	67.1	0.25
Silver	<0.075	0.075
Sodium	<1	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	533	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

***SOUTHWEST RESEARCH INSTITUTE***  
SAMPLE ANALYSIS DATA SHEET

Sample ID  
UR7J-1

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Water  
Lab System ID: 213295

Client: Division 20  
Date Received: 09/24/02  
Project No.: 20.06002.01.071  
Work Order: 23188

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	48.1	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.88	1
Selenium	<0.1	0.1
Silicon	61.3	0.25
Silver	<0.075	0.075
Sodium	2.55	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	520	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213296

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7K-1

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	50.3	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.07	1
Selenium	<0.1	0.1
Silicon	65.2	0.25
Silver	<0.075	0.075
Sodium	1.54	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	601	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

Lab System ID: 213297

Client: Division 20

Date Received: 09/24/02

Project No.: 20.06002.01.071

Work Order: 23188

Sample ID  
UR7K-2

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Aluminum	<1.5	1.5
Antimony	<0.1	0.1
Arsenic	<0.1	0.1
Barium	<0.025	0.025
Beryllium	<0.025	0.025
Bismuth	<0.1	0.1
Boron	<0.2	0.2
Cadmium	<0.025	0.025
Calcium	50.0	0.5
Chromium	<0.2	0.2
Cobalt	<0.025	0.025
Copper	<0.1	0.1
Iron	<2	2
Lanthanum	<0.05	0.05
Lead	<0.1	0.1
Lithium	<0.025	0.025
Magnesium	<4	4
Manganese	<0.025	0.025
Molybdenum	<0.025	0.025
Nickel	<0.15	0.15
Palladium	<0.1	0.1
Phosphorus	<0.25	0.25
Potassium	1.11	1
Selenium	<0.1	0.1
Silicon	65.0	0.25
Silver	<0.075	0.075
Sodium	1.55	1
Strontium	<0.05	0.05
Sulfur	<0.2	0.2
Thallium	<0.15	0.15
Thorium	<2.5	2.5
Tin	<0.05	0.05
Titanium	<0.1	0.1
Tungsten	<0.15	0.15
Uranium	533	1
Vanadium	<0.1	0.1
Yttrium	<0.1	0.1
Zinc	<0.1	0.1
Zirconium	<0.5	0.5

11/13/02 JJ

Preparation of control standards for uranophane solubility experiment solution analysis.

Obj - prepare standards with known concentrations of U, Ca, Si, Na, + K + Cl + NO<sub>3</sub> to determine accuracy of analysis of uranophane solubility experiment solution.

Method - use certified standards to prepare solution with known concentrations of U, Ca, Si, Na, + K + Cl + NO<sub>3</sub>.

Materials & equipment -

- volumetric flask (50 ml)
- DI water
- PP bottles - various sizes
- Eppendorf pipettes & tips
- Fixed volume glass pipettes
- Spex Certified Ca standard, 1000 ppm, lot # 9-69CA
- " " U " " 1000 ppm, lot # 8-123U
- " " Si " " 1000 ppm, lot # 8-91SI
- " " K " " 1000 ppm, lot # 8-33K-Y
- " " Na " " 1000 ppm, lot # 8-66NA
- " " Cl " " 1000 ppm, lot # 23-38AS
- " " NO<sub>3</sub> " " 1000 ppm, lot # 23-24AS
- conc. HNO<sub>3</sub> lot # 012229

Procedure

① Into 8 50 ml volumetric flasks labeled as indicated below add the volume of U, Si, Ca, Na, + K standard as specified in the table below.

Standard	U (ml)	Si (ml)	Ca (ml)	Na (ml)	K (ml)
Standard conc.	10 ppm	1000 ppm	1000 ppm	10 ppm	10 ppm
Label					
CATC-1A-1	10	1.5	19	1	5
CATC-1A-2	1	1.4	25	2	10
CATC-1A-3	0.5	2.0	24	5	4
CATC-1A-4	0.25	2.1	22	4	15
CATC-1A-5	0.1	1.8	23	7	2
CATC-1A-6	2.0	1.9	20	2	5
CATC-1A-7	5.0	1.6	21	0.5	1
CATC-1A-8	3.0	1.7	20	10	4

② The table below shows the ppm value of U, Si, Ca, Na, + K for each standard solution.

Label	U (ppm)	Si (ppm)	Ca (ppm)	Na (ppm)	K (ppm)
CATC-1A-1	2.0	15.30	380	0.2	1.0
CATC-1A-2	0.2	14.28	500	0.4	2.0
CATC-1A-3	0.1	20.40	480	1.0	0.8
CATC-1A-4	0.05	21.42	440	0.8	3.0
CATC-1A-5	0.02	18.36	460	1.4	0.4
CATC-1A-6	0.4	19.38	400	0.4	1.0
CATC-1A-7	1.0	16.32	420	0.1	0.2
CATC-1A-8	0.6	34	400	2.0	0.8

- (3) Add 0.2 ml conc.  $\text{HNO}_3$  to each flask + make up to mark with DI water.
- (4) Transfer QA solute to 60 ml PP bottles with the above labels (i.e. CATC-1A-1 thru CATC-1A-8).
- (5) Into 8 50 ml volumetric flasks labeled as indicated in the table below add the volume of Cl +  $\text{NO}_3$  standard as specified in the table.

Standard Standard conc.	Cl (ml) 1000 ppm	$\text{NO}_3$ (ml) 10 ppm
Label		
ANC-1A-1	35	10
ANC-1A-2	40	1
ANC-1A-3	34	5
ANC-1A-4	36	2
ANC-1A-5	38	4
ANC-1A-6	37	2
ANC-1A-7	35	5
ANC-1A-8	38	0.5

- (6) The table below shows the ppm value of Cl +  $\text{NO}_3$  for each standard solution.

Label	Cl (ppm)	$\text{NO}_3$ (ppm)
ANC-1A-1	700	2.0
ANC-1A-2	800	0.2
ANC-1A-3	680	1.0
ANC-1A-4	720	0.4
ANC-1A-5	760	0.8
ANC-1A-6	740	0.4
ANC-1A-7	700	1.0
ANC-1A-8	760	0.1

- (7) Make up to mark with DI water.

- (8) Transfer solution to 60 ml PP bottles <sup>11/13/02</sup> with the above labels (i.e. <sup>ANC</sup>CATC-1A-1 thru <sup>ANC</sup>CATC-1A-8).  
JP 11/13/02

- (9) Transfer approximately 10 ml of each standard CATC-1A-1 thru CATC-1A-8 to ANC-1A-1 thru ANC-1A-8 to 15 ml PP bottles labeled the same as the original 60 ml bottle.

- (10) Send the 15 ml bottle solution standards to Div 01 for ICP analysis of cations (CATC series) & IC analysis of anions (ANC series).



11/18/02 JP

Solubility experimental samples collected in experiment URSC (p 215-223), matrix samples for this experiment (p 216-217) and QA standard solutions (p 258-261) were sent to Div 01 for cation & anion analyses.

The sample list/Chain of Custody forms for the samples submitted to Div 01 are shown on the following pages.

Cations were analyzed by ICP  
Anions by ICP/MS and anion  
were analyzed by Ion Chromatography

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:	
Jim Pirkryl CNRWA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID	
				SwRI Contact Mike Damann	
		Analyses Requested		REMARKS	
		* Note - Cations of particular interest and their range of expected concentrations are listed below to assist in ICP calibration -		Preservation a = HClO <sub>4</sub> to pH < 2 b = HNO <sub>3</sub> to pH < 2 c = H <sub>2</sub> SO <sub>4</sub> to pH < 2 d = NaOH to pH > 12 e = Cool (4°C/22°C) f = Other (specify)	
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
CATC-0A-1	11/15/02	11:00	W		1
CATC-0A-2					1
CATC-0A-3					1
CATC-0A-4					1
CATC-0A-5					1
CATC-0A-6					1
CATC-0A-7					1
CATC-0A-8					1
CATC-0A-9					1
CATC-0A-10					1
Matrix Types: A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		Sample Types: D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Relinquished by (Print/Signature) Date 11/18/02 Time 0930 Received by (Print/Signature) Date 11/18/02 Time 0930 Relinquished by (Print/Signature) Date Time Received by (Print/Signature) Date Time Relinquished by (Print/Signature) Date Time	
Temp: 22.0°C		Therm #: 027		20.06002.01.071	
Comments:		325/222		Page 1 of 6	

Div 01 COC Form 01-01-001, Rev 4/02

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:	
Jim Pirkryl CNRWA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID	
				SwRI Contact Mike Damann	
		Analyses Requested		REMARKS	
		* See note on page 1		Preservation a = HClO <sub>4</sub> to pH < 2 b = HNO <sub>3</sub> to pH < 2 c = H <sub>2</sub> SO <sub>4</sub> to pH < 2 d = NaOH to pH > 12 e = Cool (4°C/22°C) f = Other (specify)	
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers
CATC-0B-1	11/15/02	11:00	W		1
CATC-0B-2					1
CATC-0B-3					1
CATC-0B-4					1
CATC-0B-5					1
CATC-0B-6					1
CATC-0B-7					1
CATC-0B-8					1
CATC-0B-9					1
CATC-0B-10					1
Matrix Types: A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		Sample Types: D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Relinquished by (Print/Signature) Date 11/18/02 Time 0930 Received by (Print/Signature) Date 11/18/02 Time 0930 Relinquished by (Print/Signature) Date Time Received by (Print/Signature) Date Time Relinquished by (Print/Signature) Date Time	
Temp: 22.0°C		Therm #: 027		20.06002.01.071	
Comments:		325/222		Page 2 of 6	

Div 01 COC Form 01-01-001, Rev 4/02



Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim P. Kuyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact	
						Mike Damann	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC
ANL-DA-1		11/13/02	1100	W		1	✓
ANL-DA-2						1	✓
ANL-DA-3						1	✓
ANL-DA-4						1	✓
ANL-DA-5						1	✓
ANL-DA-6						1	✓
ANL-DA-7						1	✓
ANL-DA-8						1	✓
ANL-DA-9						1	✓
ANL-DA-10						1	✓
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Received by (Print/Signature)		11/18/02	0930
Temp: 22.0°C		Therm #: 027		Relinquished by (Print/Signature)		Date	Time
Comments:				Received by (Print/Signature)		Date	Time
325/222				Relinquished by (Print/Signature)		Date	Time
				Samples Disposed by:		Date	Time

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Page 1 of 6

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim P. Kuyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact	
						Mike Damann	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC
ANL-7A-1		11/13/02	1100	W		1	✓
ANL-7A-2						1	✓
ANL-7A-3						1	✓
ANL-7A-4						1	✓
ANL-7A-5						1	✓
ANL-7A-6						1	✓
ANL-7A-7						1	✓
ANL-7A-8						1	✓
ANL-7A-9						1	✓
ANL-7A-10						1	✓
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Received by (Print/Signature)		11/18/02	0930
Temp: 22.0°C		Therm #: 027		Relinquished by (Print/Signature)		Date	Time
Comments:				Received by (Print/Signature)		Date	Time
325/222				Relinquished by (Print/Signature)		Date	Time
				Samples Disposed by:		Date	Time

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Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim P. Kuyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact	
						Mike Damann	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC
ANL-DB-1		11/13/02	1100	W		1	✓
ANL-DB-2						1	✓
ANL-DB-3						1	✓
ANL-DB-4						1	✓
ANL-DB-5						1	✓
ANL-DB-6						1	✓
ANL-DB-7						1	✓
ANL-DB-8						1	✓
ANL-DB-9						1	✓
ANL-DB-10						1	✓
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Received by (Print/Signature)		11/18/02	0930
Temp: 22.0°C		Therm #: 027		Relinquished by (Print/Signature)		Date	Time
Comments:				Received by (Print/Signature)		Date	Time
325/222				Relinquished by (Print/Signature)		Date	Time
				Samples Disposed by:		Date	Time

