

NF

308
Scientific Notebook # 144
Q200002040004

21
300

R

21
300

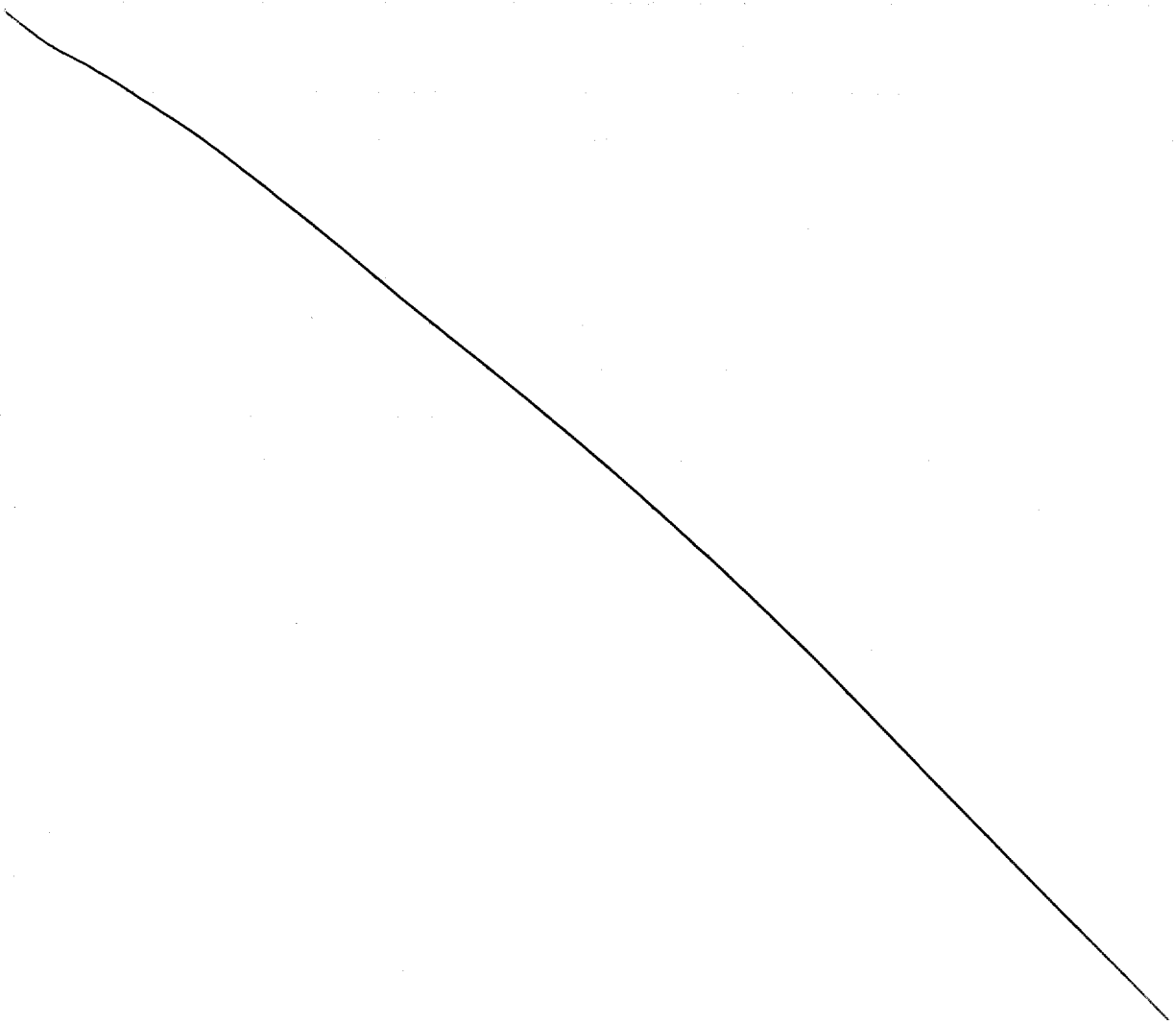
R

NF

4/7/95 JF Near-Field Processes and Variations
Research Project.

Initial entry 4/7/95 by James D. Luby JF

This notebook chronicles the laboratory
investigations of the Near-Field
Processes and Variations Research Project.



4/10/95 JP

The synthesis of uranophane (calcium uranyl silicate) will be attempted using procedure outlined on the following page.

If successful the uranophane will be used in later dissolution experiments.

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikril
REVISION NO.: 0

DATE WRITTEN: March 7, 1995
DATE REVISED:

OBJECTIVE: to synthesize uranophane $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 2.5\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 temperature controllers
Type J thermocouples
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 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 944231)
Sodium metasilicate nonahydrate $[\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}]$; MW=284.20 (lot 942853A)
Calcium acetate monohydrate $[\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$; MW=176.19
(lot 310617/1-194) 944231 JP
1 M HCl - diluted from lot 945500

JP 9/7/95
319 617/1 194

9/7/95

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 H₂O used, they state that good crystallinity is obtained if pH is less than 5.

Place 176 g Ca-acetate, 212 g U-acetate, and 142 g Na-metasilicate in a 2000 ml teflon liner and place liner in reaction vessel.

Cap and seal vessel and evacuate using a vacuum pump to remove CO₂(g).

Transfer approximately 1000 ml of ultrapure H₂O to a 1000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 1M HCl.

Degas the H₂O at a pH of 1.0 by boiling on a hotplate. After degassing cap erlenmeyer flask with a rubber stopper so that air cannot enter the flask.

0830 hrs
a)

0845 hrs
b)

0900 hrs
c)

0915 hrs
d)

4/10/95 JP

1130^{hr}
e)1135^{hr}
f)1400^{hr}
g)

After cooling record weight of the erlenmeyer flask with the degassed H₂O.

Transfer approximately 500 g of the degassed H₂O into the vessel by pulling in by vacuum. After the transfer, record weight of the erlenmeyer flask with remaining degassed H₂O to determine actual weight of H₂O introduced.

Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller and allow the reaction to proceed for one week or longer.

Weight of erlenmeyer flask with degassed H₂O = 1225.1 g
before transfer of H₂O to reaction vessel

Weight of erlenmeyer flask with degassed H₂O = 719.4 g
after transfer of H₂O to reaction vessel

Weight of degassed H₂O transferred to reaction vessel = 503.7 g

4/21/95 JP

Uranophane synthesis (cont).

0900
hr

The pressure vessel loaded with reagents + H₂O for uranophane synthesis was cooled and opened. The vessel contained and intermixed white and yellow-colored solid. No liquid was present.

An approximate 5 g portion of the solid was placed in a glass beaker and dried in an oven at ~60°C.

A second approximate 10 g portion of the intermixed solid was placed in a 100 ml glass beaker. This solid was washed with H₂O which caused the white-colored solid to dissolve leaving a yellow-colored solid. This yellow precipitate was dried in an oven at ~60°C.

4/24/95 JF

The two solid samples collected from the pressure vessel (see previous page) were analyzed by XRD to identify the phases present.

Equipment - Siemens D500 XRD housed in Div 06
Databox and JADE MDI software used to display data and determine minerals present.

Procedure - solids were powdered using a agate pestle and mortar. Powders were mounted in front loading glass sample holder.
Samples were then delivered to J. Spenser in Div 06 for processing.

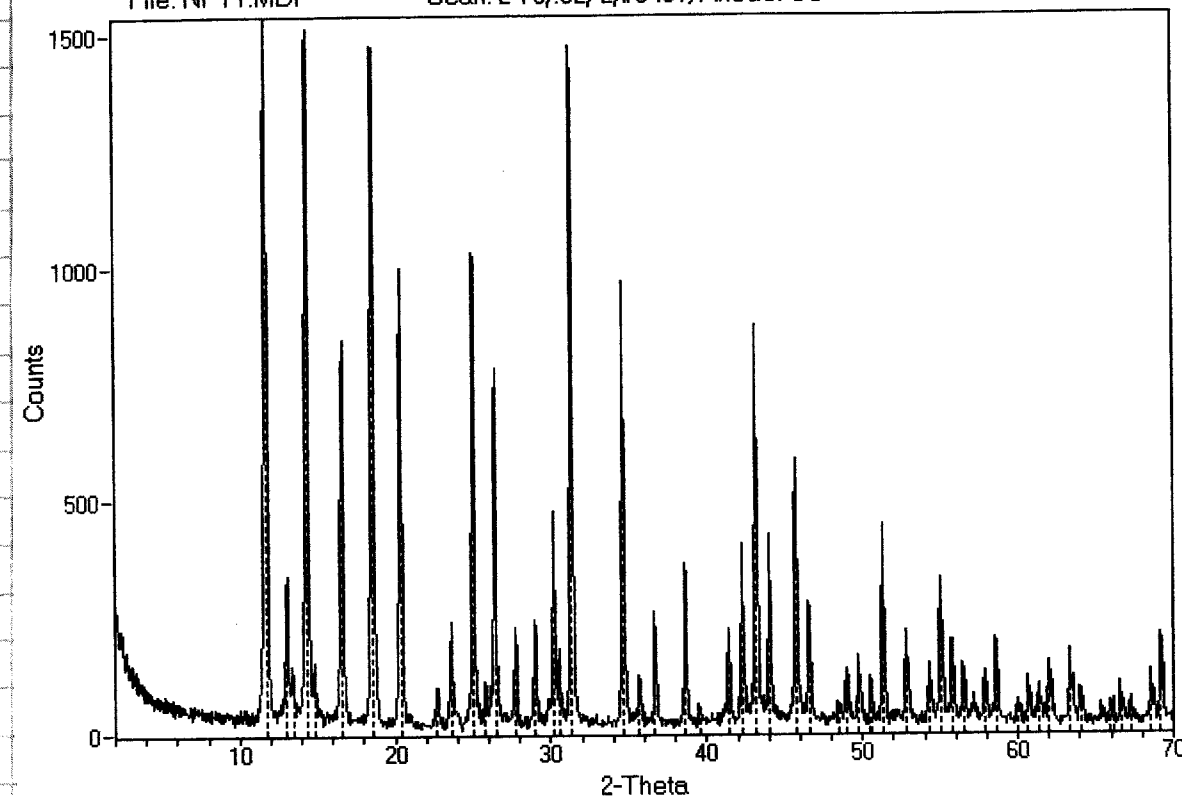
Results: results of XRD analyses are shown on the following pages.