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Page 2 of 6

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim P. Kuyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SWRI Contact	
						Mike Damann	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC
ANL-7B-1		11/13/02	1100	W		1	✓
ANL-7B-2						1	✓
ANL-7B-3						1	✓
ANL-7B-4						1	✓
ANL-7B-5						1	✓
ANL-7B-6						1	✓
ANL-7B-7						1	✓
ANL-7B-8						1	✓
ANL-7B-9						1	✓
ANL-7B-10						1	✓
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank		Received by (Print/Signature)		11/18/02	0930
Temp: 22.0°C		Therm #: 027		Relinquished by (Print/Signature)		Date	Time
Comments:				Received by (Print/Signature)		Date	Time
325/222				Relinquished by (Print/Signature)		Date	Time
				Samples Disposed by:		Date	Time

Div 01 COC Form 01-01-001, Rev 4/02

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SAMPLE LIST/CHAIN OF CUSTODY									
Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166									
Client Purchase Order/Other ID					Site/Zone ID				
Analyses Requested									
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC			
ANC-1A-1	11/30/02	1100	W		1	✓			
ANC-1A-2					1	✓			
ANC-1A-3					1	✓			
ANC-1A-4					1	✓			
ANC-1A-5					1	✓			
ANC-1A-6					1	✓			
ANC-1A-7					1	✓			
ANC-1A-8					1	✓			
UMC-OC					1	✓			
UMC-OD					1	✓			
Matrix Types: A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe						Sample Types: D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank			
Temp: 22.0°C						Therm #: 019			
Comments: 325/260									

Relinquished by (Print/Signature) *Jim P. Kyril* Date 11/18/02 Time 0930  
Received by (Print/Signature) *Mike Dammann* Date 11/18/02 Time 0930  
Relinquished by (Print/Signature) Date Date  
Received by (Print/Signature) Date Date  
Relinquished by (Print/Signature) Date Date  
Samples Disposed: Date Time  
Samples Disposed by: Date Time

Requested Turnaround:  
☐ 2 Weeks  
☐ 3 Weeks  
☒ Other: 1 week

SwRI Contact: Mike Dammann

REMARKS  
Preservation:  
a = HCl to pH <2  
b = HNO<sub>3</sub> to pH <2  
c = H<sub>2</sub>SO<sub>4</sub> to pH <2  
d = NaOH to pH >12  
e = Cool (4°C±2°C)  
f = Other (specify)

POC Jim P. Kyril  
x 5667

Nuclear Safety  
related - use  
appropriate QA  
procedure

20.06002.01.071

Div 01 COC Form 01-01-001, Rev 4/02

SAMPLE LIST/CHAIN OF CUSTODY									
Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166									
Client Purchase Order/Other ID					Site/Zone ID				
Analyses Requested									
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analysis by IC			
UMC-7C	11/30/02	1100	W		1	✓			
UMC-7D					1	✓			
Matrix Types: A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe						Sample Types: D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank			
Temp: 22.0°C						Therm #: 021			
Comments: 325/217									

Relinquished by (Print/Signature) *Jim P. Kyril* Date 11/18/02 Time 0930  
Received by (Print/Signature) *Mike Dammann* Date 11/18/02 Time 0930  
Relinquished by (Print/Signature) Date Date  
Received by (Print/Signature) Date Date  
Relinquished by (Print/Signature) Date Date  
Samples Disposed: Date Time  
Samples Disposed by: Date Time

Requested Turnaround:  
☐ 2 Weeks  
☐ 3 Weeks  
☒ Other: 1 week

SwRI Contact: Mike Dammann

REMARKS  
Preservation:  
a = HCl to pH <2  
b = HNO<sub>3</sub> to pH <2  
c = H<sub>2</sub>SO<sub>4</sub> to pH <2  
d = NaOH to pH >12  
e = Cool (4°C±2°C)  
f = Other (specify)

POC Jim P. Kyril  
x 5667

Nuclear Safety  
related - use  
appropriate QA  
procedure

20.06002.01.071

Div 01 COC Form 01-01-001, Rev 4/02

1/7/03 *JP* Results of cation analyses conducted by  
DIO 01.

Results of chemical analyses of samples from uranophane solubility experiment URSC

All analyses are in mg/L

Sample ID	Aluminum	Antimony	Arsenic	Barium	Beryllium	Bismuth	Boron	Cadmium	Calcium	Chromium	Cobalt	Copper	Iron
<i>Matrix Solns</i>													
UMC-0A	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	375	<0.05	<0.025	<0.05	<0.5
UMC-0B	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	373	<0.05	<0.025	<0.05	<0.5
UMC-7A	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	373	<0.05	<0.025	<0.05	<0.5
UMC-7B	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	378	<0.05	<0.025	<0.05	<0.5
<i>Sample Solns</i>													
CATC-0A-1	<0.5	<0.1	<0.1	1.23	<0.025	<0.1	<0.5	<0.025	381	<0.05	<0.025	<0.05	<0.5
CATC-0A-2	<0.5	<0.1	<0.1	0.485	<0.025	<0.1	<0.5	<0.025	391	<0.05	<0.025	<0.05	<0.5
CATC-0A-3	<0.5	<0.1	<0.1	1.36	<0.025	<0.1	<0.5	<0.025	393	<0.05	<0.025	<0.05	<0.5
CATC-0A-4	<0.5	<0.1	<0.1	0.451	<0.025	<0.1	<0.5	<0.025	395	<0.05	<0.025	<0.05	<0.5
CATC-0A-5	<0.5	<0.1	<0.1	0.465	<0.025	<0.1	<0.5	<0.025	396	<0.05	<0.025	<0.05	<0.5
CATC-0A-6	<0.5	<0.1	<0.1	1.25	<0.025	<0.1	<0.5	<0.025	396	<0.05	<0.025	<0.05	<0.5
CATC-0A-7	<0.5	<0.1	<0.1	0.488	<0.025	<0.1	<0.5	<0.025	412	<0.05	<0.025	<0.05	<0.5
CATC-0A-8	<0.5	<0.1	<0.1	0.459	<0.025	<0.1	<0.5	<0.025	406	<0.05	<0.025	<0.05	<0.5
CATC-0A-9	<0.5	<0.1	<0.1	0.439	<0.025	<0.1	<0.5	<0.025	413	<0.05	<0.025	<0.05	<0.5
CATC-0A-10	<0.5	<0.1	<0.1	0.500	<0.025	<0.1	<0.5	<0.025	432	<0.05	<0.025	<0.05	<0.5
<i>Matrix Solns</i>													
CATC-0B-1	<0.5	<0.1	<0.1	0.324	<0.025	<0.1	<0.5	<0.025	379	<0.05	<0.025	<0.05	<0.5
CATC-0B-2	<0.5	<0.1	<0.1	0.490	<0.025	<0.1	<0.5	<0.025	383	<0.05	<0.025	<0.05	<0.5
CATC-0B-3	<0.5	<0.1	<0.1	1.31	<0.025	<0.1	<0.5	<0.025	381	<0.05	<0.025	<0.05	<0.5
CATC-0B-4	<0.5	<0.1	<0.1	1.23	<0.025	<0.1	<0.5	<0.025	390	<0.05	<0.025	<0.05	<0.5
CATC-0B-5	<0.5	<0.1	<0.1	1.37	<0.025	<0.1	<0.5	<0.025	389	<0.05	<0.025	<0.05	<0.5
CATC-0B-6	<0.5	<0.1	<0.1	0.432	<0.025	<0.1	<0.5	<0.025	397	<0.05	<0.025	<0.05	<0.5
CATC-0B-7	<0.5	<0.1	<0.1	0.480	<0.025	<0.1	<0.5	<0.025	404	<0.05	<0.025	<0.05	<0.5
CATC-0B-8	<0.5	<0.1	<0.1	0.560	<0.025	<0.1	<0.5	<0.025	403	<0.05	<0.025	<0.05	<0.5
CATC-0B-9	<0.5	<0.1	<0.1	1.63	<0.025	<0.1	<0.5	<0.025	408	<0.05	<0.025	<0.05	<0.5
CATC-0B-10	<0.5	<0.1	<0.1	0.518	<0.025	<0.1	<0.5	<0.025	430	<0.05	<0.025	<0.05	<0.5
<i>Matrix Solns</i>													
CATC-7A-1	<0.5	<0.1	<0.1	0.419	<0.025	<0.1	<0.5	<0.025	387	<0.05	<0.025	<0.05	<0.5
CATC-7A-2	<0.5	<0.1	<0.1	0.460	<0.025	<0.1	<0.5	<0.025	380	<0.05	<0.025	<0.05	<0.5
CATC-7A-3	<0.5	<0.1	<0.1	1.21	<0.025	<0.1	<0.5	<0.025	382	<0.05	<0.025	<0.05	<0.5
CATC-7A-4	<0.5	<0.1	<0.1	1.27	<0.025	<0.1	<0.5	<0.025	386	<0.05	<0.025	<0.05	<0.5
CATC-7A-5	<0.5	<0.1	<0.1	1.41	<0.025	<0.1	<0.5	<0.025	390	<0.05	<0.025	<0.05	<0.5
CATC-7A-6	<0.5	<0.1	<0.1	1.31	<0.025	<0.1	<0.5	<0.025	394	<0.05	<0.025	<0.05	<0.5
CATC-7A-7	<0.5	<0.1	<0.1	1.27	<0.025	<0.1	<0.5	<0.025	408	<0.05	<0.025	<0.05	<0.5
CATC-7A-8	<0.5	<0.1	<0.1	1.19	<0.025	<0.1	<0.5	<0.025	409	<0.05	<0.025	<0.05	<0.5
CATC-7A-9	<0.5	<0.1	<0.1	1.34	<0.025	<0.1	<0.5	<0.025	414	<0.05	<0.025	<0.05	<0.5
CATC-7A-10	<0.5	<0.1	<0.1	0.452	<0.025	<0.1	<0.5	<0.025	438	<0.05	<0.025	<0.05	<0.5
<i>Matrix Solns</i>													
CATC-7B-1	<0.5	<0.1	<0.1	0.359	<0.025	<0.1	<0.5	<0.025	376	<0.05	<0.025	<0.05	<0.5
CATC-7B-2	<0.5	<0.1	<0.1	1.21	<0.025	<0.1	<0.5	<0.025	376	<0.05	<0.025	<0.05	<0.5
CATC-7B-3	<0.5	<0.1	<0.1	1.31	<0.025	<0.1	<0.5	<0.025	383	<0.05	<0.025	<0.05	<0.5
CATC-7B-4	<0.5	<0.1	<0.1	1.33	<0.025	<0.1	<0.5	<0.025	389	<0.05	<0.025	<0.05	<0.5
CATC-7B-5	<0.5	<0.1	<0.1	1.24	<0.025	<0.1	<0.5	<0.025	390	<0.05	<0.025	<0.05	<0.5
CATC-7B-6	<0.5	<0.1	<0.1	0.494	<0.025	<0.1	<0.5	<0.025	396	<0.05	<0.025	<0.05	<0.5
CATC-7B-7	<0.5	<0.1	<0.1	0.502	<0.025	<0.1	<0.5	<0.025	401	<0.05	<0.025	<0.05	<0.5
CATC-7B-8	<0.5	<0.1	<0.1	1.23	<0.025	<0.1	<0.5	<0.025	404	<0.05	<0.025	<0.05	<0.5
CATC-7B-9	<0.5	<0.1	<0.1	0.482	<0.025	<0.1	<0.5	<0.025	415	<0.05	<0.025	<0.05	<0.5
CATC-7B-10	<0.5	<0.1	<0.1	0.486	<0.025	<0.1	<0.5	<0.025	422	<0.05	<0.025	<0.05	<0.5
<i>QA Stds.</i>													
CATC-1A-1	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	361	<0.05	<0.025	<0.05	<0.5
CATC-1A-2	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	471	<0.05	<0.025	<0.05	<0.5
CATC-1A-3	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	450	<0.05	<0.025	<0.05	<0.5
CATC-1A-4	1.22	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	410	<0.05	<0.025	<0.05	<0.5
CATC-1A-5	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	433	<0.05	<0.025	<0.05	<0.5
CATC-1A-6	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	376	<0.05	<0.025	<0.05	<0.5
CATC-1A-7	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	392	<0.05	<0.025	<0.05	<0.5
CATC-1A-8	<0.5	<0.1	<0.1	<0.025	<0.025	<0.1	<0.5	<0.025	399	<0.05	<0.025	<0.05	<0.5
<i>Matrix Solns</i>													
Reporting Limit (mg/L)	0.5	0.1	0.1	0.025	0.025	0.1	0.5	0.025	0.5	0.05	0.025	0.05	0.5

Lanthanum	Lead	Lithium	Magnesium	Manganese	Molybdenum	Nickel	Palladium	Phosphorus	Potassium	Selenium	Silicon	Silver	Sodium
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.5	<0.05	<0.333
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.4	<0.05	<0.333
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.1	<0.05	<0.333
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.5	<0.05	<0.333
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.6	<0.05	8.41
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.1	<0.05	1.98
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.3	<0.05	8.92
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.2	<0.05	2.05
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.1	<0.05	1.78
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.6	<0.05	8.45
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	32.3	<0.05	2.22
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	32.0	<0.05	2.23
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.13	<0.1	32.5	<0.05	2.40
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.43	<0.1	33.8	<0.05	2.54
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.5	<0.05	1.71
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.8	<0.05	1.69
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	0.750	<0.1	30.5	<0.05	8.56
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.01	<0.1	31.0	<0.05	8.88
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.9	<0.05	9.51
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.5	<0.05	1.88
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.8	<0.05	2.01
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	14.9	<0.1	31.8	<0.05	1.78
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	7.22	<0.1	32.3	<0.05	13.1
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	16.1	<0.1	33.6	<0.05	1.76
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.7	<0.05	2.03
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.1	<0.05	1.68
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.4	<0.05	8.34
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.7	<0.05	9.11
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.8	<0.05	8.97
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.2	<0.05	8.71
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	32.1	<0.05	8.53
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	32.1	<0.05	8.47
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	32.4	<0.05	8.98
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	34.1	<0.05	2.35
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	29.8	<0.05	2.42
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.0	<0.05	8.93
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.3	<0.05	8.87
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.6	<0.05	9.39
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.8	<0.05	8.94
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.9	<0.05	2.71
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.3	<0.05	2.55
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	31.6	<0.05	8.53
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	0.798	<0.1	32.3	<0.05	2.76
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.27	<0.1	32.7	<0.05	2.83
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.47	<0.1	29.5	<0.05	0.863
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	2.29	<0.1	27.3	<0.05	0.705
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.39	<0.1	38.5	<0.05	1.17
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	3.57	<0.1	40.1	<0.05	0.872
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.25	<0.1	34.9	<0.05	1.65
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.63	<0.1	36.8	<0.05	0.736
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	<0.667	<0.1	30.8	<0.05	0.386
<0.05	<0.1	<0.025	<0.5	<0.025	<0.05	<0.05	<0.1	<0.25	1.34	<0.1	34.2	<0.05	2.09
0.05	0.1	0.025	0.5	0.025	0.05	0.05	0.1	0.25	0.667	0.1	0.25	0.05	0.333



1/7/03 JH  
Results of catin analysis continued from  
previous page.

Strontium	Sulfur	Thallium	Thorium	Tin	Titanium	Tungsten	Uranium	Vanadium	Yttrium	Zinc	Zirconium	Sample ID
Matrix Solns												
0.246	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0781	<0.05	<0.05	<0.05	<0.05	UMC-0A
0.245	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0793	<0.05	<0.05	<0.05	<0.05	UMC-0B
0.248	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0235	<0.05	<0.05	<0.05	<0.05	UMC-7A
0.250	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0231	<0.05	<0.05	<0.05	<0.05	UMC-7B
Sample Solns												
0.252	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0004	<0.05	<0.05	0.057	<0.05	CATC-0A-1
0.258	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0016	<0.05	<0.05	0.173	<0.05	CATC-0A-2
0.263	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0008	<0.05	<0.05	0.053	<0.05	CATC-0A-3
0.262	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0023	<0.05	<0.05	0.126	<0.05	CATC-0A-4
0.264	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0020	<0.05	<0.05	0.077	<0.05	CATC-0A-5
0.267	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0020	<0.05	<0.05	<0.05	<0.05	CATC-0A-6
0.276	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0125	<0.05	<0.05	0.098	<0.05	CATC-0A-7
0.272	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0104	<0.05	<0.05	0.076	<0.05	CATC-0A-8
0.276	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.136	<0.05	<0.05	0.090	<0.05	CATC-0A-9
0.287	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.224	<0.05	<0.05	0.119	<0.05	CATC-0A-10
0.250	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0181	<0.05	<0.05	0.086	<0.05	CATC-0B-1
0.256	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0311	<0.05	<0.05	0.208	<0.05	CATC-0B-2
0.257	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0089	<0.05	<0.05	0.076	<0.05	CATC-0B-3
0.262	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0138	<0.05	<0.05	0.143	<0.05	CATC-0B-4
0.263	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0007	<0.05	<0.05	<0.05	<0.05	CATC-0B-5
0.268	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0336	<0.05	<0.05	0.095	<0.05	CATC-0B-6
0.271	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0164	<0.05	<0.05	0.094	<0.05	CATC-0B-7
0.271	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0266	<0.05	<0.05	0.143	<0.05	CATC-0B-8
0.277	0.327	<0.1	<0.1	<0.05	<0.05	<0.1	0.0002	<0.05	<0.05	0.056	<0.05	CATC-0B-9
0.287	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0174	<0.05	<0.05	0.106	<0.05	CATC-0B-10
0.260	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0285	<0.05	<0.05	0.099	<0.05	CATC-7A-1
0.256	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0019	<0.05	<0.05	0.069	<0.05	CATC-7A-2
0.260	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0077	<0.05	<0.05	0.075	<0.05	CATC-7A-3
0.263	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0003	<0.05	<0.05	0.058	<0.05	CATC-7A-4
0.265	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0006	<0.05	<0.05	0.062	<0.05	CATC-7A-5
0.269	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0169	<0.05	<0.05	<0.05	<0.05	CATC-7A-6
0.279	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0073	<0.05	<0.05	0.094	<0.05	CATC-7A-7
0.277	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0035	<0.05	<0.05	0.059	<0.05	CATC-7A-8
0.281	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0003	<0.05	<0.05	<0.05	<0.05	CATC-7A-9
0.297	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0144	<0.05	<0.05	0.091	<0.05	CATC-7A-10
0.252	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0006	<0.05	<0.05	0.086	<0.05	CATC-7B-1
0.256	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0022	<0.05	<0.05	0.070	<0.05	CATC-7B-2
0.260	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0037	<0.05	<0.05	0.068	<0.05	CATC-7B-3
0.264	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0025	<0.05	<0.05	0.060	<0.05	CATC-7B-4
0.267	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0007	<0.05	<0.05	0.062	<0.05	CATC-7B-5
0.268	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0012	<0.05	<0.05	0.075	<0.05	CATC-7B-6
0.271	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0245	<0.05	<0.05	0.087	<0.05	CATC-7B-7
0.276	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0229	<0.05	<0.05	0.056	<0.05	CATC-7B-8
0.280	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0389	<0.05	<0.05	0.121	<0.05	CATC-7B-9
0.286	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0020	<0.05	<0.05	<0.05	<0.05	CATC-7B-10
QA Stds.												
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	1.97	<0.05	<0.05	<0.05	<0.05	CATC-1A-1
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.233	<0.05	<0.05	<0.05	<0.05	CATC-1A-2
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.130	<0.05	<0.05	<0.05	<0.05	CATC-1A-3
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0670	<0.05	<0.05	<0.05	<0.05	CATC-1A-4
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.0338	<0.05	<0.05	<0.05	<0.05	CATC-1A-5
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.443	<0.05	<0.05	<0.05	<0.05	CATC-1A-6
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	1.06	<0.05	<0.05	<0.05	<0.05	CATC-1A-7
<0.05	<0.25	<0.1	<0.1	<0.05	<0.05	<0.1	0.683	<0.05	<0.05	<0.05	<0.05	CATC-1A-8
0.05	0.25	0.1	0.1	0.05	0.05	0.1	0.0002	0.05	0.05	0.05	0.05	Reporting Limit (mg/L)



1/15/03 JP

Solubility experiment samples collected in experiment URSB (p 209-211), matrix samples for this experiment (p 204-205) and QA standard solutes were sent to Div 01 for cation & anion analysis.

QA samples prepared on P258-261 were used and labeled as follows: Approximately 10 ml of the following standards were transferred to 30 ml PP bottles & labeled as shown below.

Standard (p259)	New label
CATC-1A-2	CAT-1A-2
CATC-1A-3	CAT-1A-3
CATC-1A-4	CAT-1A-4
CATC-1A-5	CAT-1A-5
CATC-1A-6	CAT-1A-6
CATC-1A-8	CAT-1A-8

ANL-1A-2	AN-1A-2
ANL-1A-3	AN-1A-3
ANL-1A-4	AN-1A-4
ANL-1A-5	AN-1A-5
ANL-1A-6	AN-1A-6
ANL-1A-7	AN-1A-7

The sample list/chain of custody forms for the samples submitted to Div 01 are shown on the following pages.

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim Prikey CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SwRI Contact	
						Mike Damann	
Analyses Requested							
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Cations by ICP, include Si	*Note: cations of particular interest and their range of concentration are listed below to assist in ICP calibration. U - 0.001 to 2.0 ppm Si - 25 to 45 ppm Ca - 350-550 ppm Al - 0.2 to 5.0 ppm K - 0.2 to 5.0 ppm
CAT-0A-1	10/17/02	0930	W		1	✓	POC Jim Prikey
CAT-0A-2					1	✓	x 5667
CAT-0A-3					1	✓	
CAT-0A-4					1	✓	
CAT-0A-5					1	✓	Nuclear safety related-use
CAT-0A-6					1	✓	appropriate QA procedures
CAT-0A-7					1	✓	
CAT-0A-8					1	✓	
CAT-0A-9					1	✓	
CAT-0A-10					1	✓	
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank					
Temp:		Therm #:		Received by (Print/Signature)		Date	Time
Comments:		325/210		Relinquished by (Print/Signature)		Date	Time
				Relinquished by (Print/Signature)		Date	Time

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Page 1 of 3

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY				Requested Turnaround:	
Jim Prikey CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166				<input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input checked="" type="checkbox"/> Other: 4 weeks	
Client		Client Purchase Order/Other ID		Site/Zone ID		SwRI Contact	
						Mike Damann	
Analyses Requested							
Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Cations by ICP, include Si	* see note on page 1 U - 0.001 to 2.0 ppm Si - 25 to 45 ppm Ca - 350-550 ppm Al - 0.2 to 5.0 ppm K - 0.2 to 5.0 ppm
CAT-7B-1	10/17/02	0930	W		1	✓	POC Jim Prikey
CAT-7B-2					1	✓	x 5667
CAT-7B-3					1	✓	
CAT-7B-4					1	✓	
CAT-7B-5					1	✓	Nuclear safety related-use
CAT-7B-6					1	✓	appropriate QA procedures
CAT-7B-7					1	✓	
CAT-7B-8					1	✓	
CAT-7B-9					1	✓	
CAT-7B-10					1	✓	
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)		Date	Time
A - Air B - Biota D - Dust E - Emission/Stack P - Product S - Soil SED - Sediment T - Tissue W - Water WP - Wipe		D - Duplicate ER - Equipment Rinse FB - Field Blank FD - Field Duplicate MS - Matrix Spike MSD - Matrix Spike Dup TB - Trip Blank					
Temp:		Therm #:		Received by (Print/Signature)		Date	Time
Comments:		325/210		Relinquished by (Print/Signature)		Date	Time
				Relinquished by (Print/Signature)		Date	Time