Yellow solid

ID: 4/25/95 - NFY1-XRD1

File: NFY1.MDI

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



Sw Southwest Research Institute Wed Apr 26 1995 04:21pm

Jade: Hit List from Search/Match

NFY1.MDI> 4/25/95 - NFY1-XRD1

19 Hits Sorted on Figure-of-Merit	QW*	PDF-#	FOI	I3	OFF	RIR	Space-Group	a	b	c	Vol*	S	Dx*
1> Comptignacite, syn - K2(UO2)6O4	+	33-1049	10.7	54	-18		Pnmm (58)	12.19	14.85	7.16	1296	2	5.146
2> Becquerelite, syn - Ca(UO2)6O4(O	+	29-389	11.6	65	-12		Pna21 (33)	13.82	14.94	12.39	2558	4	5.116
3> Parascapelite, syn - UO2.8611.5H	+	23-1461	13.4	35	-12		Pcab (61)	15.04	18.98	14.23	4062	44	5.590
4> Boltwoodite - K(H3O)(UO2)(SiO4)	+	35-490	14.5	18	0.06		P21 (4)	7.07	7.06	6.64	319	2	4.372
5> Schoepite - UO3!2H2O	+	13-407	14.8	85	-42		Pcab (61)	14.73	16.79	14.33	3544	32	4.829
6> Ianthinite - U6O7(OH)2O	+	12-272	16.8	28	0.30		C (1)	11.62	30.30	7.15	1248	4	5.004
7> Metascapelite, syn - UO3!2H2O	+	43-364	17.3	76	-36		Pcab (61)	14.67	16.70	13.98	3424		
8> Schoepite, syn - UO3!2H2O	+	29-1376	17.5	28	-12		Pcab (61)	14.74	16.66	14.36	3526	32	4.853
9> Comptignacite - K2(UO2)6O4(OH)6	+	17-167	18.0	43	-30		Pnmm (58)	12.14	14.88	7.16	1293	2	5.157
10> Unnamed mineral (MR) - UO3-H2O	?	15-569	23.5	22	0.42								
11> Schoepite - UO3!2H2O	?	13-241	24.6	77	-24		Pcab (61)	14.73	16.79	14.33	3544	32	4.829
12> Metacalcicuranite - CaU2O7!11H2	+	26-1003	28.7	13	-06								
13> Metacalcicuranite - CaU2O7!11H2	+	8-301	30.0	22	0.30		P21/a (14)	14.01	15.55	6.64	1446	4	4.620
14> Uranophane-SOB - Ca(UO2)2(SiO3OH)2!5	+	39-1360	32.2	28	0.42		P21 (4)	15.92	7.01	6.67	739	2	3.849
15> Uranophane - Ca(UO2)2(SiO3OH)2!5	+	29-1044	33.8	14	-18		P212121 (19)	7.02	27.40	6.65	1279	8	4.100
16> Soddyite - (UO2)2(SiO4)!2H2O	+	35-491	40.8	36	0.24		Pddd (70)	11.21	18.71	8.32	436	5	5.087
17> Metastudtite - UO4!2H2O	+	35-571	41.0	41	0.18		Immm (71)	6.51	8.78	4.21	120	2	4.666
18> Uranosillite - USi7O17	+	37-417	49.4	36	-24		P21212 (18)	12.83	14.68	11.60	2185	6	3.222
19> Coffinite, syn - USiO4	+	11-420	53.7	33	-42		I41/amd (141)	6.98	6.98	6.25	152	4	7.201

* QW - ? for Doubtful Quality or Non-ambient patterns; + for Indexed patterns with Space-Group, Unit Cell & Density.
* vol - reduced-cell volume; dx - measured density if calculated is not available.

Current S/M parameters:

PDF Subfile to Search MINERALS
Chemistry Filter NF.CEM (Yes)
Search Focus on Major Phase(s)
Preferred Orientation in Sample No
Search only High Quality Phases No
Exclude Questionable Phases No

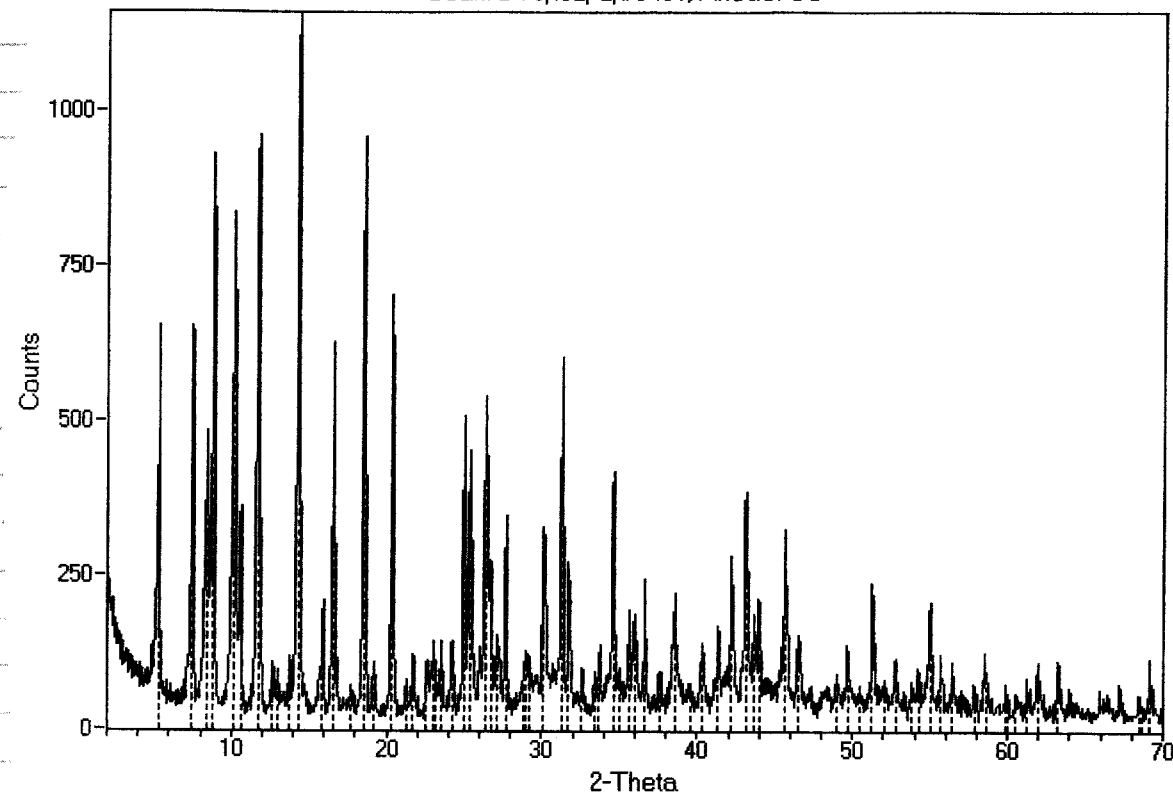
2-Theta Error Window42
Sensitivity to Matching Intensity 5
Sensitivity to Matching 2-Theta 5
Maximum Number of Hits to Save 40
Rubber-Ruler Search - Elasticity 0
Required 100% Line in Srded Range No

Intermixed white + yellow solids

ID: 4/25/95 - NFYW1-XRD1

File: NFYW1.MDI

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



XRD results indicate that the yellow solid consists of a combination of two or more uranium phases. The most likely phases appear to be a uranyl oxide hydrate (schoepite or becquerelite) and a K-rich uranyl silicate phase (e.g. boltwoodite).

The intermixed white + yellow pattern consists of the U phases described above plus white solids which may be unreacted starting reagents or other Ca-Si-Na phases.