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Shipper Name / Address <div style="font-size: 1.2em; margin-top: 5px;">Jim Pickryl CNRPA, Div 20, Bldg 57</div>		<b>SAMPLE LIST/CHAIN OF CUSTODY</b> Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166										Requested Turnaround: <input type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks Other: <span style="border: 1px solid black; padding: 2px;">1 week</span>			
Client		Client Purchase Order/Other ID					Site/Zone ID					SWRI Contact <div style="font-size: 1.1em; margin-top: 5px;">Mike Dumanow</div>			
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	Analyses Requested					REMARKS			
AN-7B-1	10/17/02	09:00	W			1	✓	ANIONIC BY IC							Preservation a = HCl to pH <2 b = HNO <sub>3</sub> to pH <2 c = H <sub>2</sub> SO <sub>4</sub> to pH <2 d = NaOH to pH >12 e = Cool (4°C±2°C) f = Other (specify)
AN-7B-2						1	✓							aoc - Jim Pickryl x SLUT	
AN-7B-3						1	✓								
AN-7B-4						1	✓								
AN-7B-5						1	✓								
AN-7B-6						1	✓								
AN-7B-7						1	✓								
AN-7B-8						1	✓								
AN-7B-9						1	✓								
AN-7B-10						1	✓								Nuclear safety related - use appropriate QA procedures.  20.06002.01.071
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)					Date Time		SWRI Project:				
B - Biota		ER - Equipment Rinseate		Received by (Print/Signature)					Date Time		Received by SWRI Lab: (Signature)				
D - Dust		FB - Field Blank		Relinquished by (Print/Signature)					Date Time		Date Time				
E - Emission/Stack		FD - Field Duplicate		Received by (Print/Signature)					Date Time		Date Time				
P - Product		MS - Matrix Spike		Relinquished by (Print/Signature)					Date Time		Date Time				
S - Soil		MSD - Matrix Spike Dup		Received by (Print/Signature)					Date Time		Date Time				
SED - Sediment		TB - Trip Blank		Relinquished by (Print/Signature)					Date Time		Date Time				
T - Tissue				Received by (Print/Signature)					Date Time		Date Time				
W - Water				Relinquished by (Print/Signature)					Date Time		Date Time				
WP - Wipe				Relinquished by (Print/Signature)					Date Time		Date Time				
Temp:		Therm #:		Relinquished by (Print/Signature)					Date Time		Date Time				
Comments:				Relinquished by (Print/Signature)					Date Time		Date Time				

1/20/03 JP

Portions of the recovered solids from solubility experiments URSB + URSC were taken to Div 18 for XRD analysis.

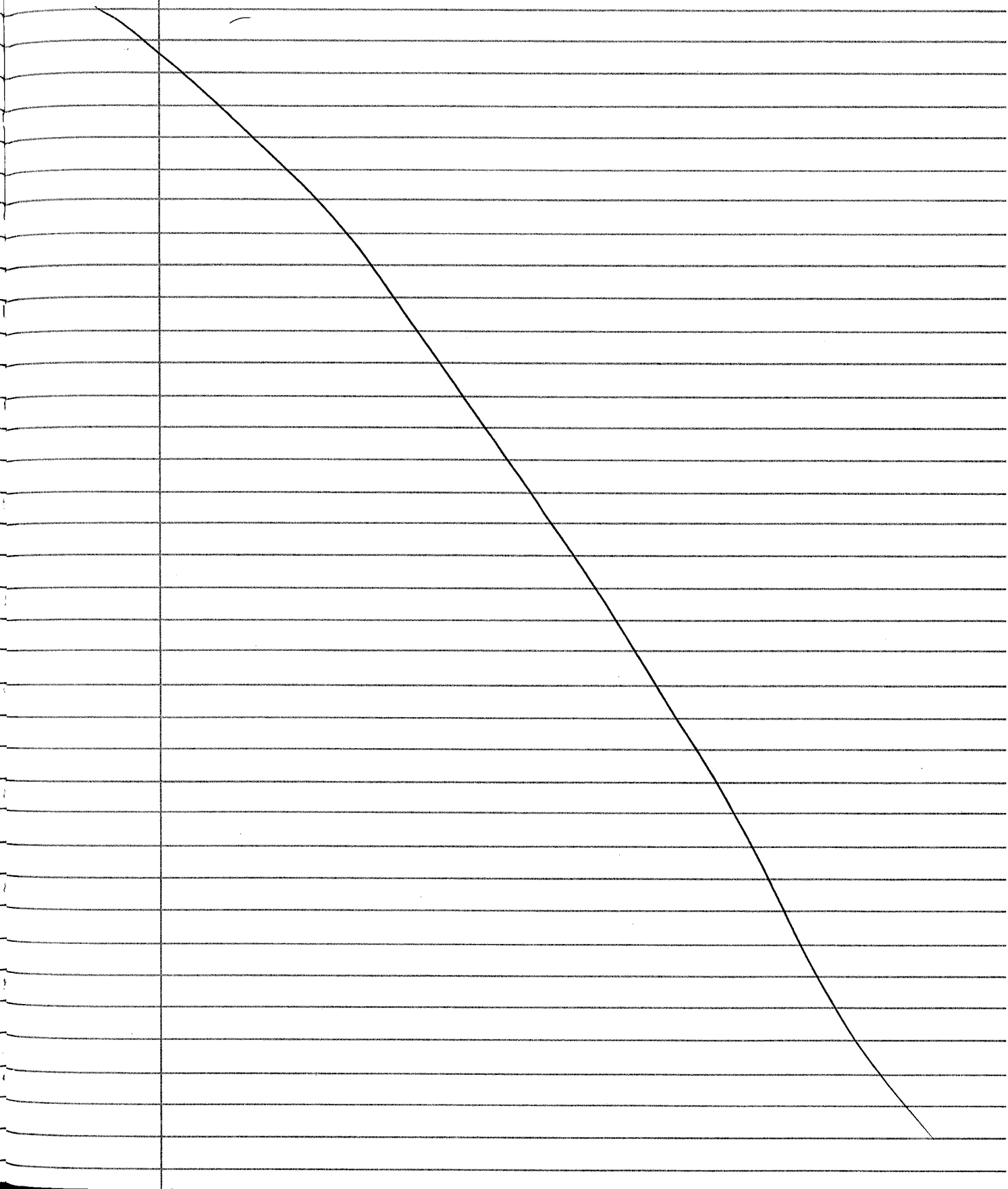
### Preparation -

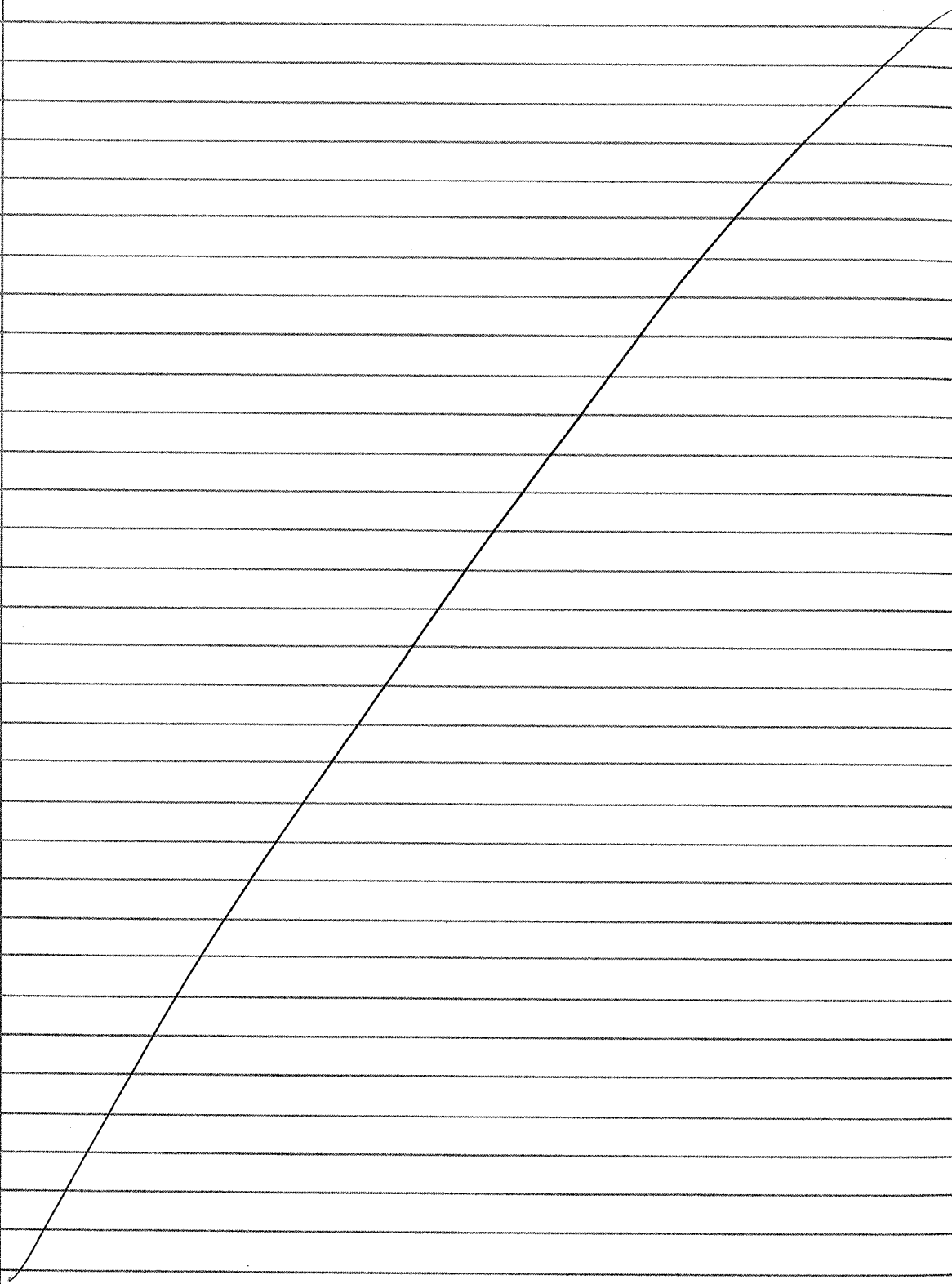
Portions of the samples listed below were crushed in an agate mortar & pestle & placed in plastic vials labeled as shown below.

Original sample (p234 + 236)	Label for XRD analysis
URSB-0A-S	URSB-0A-XRD
URSB-7B-S	URSB-7B-XRD
URSC-0A-S	URSC-0A-XRD
URSC-0B-S	URSC-0B-XRD
URSC-7A-S	URSC-7A-XRD
URSC-7B-S	URSC-7B-XRD

Samples were delivered to Jim Spencer in Div 18 for XRD analysis.

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1/21/03 JF

Results of urine analyses conducted by Div 01.

## Results of chemical analyses of samples from uranophane solubility experiment URSC

All analyses are in mg/L

Sample ID	Fluoride	Chloride	Nitrite-N	Bromide	Nitrate-N	Phosphate-P	Sulfate
<i>Matrix Solns</i>							
UMC-0C	0.128	706	<5	<0.1	0.220	<0.1	0.199
UMC-0D	0.103	706	<5	<0.1	0.211	<0.1	0.117
UMC-7C	0.107	705	<5	<0.1	0.125	<0.1	0.174
UMC-7D	0.116	733	<5	<0.1	0.106	<0.1	0.164
<i>Sample Solns</i>							
ANC-0A-1	0.264	717	<5	<0.1	0.140	<0.1	0.355
ANC-0A-2	0.333	769	<5	<0.1	0.297	<0.1	0.379
ANC-0A-3	0.339	733	<5	<0.1	0.261	<0.1	0.223
ANC-0A-4	0.301	742	<5	<0.1	0.296	<0.1	0.254
ANC-0A-5	0.279	785	<5	<0.1	0.325	<0.1	0.306
ANC-0A-6	0.350	758	<5	<0.1	0.272	<0.1	0.305
ANC-0A-7	0.365	766	<5	<0.1	0.293	<0.1	0.289
ANC-0A-8	0.362	792	<5	<0.1	0.335	<0.1	0.350
ANC-0A-9	0.373	803	<5	<0.1	0.298	<0.1	0.306
ANC-0A-10	0.370	810	<5	<0.1	0.293	<0.1	0.296
ANC-0B-1	0.292	722	<5	<0.1	0.290	<0.1	0.298
ANC-0B-2	0.284	745	<5	<0.1	0.123	<0.1	0.479
ANC-0B-3	0.292	745	<5	<0.1	0.278	<0.1	0.313
ANC-0B-4	0.327	747	<5	<0.1	0.314	<0.1	0.250
ANC-0B-5	0.303	759	<5	<0.1	0.290	<0.1	0.235
ANC-0B-6	0.339	781	<5	<0.1	0.266	<0.1	0.266
ANC-0B-7	0.360	785	<5	<0.1	0.295	<0.1	0.238
ANC-0B-8	0.326	801	<5	<0.1	0.290	<0.1	0.305
ANC-0B-9	0.358	819	<5	<0.1	0.304	<0.1	0.238
ANC-0B-10	0.372	845	<5	<0.1	0.351	<0.1	0.310
ANC-7A-1	0.313	736	<5	<0.1	0.100	<0.1	0.377
ANC-7A-2	0.308	742	<5	<0.1	<0.1	<0.1	0.543
ANC-7A-3	0.342	739	<5	<0.1	0.165	<0.1	0.244
ANC-7A-4	0.319	753	<5	<0.1	0.179	<0.1	0.285
ANC-7A-5	0.321	769	<5	<0.1	0.272	<0.1	0.307
ANC-7A-6	0.365	788	<5	<0.1	0.176	<0.1	0.328
ANC-7A-7	0.365	764	<5	<0.1	0.216	<0.1	0.334
ANC-7A-8	0.360	783	<5	<0.1	0.235	<0.1	0.358
ANC-7A-9	0.396	810	<5	<0.1	0.195	<0.1	0.644
ANC-7A-10	0.316	847	<5	<0.1	0.225	<0.1	0.305
ANC-7B-1	0.318	733	<5	<0.1	0.159	<0.1	0.236
ANC-7B-2	0.288	719	<5	<0.1	0.199	<0.1	0.372
ANC-7B-3	0.335	710	<5	<0.1	0.202	<0.1	0.297
ANC-7B-4	0.364	756	<5	<0.1	0.219	<0.1	0.298
ANC-7B-5	0.340	746	<5	<0.1	0.171	<0.1	0.306
ANC-7B-6	0.344	873	<5	<0.1	0.231	<0.1	0.344
ANC-7B-7	0.304	805	<5	<0.1	0.206	<0.1	0.396
ANC-7B-8	0.350	797	<5	<0.1	0.197	<0.1	0.382
ANC-7B-9	0.380	905	<5	<0.1	0.224	<0.1	0.363
ANC-7B-10	0.390	822	<5	<0.1	0.222	<0.1	0.283
<i>QA Stds.</i>							
ANC-1A-1	<0.1	713	<5	<0.1	0.414	<0.1	<0.1
ANC-1A-2	<0.1	815	<5	<0.1	<0.1	<0.1	<0.1
ANC-1A-3	<0.1	676	<5	<0.1	0.246	<0.1	<0.1
ANC-1A-4	<0.1	730	<5	<0.1	0.122	<0.1	<0.1
ANC-1A-5	<0.1	771	<5	<0.1	0.159	<0.1	<0.1
ANC-1A-6	<0.1	760	<5	<0.1	0.122	<0.1	<0.1
ANC-1A-7	<0.1	719	<5	<0.1	0.223	<0.1	<0.1
ANC-1A-8	<0.1	772	<5	<0.1	0.935	<0.1	4.63
Reporting Limit (mg/L)	0.1	5.0	5.0	0.1	0.1	0.1	0.1

1/23/03 JP

Results of U analyses of solution collected in solubility experiment UNSC suggest that U may be lost during filtering of the samples thru the 0.2  $\mu$ m PTFE membrane filter used in the experiments. For example, the U contents of solution vary widely & show no consistent trend with time. This is in contrast to other cations of interest (i.e. Ca & Si) which show consistent trends with time.

In order to test whether U is affected by the filtering process the experiment described in the following paper was conducted.

1/24/03 JP

## Filtering test FU

Obj - Determine if filter samples has an effect on U concentration of solutions due to sorption on the filter

Method - Filter U-bearing solution thru the filter and determine their U contents. One set of samples will be acidified to pH < 2.0 - a pH at which U sorption is not important. Another set of samples will have pHs of ~6.0 - a pH where U sorption is important.

## Equipment & reagents

- previously prepared  $10^{-3}$  M  $\text{SiO}_2$  soln (p73-74)
- $\text{CaCl}_2 \cdot \text{H}_2\text{O}$  (lot 015282)
- $\text{CaCO}_3$  (lot 986396)
- previously prepared  $10^{-4}$  M U soln (p204)
- 250 ml PP bottles
- Orion pH meter
- Orion combination pH electrode
- pH buffer solutions
- Mettler analytical balance AE240 + PM4600
- weighing boats & paper
- rotating shaker
- pipette & tips
- syringe filter; 0.2  $\mu$ m; Whatman PTFE

Procedure:

1/24/03  
1010hr  
JP

1) Label 6 250 ml PP bottles as follows.

$3.33 \times 10^{-6}$  mU  
 $1.0 \times 10^{-6}$  mU  
 $3.33 \times 10^{-7}$  mU  
 $1.0 \times 10^{-7}$  mU  
 $3.33 \times 10^{-8}$  mU  
 $1.0 \times 10^{-8}$  mU

1/24/03  
1020hr  
JP2) Transfer about 100 g of the  $10^{-3}$  M  $\text{SiO}_2$  solution to each bottle.1/24/03  
1030hr  
JP3) Add the following quantities of reagents +  $10^{-4}$  M U soln to the specified bottle.

Bottle label	$\text{CaCl}_2 \cdot \text{H}_2\text{O}$	$\text{CaCO}_3$	$10^{-4}$ M U soln
$3.33 \times 10^{-6}$ mU	.36734 g	.00014 g	7.5 g
$1.0 \times 10^{-6}$ mU	.36742 g	.00009 g	2.5 g
$3.33 \times 10^{-7}$ mU	.36744 g	.00007 g	0.75 g
$1.0 \times 10^{-7}$ mU	.36745 g	.00006 g	0.25 g
$3.33 \times 10^{-8}$ mU	.36746 g	.00005 g	0.075 g
$1.0 \times 10^{-8}$ mU	.36747 g	.00004 g	0.025 g

1/24/03  
1105hr  
JP4) After adding reagents make up to final wt of 250 g with the  $10^{-3}$  M  $\text{SiO}_2$  soln.1/26/03  
JP

5) Place bottle in gyratory shaker set at about 120 rpm. Place baffle over bottle opening to allow contact with air.

6) Periodically measure pH of solns to determine when they reach equilibrium with air.

Sample -  $3.33 \times 10^{-6}$  mU  $1.0 \times 10^{-6}$  mU  $3.33 \times 10^{-7}$  mU  $1.0 \times 10^{-7}$  mU  $3.3 \times 10^{-8}$  mU  $1.0 \times 10^{-8}$  mU

Date/Time

1/27/03 0800 5.86 5.92 5.65 5.99 5.74 5.65

1/28/03 1000 5.94 5.90 5.89 5.97 6.11 6.07

1/28/03  
JP 1015hr7) Label six 15 ml PP bottles as follows and transfer about 10 ml of each sample solution as shown below into the 15 ml PP bottles. After transfer add 20  $\mu$ l of 6M  $\text{HNO}_3$  to each bottle.

Label	Sample solution transferred
FU-1	$3.33 \times 10^{-6}$ mU
FU-2	$1.0 \times 10^{-6}$ mU
FU-3	$3.33 \times 10^{-7}$ mU
FU-4	$1.0 \times 10^{-7}$ mU
FU-5	$3.33 \times 10^{-8}$ mU
FU-6	$1.0 \times 10^{-8}$ mU

1/28/03 JP  
1030hr Label 6 15 ml PP bottles as shown below. Filter 10 ml of each sample shown below thru a Whatman 0.2  $\mu$ m PTFE syne filter + place in 15 ml PP bottle. After filtering acidified each sample with 20  $\mu$ l 6M  $\text{HNO}_3$ .

Label	Sample soln transferred
FU-7	$3.33 \times 10^{-6}$ mU
FU-8	$1.0 \times 10^{-6}$ mU
FU-9	$3.33 \times 10^{-7}$ mU
FU-10	$1.0 \times 10^{-7}$ mU
FU-11	$3.33 \times 10^{-8}$ mU
FU-12	$1.0 \times 10^{-8}$ mU

1/28/03 JP  
1045hr Label 6 15 ml PP bottles as follows. Transfer 10 ml of each sample solution to a 20 ml PP beaker & acidify with 20  $\mu$ l 6M  $\text{HNO}_3$ . Allow to stand for several minutes. Label Then filter the acidified soln thru a 0.2  $\mu$ m PTFE filter into each 15 ml PP bottle.

Label	Sample soln transferred
FU-13	$3.33 \times 10^{-6}$ mU
FU-14	$1.0 \times 10^{-6}$ mU
FU-15	$3.33 \times 10^{-7}$ mU
FU-16	$1.0 \times 10^{-7}$ mU
FU-17	$3.33 \times 10^{-8}$ mU
FU-18	$1.0 \times 10^{-8}$ mU

1/28/03 JP  
1130hr

Samples FU-1 thru FU-18 were sent to Div 01 for ICP/MS analysis of U. Since this is a test to determine whether U is affected by filtering and data will not be used in any analyses for the Yucca Mountain project, nuclear safety related QA procedures were not conducted for these samples.

The sample list/chart of custody form for the samples submitted to Div 01 are shown on the following page.

Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:	
Jim Pirkyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		<input checked="" type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input type="checkbox"/> Other:	
Client		Client Purchase Order/Other ID		Site/Zone ID	
Analyses Requested		SwRI Contact		REMARKS	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type
FU-1		1/23/03	1000	W	
FU-2					
FU-3					
FU-4					
FU-5					
FU-6					
FU-7					
FU-8					
FU-9					
FU-10					
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)	
A - Air		D - Duplicate		Date	
B - Biota		ER - Equipment Rinse		Time	
D - Dust		FB - Field Blank		Received by (Print/Signature)	
E - Emission/Stack		FD - Field Duplicate		Date	
P - Product		MS - Matrix Spike		Time	
S - Soil		MSD - Matrix Spike Dup		Relinquished by (Print/Signature)	
SED - Sediment		TB - Trip Blank		Date	
T - Tissue				Time	
W - Water				Received by (Print/Signature)	
WP - Wipe				Date	
Temp:		Therm #:		Time	
Comments:				Samples Disposed:	
325/285				Date	
				Time	
				Samples Disposed by:	
				Date	
				Time	