Southwest Research Institute Wed Apr 26 1995 04:32pm

Jade: Hit List from Search/Match

NFYW1.MDI 4/25/95 - NFYW1-XRD1

40 Hits Sorted on Figure-Of-Merit	QW*	PDF-I	POM	I _h	OFF	RIR	Space-Group	a	b	c	Vol*	Z	Dx*
1> Unnamed mineral, syn [NR] - C4H6	30-	221	5.7	39	-06			13.36	17.54	11.87	2782	16	1.683
2> Moissanite-3\ITC\RG, syn - SiC	+	29-1129	7.0	10	0.00		F-43m (216)	4.36	4.36	4.36	21	4	3.216
3> Hoelite, syn - C6H4(CO)2C6H4	+	28-2002	7.3	35	0.06		P21/a (14)	15.81	3.97	7.88	482	2	1.435
4> Schoepite - UO3·2H2O	?	13-241	7.6	80	-30		Pcab (61)	14.73	16.79	14.33	3544	32	4.829
5> Wyartite-17A - Ca3U(OO2)6(CO3)2	?	12-636	8.3	62	-24		Pnmb (62)	11.25	16.83	7.10	1344	2	6.074
6> Becquerelite, syn - Ca(UO2)6O4	+	29-389	8.3	42	-18		Pna21 (33)	13.82	14.94	12.39	2558	4	5.116
7> Posidagite, syn - Ca4(SiO3)3(OH)2	+	29-377	8.7	25	-12		P21/n (11)	10.41	3.66	7.03	256	1	2.739
8> Diamond - C	+	6-675	9.0	12	0.06		P63m (227)	3.57	3.57	3.57	11	8	3.517
9> Wagscheiderite - Na2CO3·3NaHCO3	+	15-653	9.1	22	-18		P-1 (2)	10.04	15.56	3.47	509	2	2.336
10> Uranophane - Ca(UO2)2(SiO3OH)2·5H2O	+	39-1360	9.4	33	0.42		P21 (4)	15.92	7.01	6.67	739	2	3.849
11> Compregnacite, syn - K2(UO2)6O4	+	33-1049	9.5	31	0.00		Pnmm (58)	12.19	14.85	7.16	1296	2	5.146
12> Wyartite-21A - Ca(UO2)2(CO3)2(OH)·16H2O	+	12-635	9.8	75	0.30		P212121 (19)	11.25	20.80	7.10	1661	2	5.011
13> Wollastonite-1\ITC\RG - CaSiO3	+	42-550	9.9	24	0.06	0.9	P-1 (2)	7.31	7.91	7.06	396	6	2.925
14> Zellerite - Ca(UO2)2(CO3)2·5H2O	+	19-257	9.9	31	-18		Pn* (59)	11.22	19.25	4.93	1066	4	3.243
15> Monohydrocalcite - CaCO3·H2O	+	29-306	10.4	56	-18		P31 (144)	10.57	10.57	7.57	732	9	2.411
16> Metaschoepite, syn - UO3·2H2O	+	43-364	10.6	51	-42			14.67	16.70	13.98	3424		
17> Compregnacite - K2(UO2)6O4(OH)6	+	17-167	10.7	26	-36		Pnmm (58)	12.14	14.88	7.16	1293	2	5.157
18> Wollastonite-1\ITC\RG - CaSiO3	+	42-547	10.7	22	0.06	1.0	P-1 (2)	7.30	7.90	7.05	394	6	2.937
19> Karpaitite, syn - C6H2(C4H2)4C2H2	+	28-2008	10.8	25	-42		P21/n (14)	15.56	4.70	10.03	704	2	1.416
20> Kilchoanite, syn - Ca6(SiO4)(Si3)	+	29-370	10.9	29	0.18		Im2a (46)	11.43	22.02	5.08	639	4	2.996
21> Urancalcite - Ca(UO2)3CO3(OH)6	+	38-350	11.1	30	-36		Pbm* (62)	15.42	16.08	6.97	1728	4	4.098
22> Graphite-3\ITC\RG, syn [NR] - C	+	26-1079	11.1	20	0.06		R3 (146)	2.46	2.46	10.04	17	6	2.281
23> Unnamed mineral [NR] - Ca-O-Si-O	?	15-529	11.6	29	0.36								
24> Schoepite, syn - UO3·2H2O	+	29-1376	11.6	51	-36		Pcab (61)	14.74	16.66	14.36	3526	32	4.853
25> Killalaite - Ca3.2(HO.6Si2O7)(OH)	+	29-332	11.8	37	-36	0.8	P21/n (11)	6.81	15.46	6.81	710	4	2.937
26> Paraspurrite - Ca5(SiO4)2CO3	+	29-307	11.9	35	-30		P21/c (14)	27.78	6.71	10.47	1950	8	3.028
27> Wollastonite-2\ITC\RG - CaSiO3	+	27-88	12.0	24	0.12		P21/a (14)	15.43	7.32	7.07	794	12	2.914
28> Paraspurrite, syn - UO2·8Si·5H2O	+	23-1461	12.5	21	-18		Pcab (61)	15.04	18.98	14.23	4062	44	5.590
29> Karpaitite, syn - C6H2(C4H2)4C2H2	+	28-2007	12.6	68	-42		P21/n (14)	15.60	4.67	10.16	710	2	1.404
30> Hillebrandite - Ca2(SiO3)(OH)2	+	42-538	12.7	24	-30		A*a* (63)	11.84	16.32	3.64	352	6	2.693
31> Hydroxycarphylite - KCa4Si8O20	+	30-920	12.7	42	0.30	0.8	P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
32> Wollastonite-2\ITC\RG - CaSiO3	+	43-1460	12.9	24	0.12		P21/a (14)	15.43	7.32	7.07	795	12	2.910
33> Unnamed mineral [NR] - UO3·H2O	?	15-569	13.2	26	0.42								
34> Schoepite - UO3·2H2O	+	13-407	13.2	80	-42		Pcab (61)	14.73	16.79	14.33	3544	32	4.829
35> Grunantite - NaSi2O4(OH)·H2O	+	42-1331	13.4	35	0.00		F ()	16.00	18.24	7.18	524	16	2.259
36> Mountinite - (Ca,Na2,K)2Si4O10	+	25-676	13.4	22	-30		P21/* (11)	13.51	13.10	13.51	2320	8	2.360
37> Ianthinite - U6O7(OH)2O	+	12-272	13.5	39	0.24		C ()	11.52	30.30	7.15	1248	4	5.004
38> Metazellerite, syn - CaUO2(CO3)2	+	19-258	13.5	60	0.42		Pbm* (62)	9.72	18.23	4.97	879	4	3.657
39> Fukalite - Ca4Si2O6(CO3)(OH)2	+	29-308	13.6	42	-06		C2c* (63)	5.48	23.42	3.78	243	2	2.783
40> Tridymite-20\ITC\RG, syn - SiO2	+	14-260	13.6	53	-42		P ()	9.92	9.92	81.50	6946	160	2.298

* QW - ? for doubtful quality or non-ambient patterns; + for indexed patterns with Space-Group, Unit Cell & Density.

* vol - reduced-cell volume; dx - measured density if calculated is not available.

Current S/M parameters:

PDF Subfile to Search MINERALS
Chemistry Filter NF,CHM (Yes)
Search Focus on Major Phase(s)
Preferred Orientation in Sample No
Search only High Quality Phases No
Exclude Questionable Phases No

2-Theta Error Window42
Sensitivity to Matching Intensity 5
Sensitivity to Matching 2-Theta 5
Maximum Number of Hits to Save 40
Rubber-Ruler Search - Elasticity 0
Required 100% Line in Srded Range ... No

Apparently not enough H₂O was added for the reaction of the reagents to completely dissolve & form U phases only. Additional H₂O will be added to the solids and the experiment will continue (see next page for details).

4/24/95 JP

The unanophane synthesis experiment on p3+4 was again conducted but instead of using new reagents the solids formed in the first synthesis were placed in the vessel.

In addition, instead of 500g, approximately 300g of degassed H_2O was transferred into the vessel.

Wt of erlenmeyer flask with degassed H_2O = ~~1040.1~~ 1086.4 g
before transfer of H_2O to reaction vessel

Wt of erlenmeyer flask with degassed H_2O = 782.2 g
after transfer of H_2O to reaction vessel

Wt of degassed H_2O transferred to reaction vessel = 304.2 g

5/1/95 JP

0800 hrs.

The pressure vessel in the unanophane synthesis experiment was cooled and opened to investigate the solids produced.

The vessel contained a slushy yellow-white material. The slush was too dense to take a pH measurement.

About 5 to 10g of the material was taken out & placed in a glass beaker for mineralogical characterization.

The pressure vessel was resealed and the synthesis continued. About 100g of degassed H_2O was added to the vessel before bringing the temperature up to 150°C.

Wt of erlenmeyer flask with degassed H_2O = 989.6 g
before transfer of H_2O to reaction vessel

Wt of erlenmeyer flask with degassed H_2O = 816.8 g
after transfer of H_2O to reaction vessel

Wt of degassed H_2O transferred to reaction vessel = 172.8 g

5/2/95

JP

0900hrs

Yellow white material taken from pressure vessel (see page 11) was washed with H_2O and dried.

The solid was then analyzed by XRD to identify its mineralogy.

XRD procedure outlined on p 6 was used to prepare and analyze the sample.

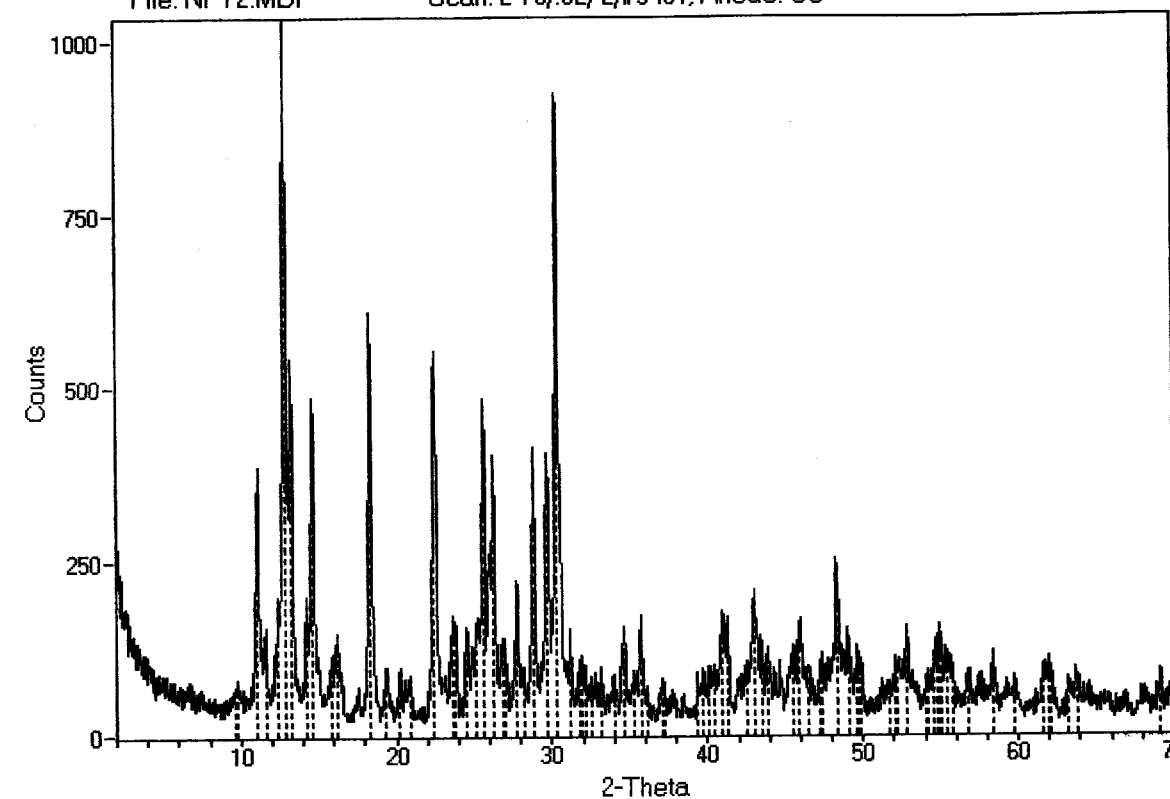
Results of the XRD analysis is shown on the following page.

Yellow-white material

ID: 5/2/95 - NFY2-XRD1

File: NFY2.MDI

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



XRD analysis indicates that the yellow-white material consists of unenphane, bottwoodite, and wechsite.