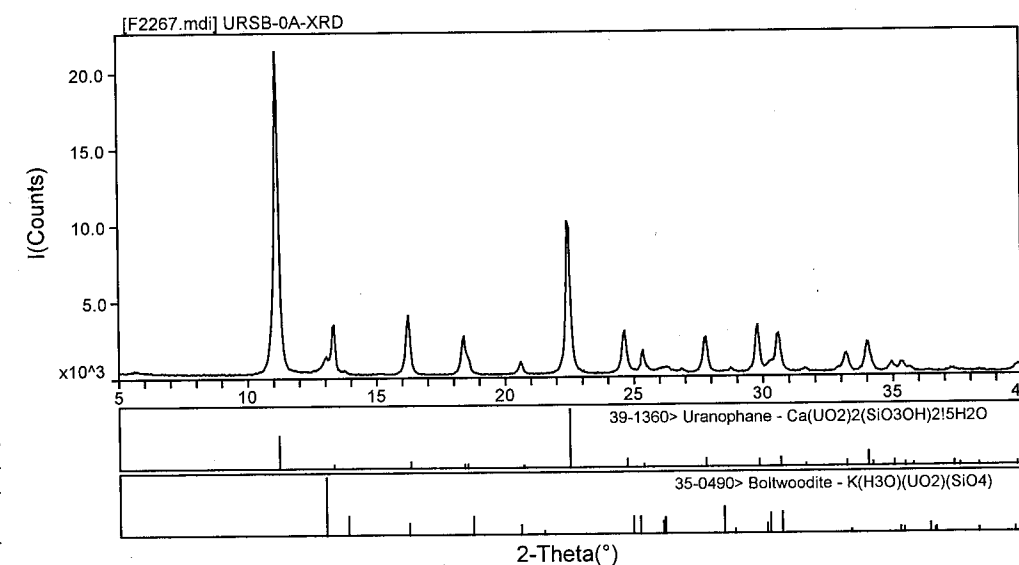
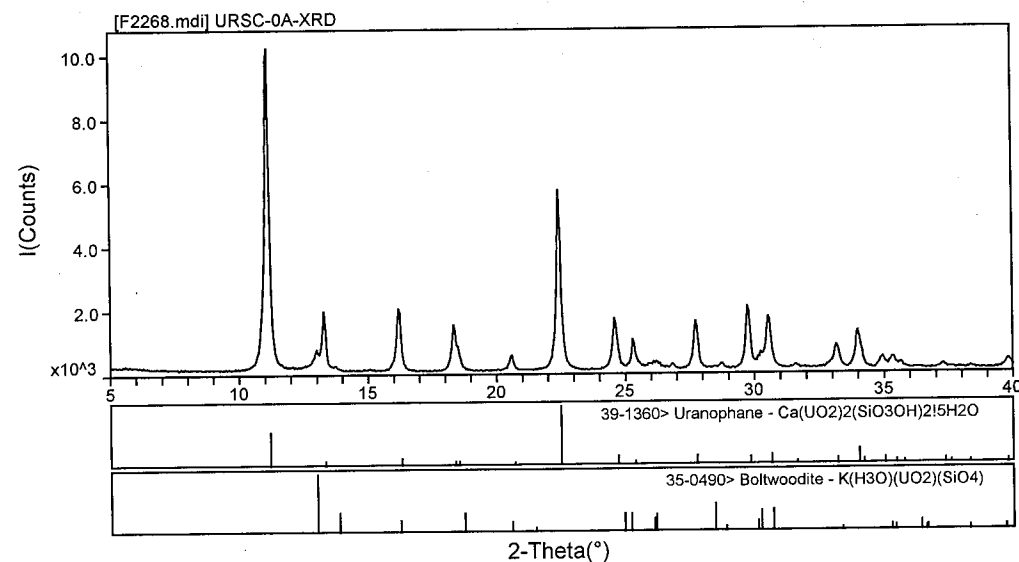
Div 01 COC Form 01-01-001, Rev 4/02

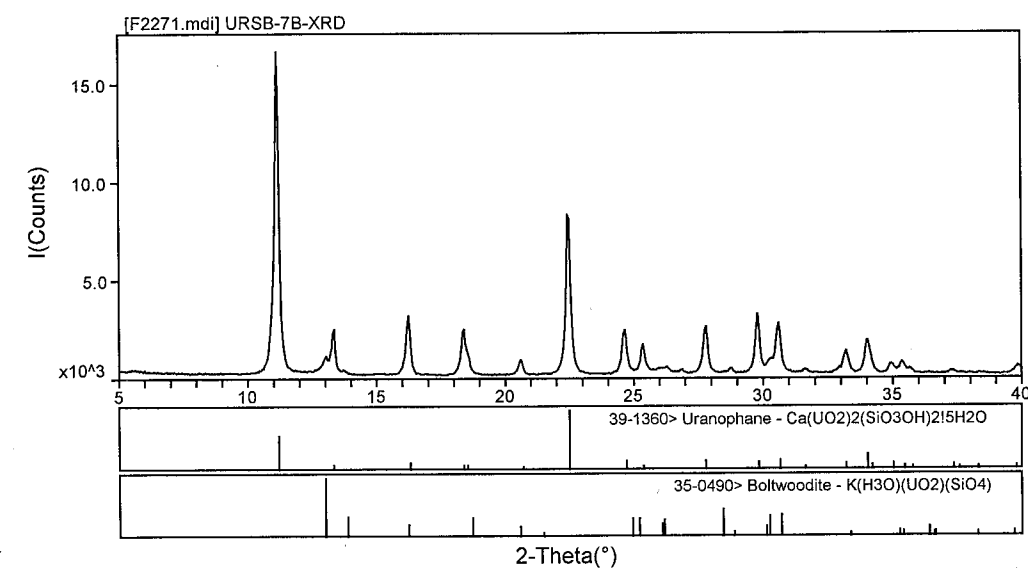
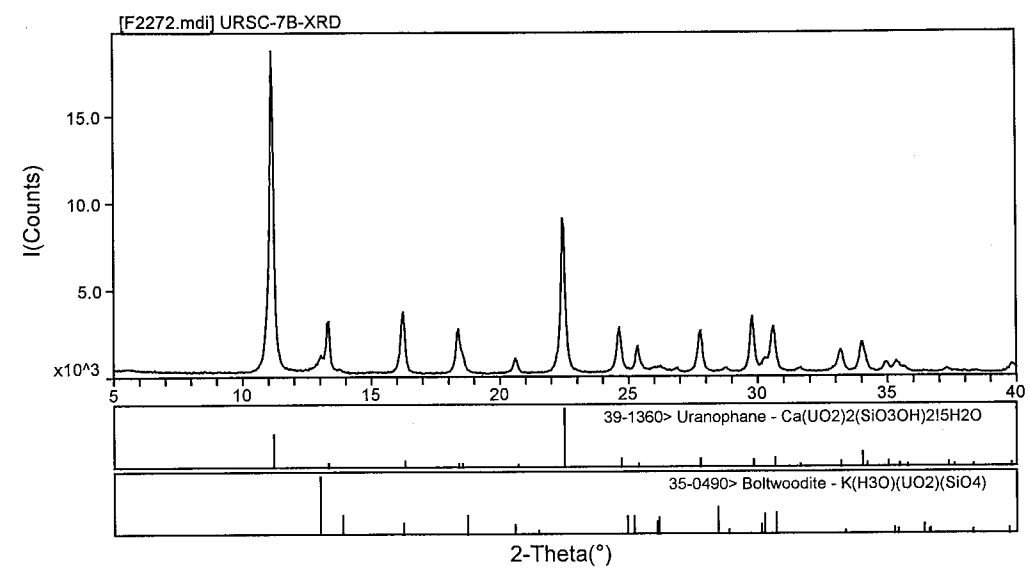
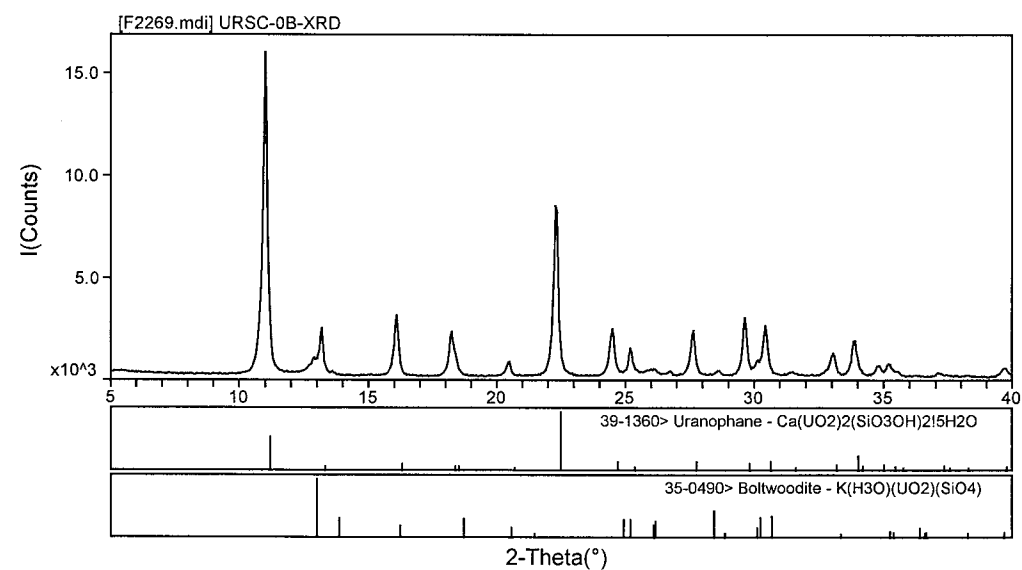
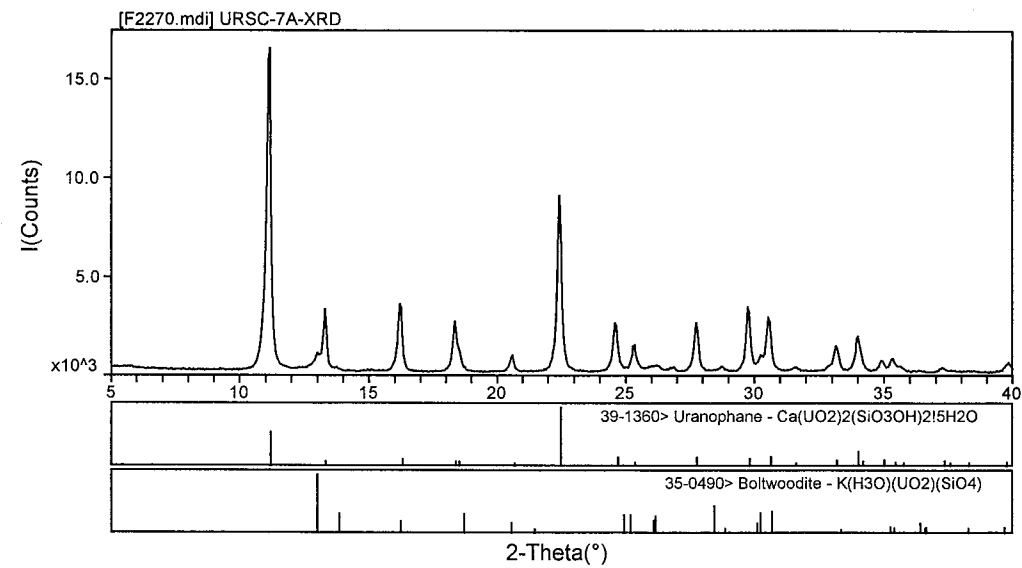
Shipper Name/Address		SAMPLE LIST/CHAIN OF CUSTODY		Requested Turnaround:	
Jim Pirkyl CNWRA, Div 20, Bldg 57		Southwest Research Institute Chemistry and Chemical Engineering Division 6220 Culebra Road San Antonio, Texas 78238-5166		<input checked="" type="checkbox"/> 2 Weeks <input type="checkbox"/> 3 Weeks <input type="checkbox"/> Other:	
Client		Client Purchase Order/Other ID		Site/Zone ID	
Analyses Requested		SwRI Contact		REMARKS	
Sample ID		Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type
FU-11		1/23/03	1000	W	
FU-12					
FU-13					
FU-14					
FU-15					
FU-16					
FU-17					
FU-18					
Matrix Types:		Sample Types:		Relinquished by (Print/Signature)	
A - Air		D - Duplicate		Date	
B - Biota		ER - Equipment Rinse		Time	
D - Dust		FB - Field Blank		Received by (Print/Signature)	
E - Emission/Stack		FD - Field Duplicate		Date	
P - Product		MS - Matrix Spike		Time	
S - Soil		MSD - Matrix Spike Dup		Relinquished by (Print/Signature)	
SED - Sediment		TB - Trip Blank		Date	
T - Tissue				Time	
W - Water				Received by (Print/Signature)	
WP - Wipe				Date	
Temp:		Therm #:		Time	
Comments:				Samples Disposed:	
325/285				Date	
				Time	
				Samples Disposed by:	
				Date	
				Time	

Div 01 COC Form 01-01-001, Rev 4/02

2/10/03 JP

Results of XRD analysis of recovered solids from experiments URS B + URS C (see p. 278) are shown on the following pages.







2/12/03

Results of filtering test to determine U retention  
on PTFE syringe filter used in uracil  
solubility experiment are shown below.

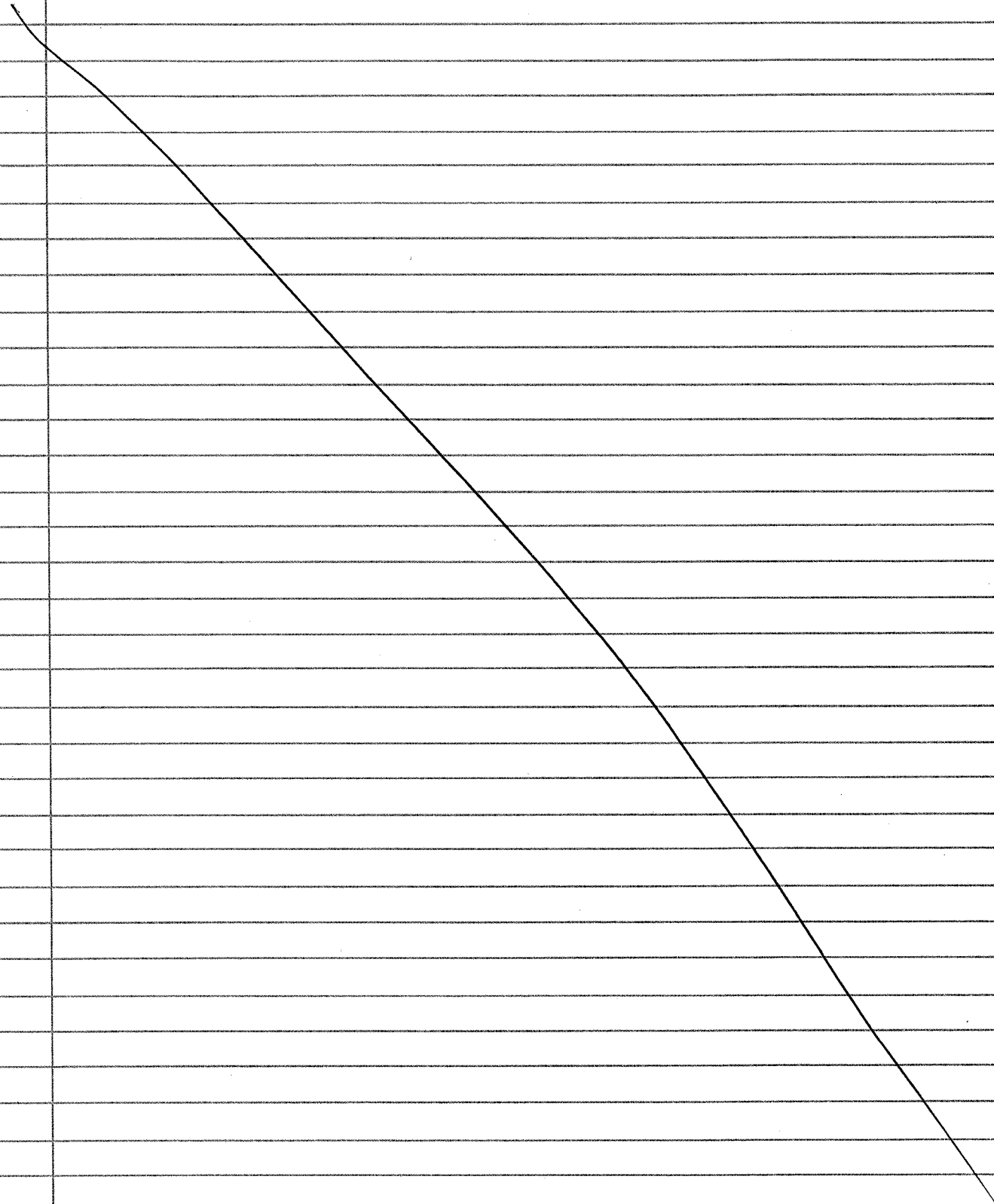
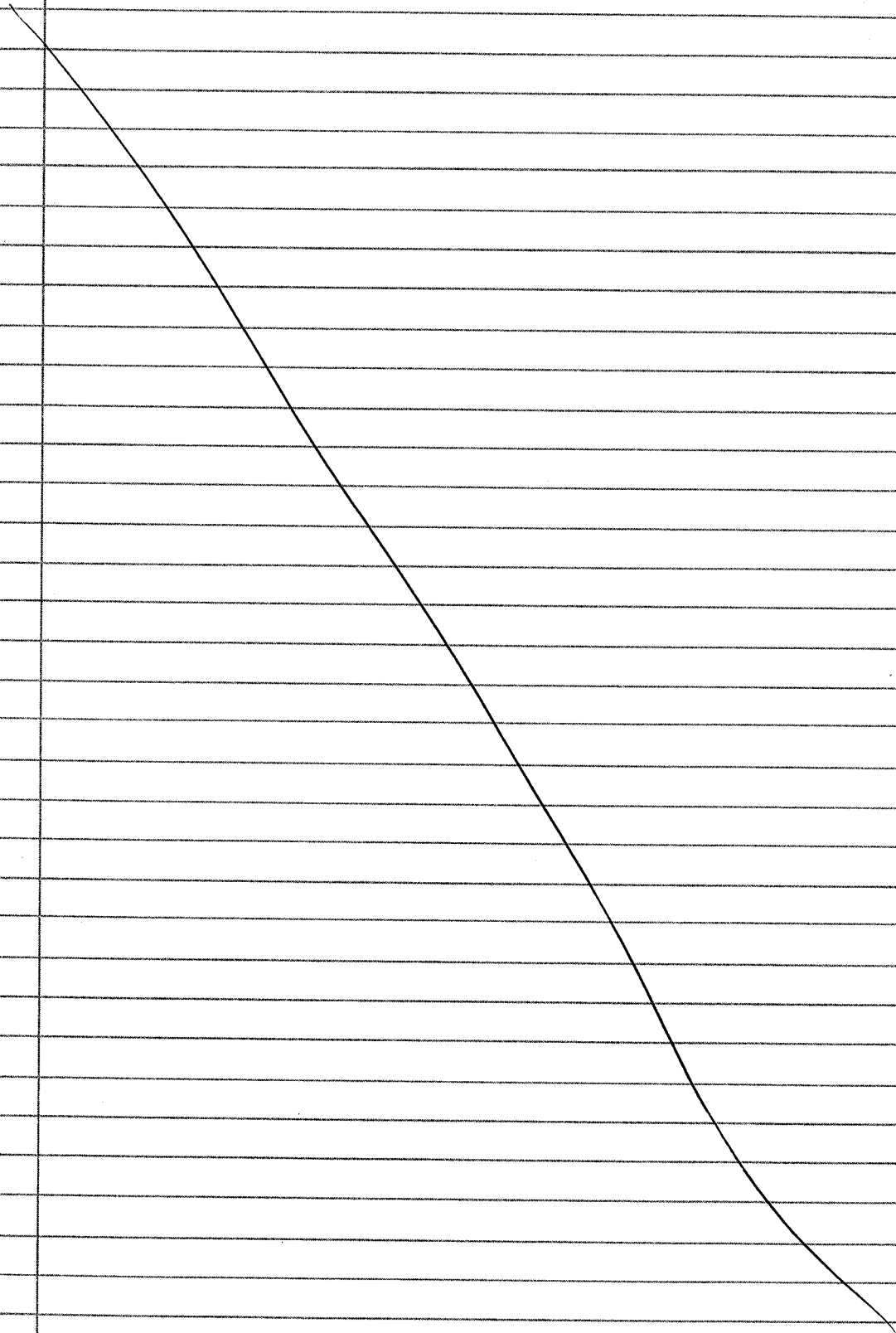
# Results of filtering test to determine retention of U on PTFE syringe filters

Nonfiltered sample labels	Measured U conc (ppb)	Filtered and acidified sample labels	Measured U conc (ppb)	Acidified and filtered sample labels	Measured U conc (ppb)
FU-1	708	FU-7	3.16	FU-13	717
FU-2	198	FU-8	1.31	FU-14	192
FU-3	72.3	FU-9	0.398	FU-15	72.1
FU-4	22.2	FU-10	0.44	FU-16	23.1
FU-5	4.37	FU-11	<0.2	FU-17	4.23
FU-6	1.26	FU-12	0.419	FU-18	1.21

Reporting limit: 0.2 ppb

The above results clearly indicate that  
U is retained on the PTFE filter if  
it is the U solution is not acidified  
prior to filtering. Measured U concentrations  
are essentially the same if the  
solution is acidified prior to filtering.

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Preparation of solutions for uranophane solubility experiment URSD

Written by: J. D. Prikryl  
Date: 02/13/03

Objective: Prepare 2 solutions containing U, Ca, and SiO<sub>2</sub> for use in the uranophane solubility experiment URSD. The solutions will have a Ca content of 10<sup>-2</sup> M and a SiO<sub>2</sub> content of 10<sup>-3</sup> M. U content will be 10<sup>-7</sup> M in one solution and U will be absent in the other solution. The pH of the solutions will be approximately 6.0.

Method: Addition of chemical reagents containing Ca and U to an approximate 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Materials and Equipment:

- previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution (see p. 73-74)
- CaCl<sub>2</sub>·2H<sub>2</sub>O; F.W. 147.02; (lot no. 015282)
- UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O; F.W. 502.13; (lot no. D19214)
- CaCO<sub>3</sub>; F.W. 100.09; (lot no. 986396)
- Polycarbonate bottles; various volumes
- Polypropylene bottles (15 ml)
- Orion pH meter
- Orion combination pH electrode
- ATC probe
- pH buffer solutions
- Mettler analytical balance (AE240) + PM400
- weighing paper and boats
- Fisher Versabath Model 236
- Gyatory shaker

Procedure

- 2/14/03 1300hr
1. Transfer 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution to a 250 ml polycarbonate bottle. Add 0.00502 g of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to the bottle. Label the bottle as 10<sup>-4</sup> m U.
  2. Label 2 500 ml polycarbonate bottles as follows:  
2/14/03 1320hr  
1.0x10<sup>-7</sup> m U  
0.0 m U
  3. 2/14/03 1335hr  
Transfer about 100 g of the previously prepared 10<sup>-3</sup> M SiO<sub>2</sub> solution into each 500 ml bottle.
  4. 2/14/03 1337hr  
Carefully add the following quantities of reagents and 10<sup>-4</sup> M U solution to the specified bottle. Use weighing paper and weigh out reagents as accurately as possible. For the 10<sup>-4</sup> M U solution use plastic weighing boats and rinse boats using the 10<sup>-3</sup> M SiO<sub>2</sub> solution. After adding reagents make up to a final weight of 500 g with the 10<sup>-3</sup> M SiO<sub>2</sub> solution.

Bottle Label	CaCl <sub>2</sub> ·2H <sub>2</sub> O	CaCO <sub>3</sub>	10 <sup>-4</sup> M U solution
1.0x10 <sup>-7</sup> m U	0.73490 g	0.00013 g	0.5 g
0.0 m U	0.73491 g	0.00013 g	0.0 g

- 2/14/03 1345hr.
5. Place bottles in shaker water bath set at 25 °C. Allow solutions to equilibrate with air by loosely capping bottles.

- 6. Every 3 or 4 days take 5 ml aliquots from each bottle and use to measure and record pH to determine when solutions reach equilibrium with air.
- 7. When equilibrium is reached (i.e., when pH is stable) record the weight of each U matrix solution bottle.

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	
0.0 m U	

- 8. Take two 5ml sample aliquots from each U matrix solution and place in 15 ml polypropylene bottles for cation analysis by ICP. Use an Oxford pipettor and 5 ml plastic tips to take samples. Preserve the samples for cation analysis by adding 10 µL of 6 M HNO<sub>3</sub> to each 15 ml sample bottle. Chemical analysis will show the initial Ca, SiO<sub>2</sub>, and U contents of the solutions before addition of uranophane. Label the samples as shown below.

Label	Analysis type	U matrix solution sampled
UMD-7A and UMD-7B	Cations (ICP)	1.0x10 <sup>-7</sup> m U
UMD-0A and UMD-0B	Cations (ICP)	0.0 m U

- 9. After sampling record the weight of each U matrix solution bottle.