Southwest Research Institute
Tue May 02 1995 @3:55pm

Jade: Hit List from Search/Match
NPY2.MDI> 5/2/95 - NPY2-IRDI

40 Hits Sorted on Figure-of-Merit

Q#	PDF#	POW	I _h	OFF	RIR	Space-Group	a	b	c	Vol*	Z	Dx*
1>	Hillebrandite - Ca ₂ (SiO ₃)(OH) ₂	42-538	6.4	43	-18	A* ² (63)	11.84	16.32	3.64	352	6	2.693
2>	Uranophane - Ca(UO ₂) ₂ (SiO ₃ OH) ₂ ·5H ₂ O	39-1360	6.5	29	-12	P2 ₁ (4)	15.92	7.01	6.67	739	2	3.849
3>	Uranocalcarite - Ca(UO ₂) ₂ CO ₃ (OH) ₆	38-350	7.5	28	0.12	Pbm* (62)	15.42	16.08	6.97	1728	4	4.098
4>	Lonsdaleite, syn - C	19-268	7.8	12	0.06	P6 ₃ /mmc (194)	2.52	2.52	4.12	23	4	3.521
5>	Boltwoodite - K(H ₃ O)(UO ₂)(SiO ₄)	35-490	7.9	39	0.00	P2 ₁ (4)	7.07	7.06	6.64	319	2	4.372
6>	Grumantite - NaSi ₂ O ₄ (OH)·H ₂ O	42-1331	8.0	34	0.18	P (1)	16.00	18.24	7.18	524	16	2.259
7>	Diamond - C	6-675	9.0	8	0.00	Fd3m (227)	3.57	3.57	3.57	11	8	3.517
8>	Hydroxypaphyphylite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	29-994	9.0	36	0.12	P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
9>	Scawtite - Ca ₇ (Si ₆ O ₁₈)(CO ₃) ₂ ·2H ₂ O	31-261	10.2	30	0.12	I2/m (12)	10.12	15.18	6.62	500	2	2.766
10>	Rosenbuhite - Ca ₃ (Si ₃ O ₈)(OH) ₂	29-378	10.4	27	0.12	P-1 (2)	6.95	9.48	6.81	420	2	2.899
11>	Hydroxypaphyphylite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	30-920	10.7	18	-18	P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
12>	Dellite, syn - Ca ₆ (SiO ₄)(Si ₂ O ₇)	29-376	11.0	11	0.12	P-1 (2)	6.93	12.88	6.82	597	2	2.975
13>	Tobermorite-11a, syn - Ca ₅ (OH) ₂ Si ₂ O ₇ (CO ₃) ₂	19-1364	12.0	21	-06	B2212 (20)	11.27	22.74	7.35	942	4	2.578
14>	Tilleyite - Ca ₅ Si ₂ O ₇ (CO ₃) ₂	24-184	12.3	26	-06	P2 ₁ /n (14)	15.02	10.24	7.58	1132	4	2.867
15>	Foshagite, syn - Ca ₄ (SiO ₃) ₃ (OH) ₂	29-377	12.3	62	0.18	P2 ₁ /n (11)	10.41	3.66	7.03	256	1	2.739
16>	Uranophane-SGB - Ca(UO ₂) ₂ (SiO ₃ OH) ₂ ·5H ₂ O	8-301	12.7	26	-18	P2 ₁ /a (14)	14.01	15.55	6.64	1446	4	3.933
17>	Sodilubboltwoodite - (Na,K)(H ₃ O)UO ₂	29-1044	12.8	27	0.00	P2 ₁ 2121 (19)	7.02	27.40	6.65	1279	8	4.100
18>	Unnamed mineral [NR] - CO ₃ H ₂ O	15-569	13.1	26	-06							
19>	Jollite - (UO ₂)CO ₃ ·2H ₂ O	29-1378	13.3	26	0.18	P*** (47)	8.16	10.35	6.32	534	4	4.555
20>	Parasphurite - Ca ₅ (SiO ₄) ₂ CO ₃	29-307	13.3	20	0.06	P2 ₁ /c (14)	27.78	6.71	10.47	1950	8	3.028
21>	Nahcolite, syn - NaHCO ₃	15-700	13.3	62	0.00	P2 ₁ /n (14)	7.47	9.69	3.48	252	4	2.218
22>	Gaylussite - Na ₂ Ca(CO ₃) ₂ ·5H ₂ O	21-343	13.4	13	0.06	I* ² /a (15)	11.58	7.78	11.21	494	4	1.993
23>	Whewellite, syn - C ₂ CaO ₄ ·H ₂ O	20-231	13.7	21	-18	P2 ₁ /n (14)	9.98	7.29	6.29	438	4	2.217
24>	Weddellite, syn - C ₂ CaO ₄ ·2H ₂ O	17-541	13.8	14	0.18	I4/m (87)	12.35	12.35	7.36	562	8	1.941
25>	Coesite, syn - SiO ₂	14-654	13.9	30	0.00	P2 ₁ /n (14)	7.17	12.38	7.17	551	16	2.896
26>	Riversideite-9A - Ca ₅ Si ₆ O ₁₆ (OH) ₂	29-329	14.0	30	0.06	P (1)	5.57	18.79	3.64	381	1	2.871
27>	Konotite, syn - Ca ₆ Si ₆ O ₁₇ (OH) ₂	23-125	14.0	21	-06	P2/a (13)	17.02	7.35	7.00	877	2	2.709
28>	Aragonite - CaCO ₃	41-1475	14.1	26	0.00	Pnam (62)	5.74	7.97	4.96	227	4	2.927
29>	Metahalweeite - CaO ₁₂ UO ₃ 16SiO ₂ 1r	12-722	14.1	29	0.06							
30>	Rhodesite - (Ca,K,Na)Si ₁₆ O ₄₀ 111	22-1253	14.5	24	-12	P2 ₁ /n (14)	9.62	23.96	9.51	2186	2	2.360
31>	Blüthenschlitzite, syn - K ₂ Ca(CO ₃) ₂	25-626	15.0	9	-06	R-3m (166)	5.38	5.38	18.12	151	3	2.614
32>	Fluogastafite, syn - CH ₃ (OH)C ₆ H ₉ C	28-2014	15.2	18	0.06	Fdd2 (43)	18.50	22.60	11.00	1150	16	1.099
33>	Larnite, syn - Ca ₂ SiO ₄	33-302	15.3	6	0.00	P2 ₁ /n (14)	9.31	6.76	5.51	345	4	3.313
34>	Natrite - Na ₂ CO ₃	37-451	15.4	43	0.12		8.91	5.24	6.05	276	4	2.546
35>	Magadiite, syn - Na ₂ Si ₁₄ O ₂₉ 110H ₂	42-1350	15.9	34	-18	P (1)	15.71	7.28	7.30	830	1	2.168
36>	Weeksite - K ₂ (UO ₂) ₂ (Si ₆ O ₁₅) ₄ H ₂ O	12-462	16.0	10	-06	Pnna (52)	14.26	35.88	14.20	7265	16	4.018
37>	Killalaite - 2Ca ₃ Si ₂ O ₇ ·H ₂ O	26-1070	16.3	12	0.18	P2 ₁ /n (11)	6.81	15.46	6.81	710	2	2.782
38>	Vaterite - CaCO ₃	33-268	16.6	12	-18	P6 ₃ /mmc (194)	7.15	7.15	16.92	748	12	2.665
39>	Soddyite - (UO ₂) ₂ (SiO ₄) ₂ ·H ₂ O	35-491	16.6	10	0.12	Fddd (70)	11.21	18.71	8.32	436	8	5.087
40>	Killalaite - Ca ₃ 2(H ₂ O ₆ Si ₂ O ₇)(OH)	29-332	16.9	11	0.12	P2 ₁ /n (11)	6.81	15.46	6.81	710	4	2.937

* Q# - ? for Doubtful Quality or Non-ambient patterns; + for Indexed patterns with Space-Group, Unit Cell & Density.
* vol - reduced-cell volume; dx - measured density if calculated is not available.

Current S/N parameters:

PDF Subfile to Search MINERALS
Chemistry Filter NP.CHN (Yes)
Search Focus on Major Phase(s)
Preferred Orientation in Sample No
Search only High Quality Phases No
Exclude Questionable Phases No

2-Theta Error Window 18
Sensitivity to Matching Intensity 5
Sensitivity to Matching 2-Theta 5
Maximum Number of Hits to Save 40
Rubber-Ruler Search - Elasticity 0
Required 100% Line in Srded Range ... No

5/15/95 JP

Uranophane synthesis program

ON 5/8/95 a high limit temperature
default occurred in the 4841 temperature
controller

Apparently a leak occurred in the
vessel and all water was lost.

The controller was cut off and the
experiment was suspended until
5/15/95 when I returned
from vacation.

JP ON 5/15/95 the vessel was
opened and the solids inspected
visually. No water remained
confirming that a leak had
occurred. The solids consisted
of a yellow + white intermixed
solid.

1000 hrs
JP

The pressure vessel was resealed and the synthesis was restarted. About 9 g of degassed water adjusted to a pH of 1.00 was added to the vessel before bring the temperature up to 150°C.

1

Wt of flask with degassed H_2O = 1099.8 g
before transferring to reaction vessel

Wt of flask with degassed H_2O = 715.2 g
after transferring to reaction vessel

Wt of degassed H_2O transferred to vessel = 384.6 g

5/18/95

0800 hrs

JP

A second uranophane synthesis experiment was started using a much higher water to solid ratio.

This experiment will be referred to as "uranophane synthesis 2".

The procedure on p 3 + 4 will be followed with the following modifications:

a) 35 g Ca-Ac, 42 g U-Ac, and 28 g Na-metasilicate will be placed in the teflon lined vessel.

b) 1330 g of degassed H_2O will be transferred to the vessel.

This water/solid ratio should allow solids to completely react & form uranophane.

Flask A

Wt of erlenmeyer flask with degassed H_2O = 1185.6 g
before transfer of H_2O to reaction vessel

Wt of erlenmeyer flask with degassed H_2O = 768.6 g
after transfer of H_2O to reaction vessel

Wt of degassed H_2O transferred to vessel = 417 g

Flask B

Wt of erlenmeyer Flask with degassed H_2O = 1363.2 g
before transfer to reaction vessel

Wt of erlenmeyer Flask with degassed H_2O = 470.16 g
after transfer to reaction vessel

Wt of degassed H_2O Transferred to vessel = 893.04 g

Total degassed H_2O Transferred to vessel = 1310.04 g

5/22/95 JP 0800hr

The pressure vessel for the first uranophane synthesis experiment was cooled by shutting off the temperature controller.

5/25/95 JP 11:15 hr

Pressure vessel for first uranophane synthesis experiment was opened and pH was measured.

pH = 5.32

The liquid remaining in the vessel is clear. Solids consist of yellow and green material. Yellow material is probably U-mine. Green material is not expected. The thermocouples made of stainless steel are very corroded. The green material is probably a product of their corrosion.

An approximate 10g sample of the solids was collected, washed with DI water & dried. This material will be characterized by XRD using procedure on p. 4.

5/26/95 JP 0900 hrs.

Microphone synthesis 2 experiment.

A high temp limit was encountered on 5/24/95 and the pressure vessel temp controller shut off.

The vessel was allowed to cool and then was opened to inspect the liquid & solids formed.

The liquid remaining in the vessel was clear & the pH was measured.

pH = 4.74

Solid consisted of a yellow slushy material of undetermined crystallinity. An approximately 10g sample of the solid was placed in a glass beaker. This material was washed with DI water, dried, and powdered. Characterization of the solid will be performed by XRD using procedure in pb.

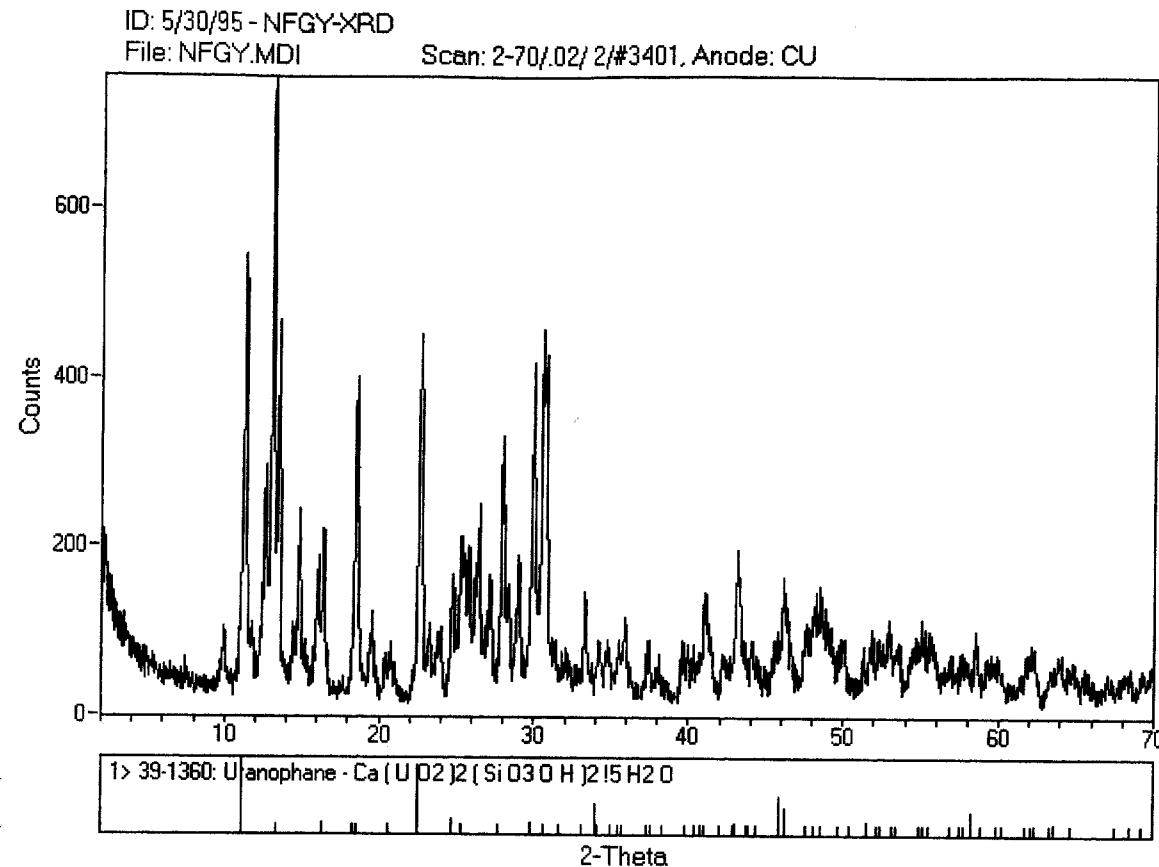
5/26/95 1100 hrs JP

The microphone synthesis experiment #2 was restarted.

The solids & remaining water in the teflon liner were placed back in the pressure vessel. The vessel was sealed. CO₂ was removed from the vessel by vacuum. The vessel was then brought to 150°C.

5/30/95 JP 1040 hrs.

Yellow/green material.



XRD analysis of yellow/green material
from the initial uvanophane
synthesis experiment. Uvanophane is
present in the material but other
phases are also present.

===== Southwest Research Institute =====
Jade: Hit List from Search/Match Tue May 30 1995 04:00pm

NFGY.MDI> 5/30/95 - NFGY-XRD

40 Hits Sorted on Figure-Of-Merit				Q#	PDF-#	FOM	I _h	OFF	RIR	Space-Group	a	b	c	Vol*	Z	Dx*
1>	Uvanophane - Ca(UO ₂) ₂ (SiO ₃ OH) ₂ ·5H ₂ O	39-1360	5.6	65	0.12					P21 (4)	15.92	7.01	6.67	739	2	3.849
2>	Metahaiweeite - CaO ₁₂ (UO ₂) ₃ (SiO ₂) ₁₂ ·xH ₂ O	12-722	6.2	35	-0.06											
3>	Weeksite - K ₂ (UO ₂) ₂ (SiO ₃) ₂ ·4H ₂ O	12-462	8.3	30	0.12					Pnna (52)	14.26	35.88	14.20	7265	16	4.018
4>	Boltwoodite - K(H ₃ O)(UO ₂)(SiO ₄)	35-490	8.7	80	0.06					P21 (4)	7.07	7.06	6.64	319	2	4.372
5>	Hillebrandite - Ca ₂ (SiO ₃)(OH) ₂	42-538	9.8	33	0.06					A*a* (63)	11.84	16.32	3.64	352	6	2.693
6>	Hydroxapophyllite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	29-994	12.2	41	0.18					P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
7>	Tilleyite - Ca ₅ Si ₂₀ (OH) ₂	24-184	12.6	24	0.12					P21/n (14)	15.02	10.24	7.58	1132	4	2.867
8>	Hoelite, syn - C ₆ H ₄ (CO) ₂ C ₆ H ₄	28-2002	12.7	20	0.00					P21/a (14)	15.81	3.97	7.88	482	2	1.435
9>	Graphite-2(1T) _h RG - C	41-1487	13.3	20	0.00				7.8	P6 ₃ /mmc (194)	2.47	2.47	6.72	36	4	2.245
10>	Nahcolite, syn - NaHCO ₃	15-700	13.4	45	0.12				0.3	P21/n (14)	7.47	9.69	3.48	252	4	2.218
11>	Whewellite, syn - C ₂ CaO ₄ ·H ₂ O	20-231	13.7	24	-12					P21/n (14)	9.98	7.29	6.29	438	4	2.217
12>	Wollastonite-2(1T) _h RG - CaSiO ₃	27-88	14.0	36	-12					P21/a (14)	15.43	7.32	7.07	794	12	2.914
13>	Matrofairchildite - Na ₂ Ca(CO ₃) ₂	25-804	14.3	24	0.00					P6 ₃ /mmc (194)	5.29	5.29	13.22	320	2	2.136
14>	Hydroxapophyllite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	30-920	14.5	63	0.00				0.8	P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
15>	Unnamed mineral [NR] - UO ₃ ·H ₂ O	15-569	14.8	57	0.18											
16>	Rosenbahnite - Ca ₃ (SiO ₃ OH) ₂	29-378	15.3	37	-12					P-1 (2)	6.95	9.48	6.81	420	2	2.899
17>	Studtite, syn - UO ₄ ·4H ₂ O	16-206	15.4	26	-18					C*/a (12)	11.85	6.78	4.24	170	2	3.647
18>	Natrite - Na ₂ CO ₃	37-451	16.0	42	-18						8.91	5.24	6.05	276	4	2.546
19>	Pectolite-1(1T) _h RG - NaCa ₂ HSi ₃ O ₉	33-1223	16.4	31	-12				2.8	P-1 (2)	7.03	8.00	7.03	384	2	2.872
20>	Rhodesite - (Ca,K,Na) ₈ Si ₁₆ O ₄₀ ·11H ₂ O	22-1253	16.8	35	0.06					P21/n (14)	9.62	23.96	9.51	2186	2	2.360
21>	Wollastonite-1(1T) _h RG - CaSiO ₃	42-547	17.6	42	-0.06				1.0	P-1 (2)	7.30	7.90	7.05	394	6	2.937
22>	Xonotlite, syn - Ca ₆ Si ₆ O ₁₇ (OH) ₂	23-125	17.8	18	0.00					P2/a (13)	17.02	7.35	7.00	877	2	2.709
23>	Wollastonite-2(1T) _h RG - CaSiO ₃	43-1460	17.9	36	-12					P21/a (14)	15.43	7.32	7.07	795	12	2.910
24>	Ianthinite - U ₆ O ₇ (OH) ₂	12-272	18.8	33	-18					C ()	11.52	30.30	7.15	1248	4	5.004
25>	Grumantite - Na ₄ Si ₂₀ (OH) ₂ ·H ₂ O	42-1331	19.0	23	-0.06					F ()	16.00	18.24	7.18	524	16	2.259
26>	Unnamed mineral [NR] - K-Ca-CO ₃	25-627	19.2	33	0.06											
27>	Quartz, syn - SiO ₂	33-1161	19.4	26	-18				3.6	P3221 (154)	4.91	4.91	5.41	113	3	2.649
28>	Tobermorite-11A, syn - Ca ₅ (OH) ₂ Si ₈ O ₂₀	19-1364	19.5	18	0.00					B2212 (20)	11.27	22.74	7.35	942	4	2.578
29>	Rameauite - K ₂ Ca(UO ₂) ₆ O ₈ ·9H ₂ O	25-631	19.5	14	0.06					C*/c (15)	13.97	14.26	13.88	1214	4	5.550
30>	Wollastonite-1(1T) _h RG - CaSiO ₃	42-550	20.3	36	-18				0.9	P-1 (2)	7.31	7.91	7.06	396	6	2.925
31>	Fairchildite, syn - K ₂ Ca(CO ₃) ₂	21-1287	20.9	36	0.00				1.5	P6 ₃ /mmc (194)	5.29	5.29	13.35	324	2	2.441
32>	Hydrohalite - NaCl·2H ₂ O	29-1197	21.1	15	-18					P21/a (14)	6.50	10.12	6.33	379	4	1.654
33>	Compreignacite, syn - K ₂ (UO ₂) ₆ O ₈	33-1049	21.2	9	-0.06					Pnma (58)	12.19	14.85	7.16	1296	2	5.146
34>	Sodiumboltwoodite - (Na,K)(H ₃ O)U	29-1044	21.8	29	0.06					P212121 (19)	7.02	27.40	6.65	1279	8	4.100
35>	Liebigite - Ca ₂ UO ₂ (CO ₃) ₃ ·10H ₂ O	11-296	21.9	60	0.18					Bba2 (41)	16.70	17.51	13.74	2010	8	2.348
36>	Tokkoite - K ₂ Ca ₄ Si ₇ O ₁₇ (OH) ₄	40-517	22.1	14	0.06					P-1 (2)	10.37	25.39	7.27	1879	4	2.741
37>	Haiweeite - Ca(UO ₂) ₂ Si ₆ O ₁₅ ·5H ₂ O	12-721	23.3	16	-0.06					A ()	17.65	18.58	14.20	2329	8	3.077
38>	Melanophlogite - C ₂ H ₁₇ O ₅ Si ₄ O ₉	25-7	23.4	26	0.06					P42/nbc (133)	26.79	26.79	13.40	9614	4	1.993
39>	Uvanophane-SCB - Ca(UO ₂) ₂ (SiO ₃ OH) ₂	8-301	23.8	63	0.00					P21/a (14)	14.01	15.55	6.64	1446	4	3.933
40>	Sodydyte, syn - (UO ₂) ₂ (SiO ₄) ₂ ·2H ₂ O	35-733	24.0	13	0.06					Pddd (70)	11.27	18.65	8.30	436	8	5.089