Bottle	Wt (g)
1.0x10 <sup>-7</sup> m U	535.18
0.0 m U	533.62

pH measurements

Date/time	0.0 m U	1.0x10 <sup>-7</sup> m U
2/17/03 0200hrs.	5.83	5.68
2/19/03 0915hrs.	5.67	5.88
2/21/03 1055hrs.	6.20	6.32
2/24/03 0715hrs.	6.11	6.20
3/5/03 0930hrs.	5.92	5.99

Uranophane solubility experiment URSD

Written by: J. D. Prikryl  
Date: 02/13/03

Objective: Determine the solubility of synthesized uranophane.  
Method: Monitor the reaction of synthesized uranophane with solutions containing U, Ca, and SiO<sub>2</sub>.

- Materials and Equipment:
- Synthesized uranophane (URANOPHANE-7)
  - Previously prepared U matrix solutions with U concentrations of 0.0 m and 10<sup>-7</sup> m
  - Polycarbonate bottles (250 ml)
  - Orion pH meter
  - Orion combination pH electrode
  - ATC probe
  - pH buffer solutions
  - gyratory shaker
  - Mettler analytical balance (AE240 and PM4600)
  - Pipets and pipet tips
  - Plasticware as needed

Procedure

- 1. Label 4 250 ml polycarbonate bottles as follows and record weights.

3/5/03  
1030hrs

Label	Weight (g)
URSD-0A	51.36
URSD-0B	51.49
URSD-7A	51.32
URSD-7B	51.48

- 2. To each sample bottle add 0.5±0.0010 g of synthesized uranophane. Record the weight of uranophane added to each bottle.

3/5/03  
1035hrs

Bottle	Wt of uranophane (g)
URSD-0A	0.5010
URSD-0B	0.5006
URSD-7A	0.5009
URSD-7B	0.5006

- 3. To each bottle add 200±0.05 g of the specified U matrix solution shown in the table below. Record the weight of matrix solution added.

3/5/03  
1040hrs.

Bottle	U matrix solution	Wt of U matrix solution (g)
URSD-0A	0.0 m U	200.03
URSD-0B	0.0 m U	200.04
URSD-7A	1.0x10 <sup>-7</sup> m U	200.04
URSD-7B	1.0x10 <sup>-7</sup> m U	200.01

4. Record the initial sample weights (bottle + synthesized uranophane + U matrix solution).

3/5/03  
1042 km.

Sample	Initial wt (g)
URSD-0A	251.85
URSD-0B	251.99
URSD-7A	251.82
URSD-7B	251.98

5. Cover bottle openings with parafilm with punched holes and place in a shaking water bath set at 25 °C.

6. At 1 week intervals:

- § The day before sampling, remove bottles from gyratory shaker and place next to balance
- § Allow bottles to sit overnight to allow any suspended solids to settle
- § For each sample bottle
  - § carefully remove parafilm cover
  - § place onto balance (avoid jostling that could resuspend solids)
  - § record the weight
  - § remove a 5 ml aliquot using a pipettor with a 5 ml tip, transfer to a 15 ml PP bottle, and preserve for cation analysis by adding 10 µl of 6 M HNO<sub>3</sub> to the 15 ml PP bottle. After several minutes draw the acidified sample up into the 5 ml tip used to take the sample, wait a few minutes, and expel the sample back into the 15 ml PP bottle. Discard 5 ml tip
  - § remove a second 5 ml aliquot using a pipettor with a 5 ml tip, transfer to a 10 ml sample cup, and use to measure pH. Discard sample and 5 ml tip after pH measurement
  - § record the weight of the bottle
- § Below are tables into which data can be recorded. Samples for cation analyses should be labeled as shown in the tables.

URSD-0A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/03 1019	249.68 g	239.69 g	9.99 g	5.94	URSD-0A-1
3/18/03 0736	238.31 g	227.99 g	10.32 g	5.81	URSD-0A-2
3/25/03 1121	225.87 g	215.84 g	10.03 g	5.72	URSD-0A-3
4/2/03 0914	213.02 g	202.65 g	10.37 g	5.71	URSD-0A-4
4/9/03 0916	200.35 g	190.08 g	10.22 g	5.59	URSD-0A-5
4/16/03 0920	188.34 g	177.76 g	10.58 g	5.61	URSD-0A-6
4/23/03 0800	175.63 g	165.60 g	10.03 g	5.61	URSD-0A-7
4/30/03 0925	163.46 g	153.27 g	10.39 g	5.55	URSD-0A-8
5/13/03 0910	150.65 g	140.17 g	10.48 g	5.44	URSD-0A-9
5/20/03 1320	138.68 g	128.62 g	10.06 g	5.45	URSD-0A-10
5/28/03 0258	126.85 g	116.83 g	10.02 g	5.46	URSD-0A-11
6/5/03 0925	115.11 g	105.06 g	10.05 g	5.47	URSD-0A-12
6/12/03 0905	103.53 g	93.49 g	10.04 g	5.43	URSD-0A-13

URSD-0B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/03 10:21	249.98 g	240.02 g	9.96 g	5.63	URSD-0B-1
3/18/03 07:39	238.55 g	228.56 g	9.99 g	5.50	URSD-0B-2
3/25/03 10:24	226.18 g	216.00 g	10.18 g	5.39	URSD-0B-3
4/2/03 09:20	213.24 g	202.70 g	10.54 g	5.24	URSD-0B-4
4/9/03 09:20	200.65 g	190.41 g	10.24 g	5.23	URSD-0B-5
4/16/03 09:21	188.17 g	177.89 g	10.28 g	5.18	URSD-0B-6
4/23/03 08:02	175.93 g	165.62 g	10.31 g	5.16	URSD-0B-7
4/30/03 09:27	163.29 g	152.77 g	10.52 g	5.09	URSD-0B-8
5/13/03 0914	149.57 g	139.52 g	10.05 g	5.04	URSD-0B-9
5/20/03 1324	137.77 g	127.94 g	9.83 g	5.01	URSD-0B-10
5/28/03 0901	125.88 g	115.87 g	10.01 g	4.99	URSD-0B-11
6/5/03 0927	113.51 g	103.58 g	9.93 g	4.97	URSD-0B-12
6/12/03 0907	101.41 g	91.39 g	10.02 g	4.92	URSD-0B-13

URSD-7A

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/03 10:24	248.93 g	238.66 g	10.27 g	5.63	URSD-7A-1
3/18/03 07:42	236.38 g	226.35 g	10.03 g	5.50	URSD-7A-2
3/25/03 11:26	222.34 g	211.82 g	10.52 g	5.35	URSD-7A-3
4/2/03 0922	207.46 g	196.93 g	10.53 g	5.27	URSD-7A-4
4/9/03 09:22	193.79 g	183.54 g	10.25 g	5.23	URSD-7A-5
4/16/03 09:23	179.68 g	169.41 g	10.27 g	5.14	URSD-7A-6
4/23/03 08:03	165.95 g	155.66 g	10.29 g	5.09	URSD-7A-7
4/30/03 09:29	153.47 g	143.47 g	10.00 g	5.06	URSD-7A-8
5/13/03 0915	140.35 g	130.34 g	10.01 g	4.99	URSD-7A-9
5/20/03 1325	128.58 g	119.02 g	9.56 g	4.99	URSD-7A-10
5/28/03 0903	116.97 g	106.96 g	10.01 g	4.96	URSD-7A-11
6/5/03 0930	104.86 g	94.84 g	10.02 g	4.93	URSD-7A-12
6/12/03 0910	93.06 g	83.17 g	9.89 g	4.89	URSD-7A-13

URSD-7B

Date / Time	Wt of bottle before sampling	Wt of bottle after sampling	Wt of solution removed	pH of solution removed	Sample Label
3/12/03 10:26	247.67 g	237.68 g	9.99 g	5.65	URSD-7B-1
3/18/03 07:44	234.99 g	224.68 g	10.31 g	5.52	URSD-7B-2
3/25/03 11:28	220.01 g	209.46 g	10.55 g	5.39	URSD-7B-3
4/2/03 0924	204.11 g	193.81 g	10.30 g	5.26	URSD-7B-4
4/9/03 09:24	190.90 g	180.34 g	10.56 g	5.22	URSD-7B-5
4/16/03 09:24	177.87 g	167.59 g	10.28 g	5.15	URSD-7B-6
4/23/03 0804	165.62 g	155.44 g	10.18 g	5.12	URSD-7B-7
4/30/03 0930	153.43 g	143.43 g	10.00 g	5.08	URSD-7B-8
5/13/03 0917	140.65 g	130.66 g	9.99 g	5.02	URSD-7B-9
5/20/03 1327	128.94 g	118.91 g	10.03 g	5.00	URSD-7B-10
5/28/03 0905	116.87 g	106.89 g	9.98 g	4.99	URSD-7B-11
6/5/03 0932	104.89 g	94.87 g	10.02 g	4.93	URSD-7B-12
6/12/03 0912	93.29 g	83.28 g	10.01 g	4.91	URSD-7B-13



5/19/03 JP

Preparation of central standards for monophase solubility experiment solution analysis.

Obj - prepare standard solutions with known concentrations of U, Ca, Si, Na, & K to determine accuracy of analysis of monophase solubility experiment solute collected in URS D experiment

Method - use certified standards to prepare solns with known concentrations of above cations.

Materials & equipment

- Volumetric flasks (50 ml)
- DI water
- PP bottles various sizes
- Eppendorf pipets & tips
- Fixed volume glass pipets
- conc.  $\text{HNO}_3$  (lot # 012229)
- Spex Certipur U Standard, 1000 ppm, lot # 9-93U
- Spex Certipur Si Standard, 1000 ppm, lot # 9-66SI
- Spex Certipur K Standard, 1000 ppm, lot # 9-03K
- Spex Certipur Na Standard, 1000 ppm, lot # 9-82NA
- Ion pure Ca Standard, 4003 ppm Ca, lot 922006

Procedure -

- ① Into 8 50ml volumetric flasks labeled as below add the volume of U, Si, K, Na, Ca standard as specified.

Standard Std (conc)	U (ml) 10 ppm	Si (ml) 1000 ppm	Ca (ml) 4003 ppm	Na (ml) 10 ppm	K (ml) 10 ppm
Label					
URSD-1A-1	10	1.5	4.75	1	5
URSD-1A-2	1	1.4	4.5	2	10
URSD-1A-3	0.5	2.0	6.0	5	4
URSD-1A-4	0.25	2.1	5.5	4	15
URSD-1A-5	0.1	1.8	5.75	7	2
URSD-1A-6	2.0	1.9	5.0	2	5
URSD-1A-7	5.0	1.6	5.25	0.5	1
URSD-1A-8	3.0	1.7	5.0	10	4

5/19/03 JP

- ② The table below shows the ppm value of U, Si, Ca, Na, & K for each standard soln.

Label	U ppm	Si ppm	Ca ppm	Na ppm	K ppm
URSD-1A-1	2.0	30	381	0.2	1.0
URSD-1A-2	0.2	28	361	0.4	2.0
URSD-1A-3	0.1	40	481	1.0	0.8
URSD-1A-4	0.05	42	441	0.8	3.0
URSD-1A-5	0.02	36	461	1.4	0.4
URSD-1A-6	0.4	38	401	0.4	1.0
URSD-1A-7	1.0	32	421	0.1	0.2
URSD-1A-8	0.6	34	401	2.0	0.8

- ③ Add 0.2 ml conc  $\text{HNO}_3$  to each flask & make up to mark with DI water.

- ④ Transfer central solution to 60 ml PP bottles with same labels as above (i.e. URSD-1A-1 thru URSD-1A-8)

5/19/03 gp

(5) Transfer ~10 ml of each standard to 15 ml bottles labeled the same as the standard & send to Div 01 for ICP analysis.

5/20/03 gp

Entries for uranophane solubility experiment  
URSD entries in scientific notebook  
#582 page 9.

I have reviewed this scientific notebook and find it in agreement with QAP-001.  
There is sufficient information regarding methods used for conducting tests,  
acquiring and analyzing data so that another qualified individual could repeat the  
activity.

ECP

9/18/03