* Q# - ? for Doubtful Quality or Non-ambient patterns; + for Indexed patterns with Space-Group, Unit Cell & Density.
* vol - reduced-cell volume; dx - measured density if calculated is not available.

Current S/M parameters:

PDF Subfile to Search MINERALS
Chemistry Filter NF.CHM (Yes)
Search Focus on Major Phase(s)
Preferred Orientation in Sample No
Search only High Quality Phases No
Exclude Questionable Phases No

2-Theta Error Window18
Sensitivity to Matching Intensity 5
Sensitivity to Matching 2-Theta 5
Maximum Number of Hits to Save 40
Rubber-Ruler Search - %Elasticity 0
Required 100% Line in Sched Range ... No

5/30/95 The first uvanophane synthesis experiment
will be terminated at this point
since pure uvanophane has not
been synthesized.

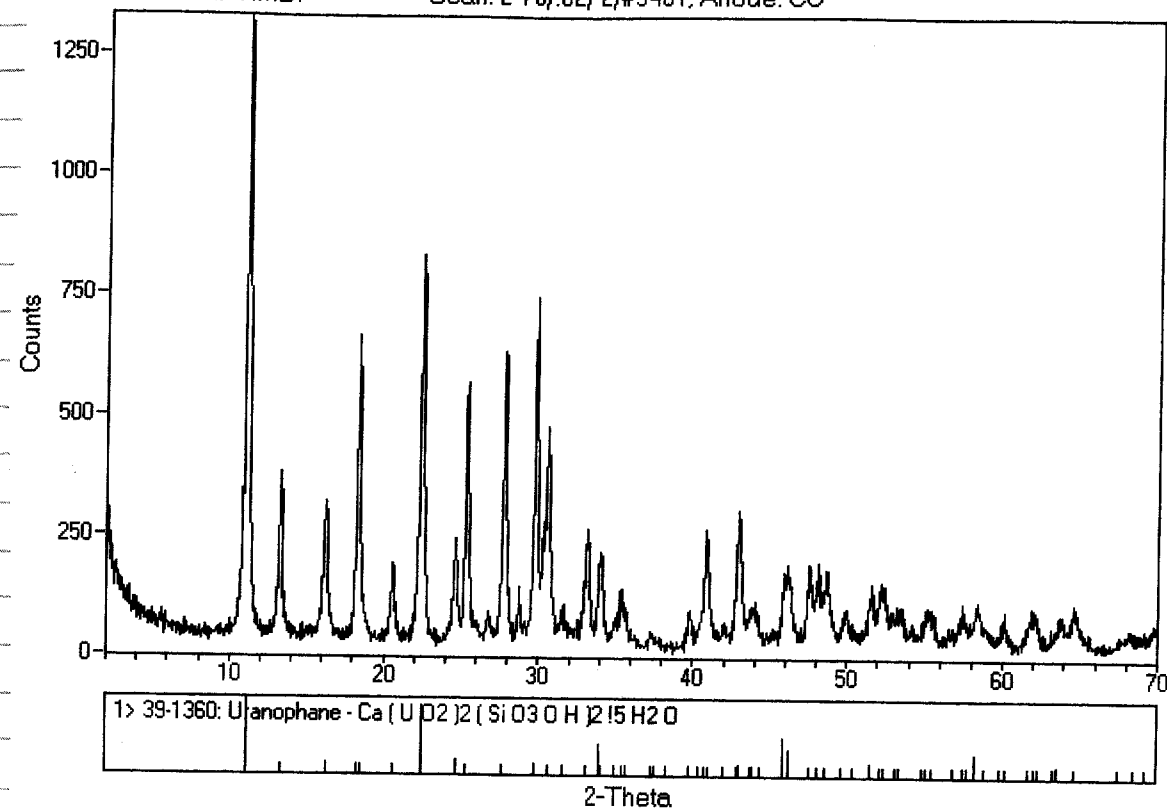
5/30/95

Yellow

ID: 5/30/95 - 2NFI-XRD

File: 2NFI.MDI

Scan: 2-70.02/2/#3401, Anode: CU



XRD analysis of yellow solid from
the 2nd uranophane synthesis
attempt

The pattern indicates that the solid
is predominantly uranophane.

Jade: Hit List from Search/Match

Southwest Research Institute

Tue May 30 1995 03:55pm

2NFI.MDI> 5/30/95 - 2NFI-XRD

40 Hits Sorted on Figure-of-Merit				QM*	PDF-#	FOM	I%	OFF	RIR	Space-Group	a	b	c	Vol*	Z	Dx*
1>	Uranophane - Ca(UO ₂) ₂ (SiO ₃ OH) ₂ ·5H ₂ O	+	39-1360	2.3	49	0.00				P21 (4)	15.92	7.01	6.67	739	2	3.849
2>	Hillebrandite - Ca ₂ (SiO ₃)(OH) ₂	+	42-538	19.5	32	0.12				A2/a* (63)	11.84	16.32	3.64	352	6	2.693
3>	Hydroxypapophyllite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	+	29-994	20.7	53	0.06				P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
4>	Tilleyite - Ca ₅ Si ₂₀ O ₇₂ (OH) ₂	+	24-184	21.7	21	-0.06				P21/n (14)	15.02	10.24	7.58	1132	4	2.867
5>	Uranocalcarite - Ca(UO ₂) ₂ (CO ₃)(OH) ₆	+	38-350	28.4	38	0.00				Pbn* (62)	15.42	16.08	6.97	1728	4	4.098
6>	Scawtite - Ca ₇ (Si ₆ O ₁₈)(CO ₃) ₂ ·2H ₂ O	+	31-261	28.9	46	0.18				I2/m (12)	10.12	15.18	6.62	500	2	2.766
7>	Hydroxypapophyllite - KCa ₄ Si ₈ O ₂₀ (OH) ₂	+	30-920	33.3	73	-1.8			0.8	P4/mnc (128)	8.98	8.98	15.83	1276	2	2.356
8>	Wollastonite-2ITM/RG - CaSiO ₃	+	27-88	34.2	47	-1.8				P21/a (14)	15.43	7.32	7.07	794	12	2.914
9>	Moissanite-3ITC/RG, syn - SiC	+	29-1129	34.3	4	0.18				F-43m (216)	4.36	4.36	4.36	21	4	3.216
10>	Fairchildite, syn - K ₂ Ca(CO ₃) ₂	+	21-1287	37.1	18	-0.06			1.5	P63/mmc (194)	5.29	5.29	13.35	324	2	2.441
11>	Nahcolite, syn - NaHCO ₃	+	15-700	38.2	20	0.06			0.3	P21/n (14)	7.47	9.69	3.48	252	4	2.218
12>	Pectolite-1ITM/RG - NaCa ₂ HSi ₃ O ₉	+	33-1223	39.4	28	-1.8			2.8	P-1 (2)	7.03	8.00	7.03	384	2	2.872
13>	Spurrite - Ca ₅ (SiO ₄) ₂ CO ₃	+	13-496	39.8	16	0.00				P21/c (14)	14.15	6.70	10.49	976	4	3.026
14>	Riversideite-9A - Ca ₅ Si ₆ O ₁₆ (OH) ₂	+	29-329	40.8	51	0.18				P ()	5.57	18.79	3.64	381	1	2.871
15>	Grunanite - NaSi ₂ O ₄ (OH) ₂	+	42-1331	40.8	31	0.00				F ()	16.00	18.24	7.18	524	16	2.259
16>	Unnamed mineral [NR] - CO ₃ ·H ₂ O	?	15-569	40.9	84	-1.2										
17>	Gaylussite - Na ₂ Ca(CO ₃) ₂ ·5H ₂ O	+	21-343	41.8	28	0.06				I/a (15)	11.58	7.78	11.21	494	4	1.993
18>	Natrosilite, syn - Na ₂ Si ₂ O ₅	+	23-529	42.1	48	0.00			0.4	P21/a (14)	12.33	4.85	8.13	471	4	2.568
19>	Natrofairchildite - Na ₂ Ca(CO ₃) ₂	?	25-804	42.7	23	-1.8				P63/mmc (194)	5.29	5.29	13.32	320	2	2.136
20>	Sodiumboltwoodite - (Na,K)(H ₃ O)U	+	29-1044	43.4	8	-0.06				P212121 (19)	7.02	27.40	6.65	1279	8	4.100
21>	Hoelite, syn - C ₆ H ₄ (CO) ₂ C ₆ H ₄	+	28-2002	44.7	35	0.00				P21/a (14)	15.81	3.97	7.88	482	2	1.435
22>	Uranophane-SGB - Ca(UO ₂) ₂ (SiO ₃ OH) ₂ ·5H ₂ O	+	8-301	44.9	31	-0.06				P21/a (14)	14.01	15.55	6.64	1446	4	3.933
23>	Wollastonite-2ITM/RG - CaSiO ₃	+	43-1460	45.5	27	-1.2				P21/a (14)	15.43	7.32	7.07	795	12	2.910
24>	Unnamed mineral [NR] - K-Ca-CO ₃	?	25-627	47.8	23	0.00										
25>	Killalaite - 2Ca ₃ Si ₂ O ₇ ·H ₂ O	+	26-1070	49.5	21	0.18				P21/n (11)	6.81	15.46	6.81	710	2	2.782
26>	Rustumite - Ca ₁₀ (Si ₂ O ₇) ₂ (SiO ₄)Cl	+	29-314	49.8	21	0.18			0.8	A2/a (15)	15.50	18.55	7.62	1062	4	2.921
27>	Moissanite-15ITM/RG, syn - SiC	+	39-1196	50.6	5	0.00				R3m (160)	3.07	3.07	37.70	103	15	3.239
28>	Wollastonite-1ITM/RG - CaSiO ₃	+	42-547	51.4	33	-1.8			1.0	P-1 (2)	7.30	7.90	7.05	394	6	2.937
29>	Boltwoodite - K(H ₃ O)(UO ₂)(SiO ₄)	+	35-490	60.1	13	0.18				P21 (4)	7.07	7.06	6.64	319	2	4.372
30>	Rustumite - Ca ₁₀ (Si ₂ O ₇) ₂ SiO ₄ Cl ₂	+	18-305	61.9	17	0.18				A2/a (15)	15.50	18.55	7.62	1062	4	2.921
31>	Ianthinite - U ₆ O ₇ (OH) ₂	+	12-272	63.2	41	0.18				C ()	11.52	30.30	7.15	1248	4	5.004
32>	Hydrohalite - NaCl·2H ₂ O	?	29-1197	65.7	33	-1.2				P21/a (14)	6.50	10.12	6.33	379	4	1.654
33>	Jollotite - (UO ₂)CO ₃ ·2H ₂ O	+	29-1378	66.6	48	0.06				P*** (47)	8.16	10.35	6.32	534	4	4.555
34>	Wollastonite-1ITM/RG - CaSiO ₃	+	29-372	68.1	40	-1.8				P1 (1)	7.37	7.89	7.04	397	6	2.913
35>	Flagstaffite, syn - CH ₃ (OH)C ₆ H ₉ C	+	28-2014	68.9	46	0.18				Fdd2 (43)	18.50	22.60	11.00	1150	16	1.099
36>	Trona - Na ₃ H(CO ₃) ₂ ·2H ₂ O	+	29-1447	68.9	13	0.18			1.0	I2/a (15)	20.11	3.49	10.33	353	4	2.124
37>	Wollastonite-1ITM/RG - CaSiO ₃	+	42-550	71.6	27	-1.8			0.9	P-1 (2)	7.31	7.91	7.06	396	6	2.925
38>	Rankinite - Ca ₃ Si ₂ O ₇	+	22-539	74.0	5	0.06				P21/n (14)	9.54	8.91	7.85	643	4	2.979
39>	Compreignacite - K ₂ (UO ₂) ₆ O ₄ (OH) ₆	+	17-167	76.3	20	0.00				Pnnn (58)	12.14	14.88	7.16	1293	2	5.157
40>	Rutherfordine - UO ₂ CO ₃	+	11-263	79.1	15	0.00				Pnnn (59)	4.85	9.24	4.33	194	2	5.657

* QM - ? for Doubtful Quality or Non-ambient patterns; + for Indexed patterns with Space-Group, Unit Cell & Density.
* vol - reduced-cell volume; dx - measured density if calculated is not available.

Current S/M parameters:

PDF Subfile to Search MINERALS
Chemistry Filter NF, CHM (Yes)
Search Focus on Major Phase(s)
Preferred Orientation in Sample No
Search only High Quality Phases No
Exclude Questionable Phases No

2-Theta Error Window 18
Sensitivity to Matching Intensity 5
Sensitivity to Matching 2-Theta 5
Maximum Number of Hits to Save 40
Rubber-Ruler Search - Elasticity 0
Required 100% Line in Srded Range ... No

6/1/95 gp
0800hs

The pressure vessel for the 2nd
uranophane synthesis experiment was
cooled and opened.

Inspection of contents revealed
presence of a fine crystalline
yellow solid suspended in the
solution.

The pH of the solution was
measured.

pH=4.62

About 75 ml of the solution with
the fine yellow suspension was
removed from the vessel and
placed in a glass beaker.
The suspension will be allowed
to settle.

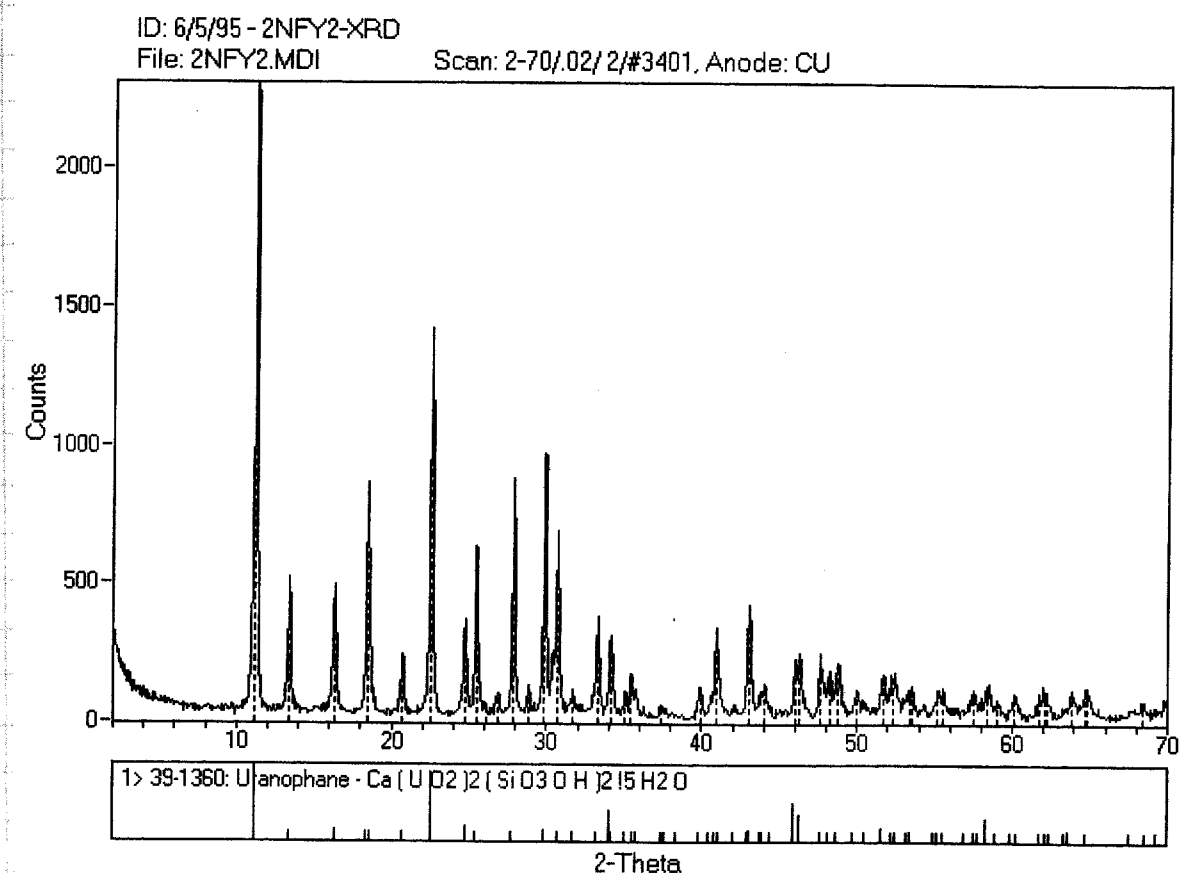
6/2/95 gp
0900hs

The yellow solid was washed
with H_2O and dried.

6/5/95 gp
0800hs

The yellow solid was powdered and
characterization of the solid was
performed by XRD using procedure
on p.6.

6/6/95 gp



XRD patterns for solids produced in
uranophane synthesis experiment 2 are
shown above. Uranophane appears to be
the only phase present.

1/7/95 GP
1000 hrs

Monophane (yellow solid) produced in the monophane synthesis experiment 2 was filtered (Whatman #5 paper), washed with DI water, dried, and weighed.

Weight of monophane synthesized = 33.42 g

The monophane was placed in a glass container and labeled URAN*SYN

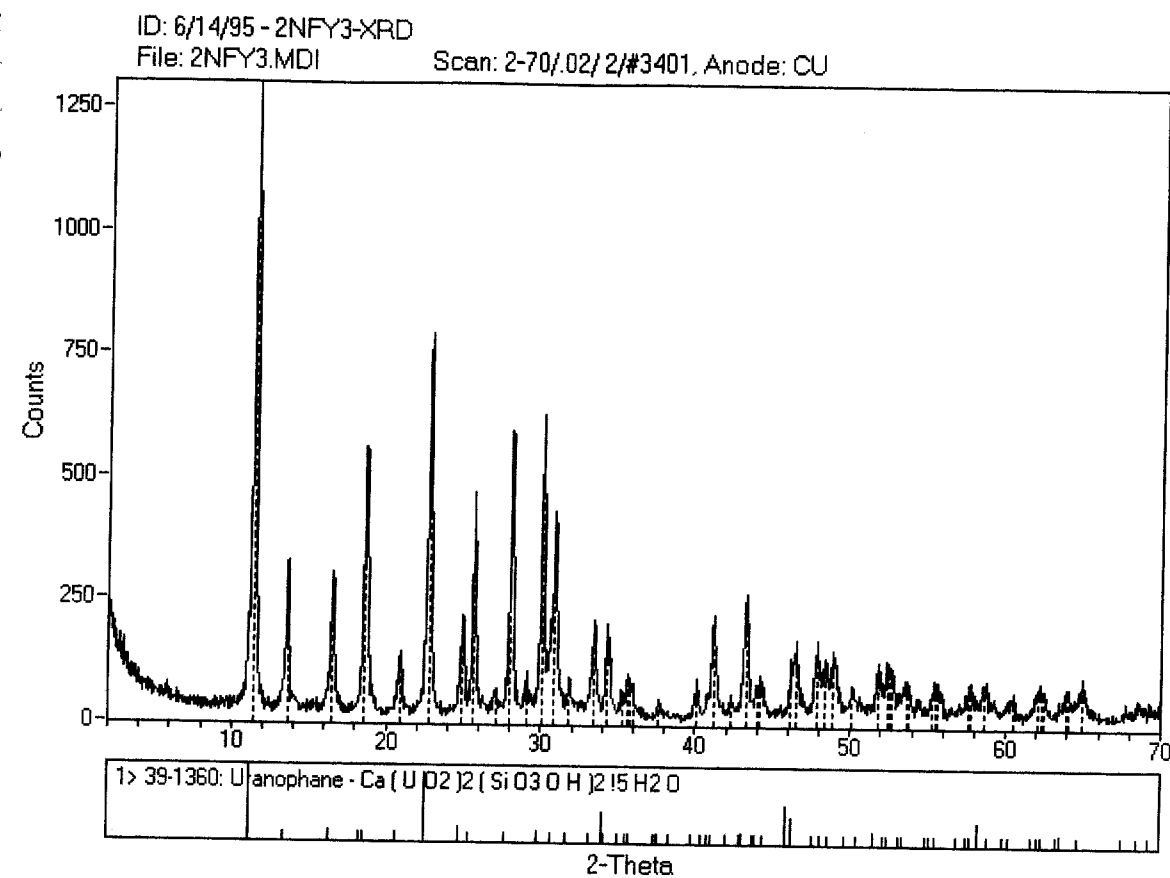
1/12/95 GP
1000 hrs

A portion of URAN*SYN was powdered and an XRD analysis was performed using procedure on p.6.

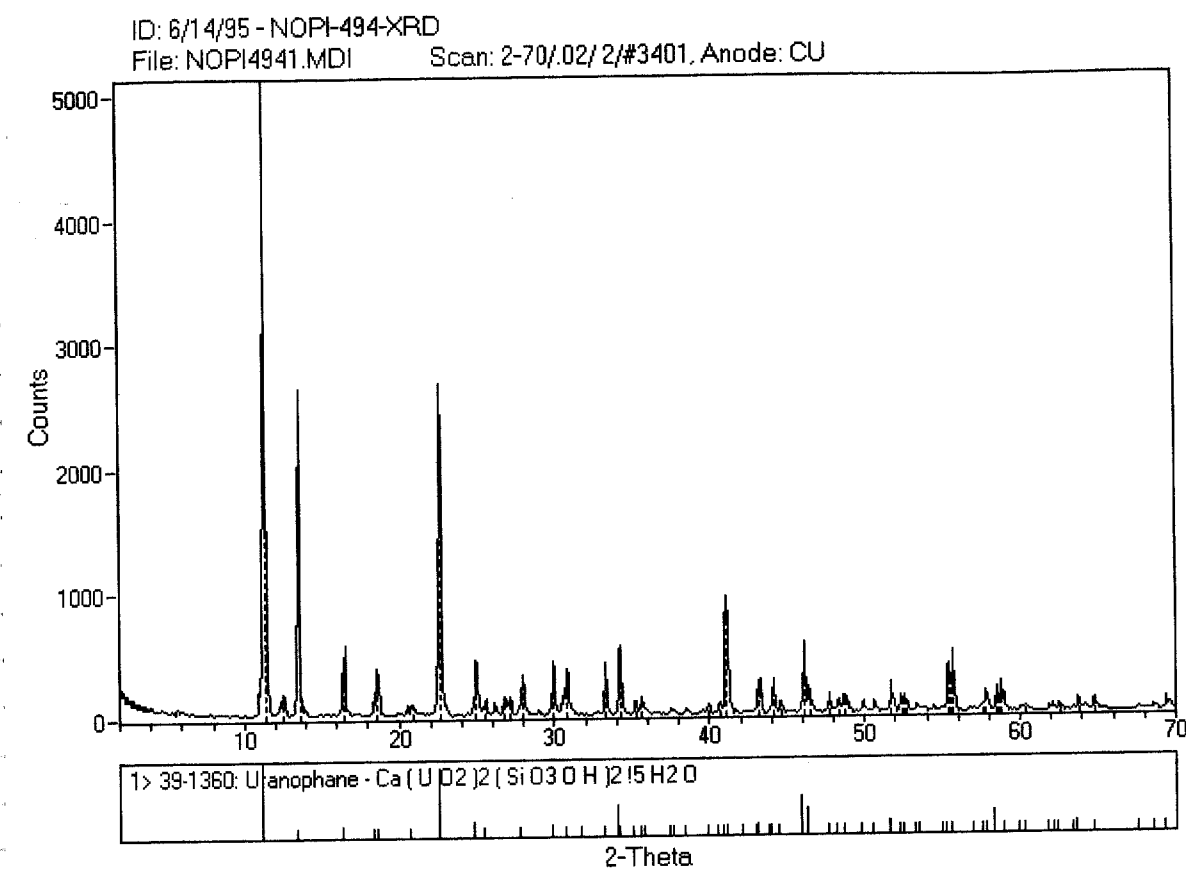
Naturally occurring monophane collected from the Wopai I U deposit was also analysed by XRD.

Acicular crystals of monophane from sample NOPP-494 were separated by hand-picking from a bulk tuff sample. The monophane was powdered and XRD analysis was performed using procedure on p.6.

6/21/95 JP



XRD pattern of URAW = SYN



XRD pattern of NOPI-494. Acicular uranophane picked from bulk sample collected at Nopal I deposit.

6/21/95 gp

SEM analysis of synthesized monophase and naturally occurring monophase from Nopal I.

Obj - analysis of monophase by scanning electron microscope to determine morphology of monophase.

Method - Scanning electron microscope

Equipment + Materials
AMRAY SEM in Div 15 gp 6/26/95
Aluminum stubs
Carbon tape.

Procedure.

- 1) A portion of URAN*SYN was placed on carbon tape attached to an aluminum stub labeled '1'.

Acicular crystals of monophase from Nopal I were placed on carbon tape attached to an aluminum stub labeled '2'.

Fracture lining monophase from Nopal I was placed on carbon tape attached to an aluminum stub labeled '3'.

6/23/95 gp 2) The 3 monophase samples were delivered to Div 06 for ^{iridium} gold coating gp 6/26/95

6/26/95 gp 3) Analyses were done in Div 15 by Ken Cook. Photos showing the morphology and crystal size of the synthesized monophase & the monophase from Nopal I were taken.

These photos are kept in a 3 ring binder entitled "Near Field Project Photos".

6/27/95

Two additional uranophane synthesis experiments were initiated to prepare additional uranophane for later experiments.

These two experiments will be referred to as "Uranophane synthesis 3" and "Uranophane synthesis 4".

The procedure on the following page will be used in these two experiments.

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 1

DATE WRITTEN: March 7, 1995
DATE REVISED: June 26, 1995

OBJECTIVE: to synthesize uranophane $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 2.5\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 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 944231)
Sodium metasilicate nonahydrate $[\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}]$; MW=284.20 (lot 942853A)
Calcium acetate monohydrate $[\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$; MW=176.19
(lot ~~31961711~~ 944231)
9 M HCl - diluted from lot 945500

JP
9/7/95
31961711 194

JP
9/7/95

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 H₂O used, they state that good crystallinity is obtained if pH is less than 5.

6/27/95
JP

- Place 35 g Ca-acetate, 42 g U-acetate, and 28 g Na-metasilicate in a 2000 ml teflon liner and place liner in reaction vessel.
- Cap and seal vessel and evacuate using a vacuum pump to remove CO₂(g).
- Transfer approximately 2000 ml of ultrapure H₂O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the H₂O at a pH of 1.0 by boiling on a hotplate. After degassing cap erlenmeyer flask with a rubber stopper so that air cannot enter the flask.
- After cooling record weight of the erlenmeyer flask with the degassed H₂O.
- Transfer approximately 1300 g of the degassed H₂O into the vessel by pulling in by vacuum. After the transfer, record weight of the erlenmeyer flask with remaining degassed H₂O to determine actual weight of H₂O introduced.
- Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller and allow the reaction to proceed for one week or longer.

6/27/95 JP

'Monophase synthesis 3'

Wt erlenmeyer flask with degassed H_2O = 2238.1 g
before transfer to vessel.

Wt erlenmeyer flask with degassed H_2O = 886.6 g
after transfer to vessel

Wt degassed H_2O transferred to vessel = 1351.5 g

'Monophase synthesis 4'

Wt erlenmeyer flask with degassed H_2O = 2256.0 g
before transfer to vessel

Wt erlenmeyer flask with degassed H_2O = 891.1 g
after transfer to vessel

Wt degassed H_2O transferred to vessel = 1364.9 g

7/5/95 JP

1630 hrs

The pressure vessel for 'Monophase Synthesis 4'
was cooled and opened

A fine yellow solid + the solution
were in the teflon container

The pH of the solution was measured

pH = 4.62

Yellow solid was filtered (Whatman #5
paper), washed with DI water,
transferred to a glass beaker, washed
with boiling DI water to remove
any remaining sticky solid, dried in
oven at 60°C, and weighed

Wt. of monophase = 31.7 g

The solid was placed in a glass
container + labeled URAW*SYN*4

7/6/95 JF
0800h

The pressure vessel for Uranophane Synthesis 3 was cooled + opened.

Contents of container included a fine yellow suspended solid + solution.

The pH of the solution was:

pH = 4.59

Yellow solid was filtered (Whatman #5), washed with DI water, transferred to a glass beaker, washed with boiling DI water, dried in oven at 60°C and weighed.

Wt of monophane = 33.45g

The solid was placed in a glass container and labelled URAN*SYN #3.

7/7/95

Uranophane Dissolution Test

Objective: dissolve uranophane so chemical analysis can be performed.

Materials: URAN*SYN
Teflon beaker
conc ~~0.1m~~ HClO₄ -
7/7/95 JF
conc HF

Procedure

JF 1000h 1) Place 50 mg URAN*SYN in a 250ml teflon beaker.

2) Add 100ml H₂O; 0.85 ml HClO₄; 1ml HF to beaker and stir occasionally with teflon rod.

JF 1330h Results

The fluid mixture was successful in dissolving the uranophane. No silica precipitation was observed.

The solution was transferred to a 250 ml teflon bottle labeled "URAN*SYN Dissolved in 0.1m HClO₄"

7/10/95 JP

Portions of URAN*SYN*3 and URAN*SYN*4 were powdered & an XRD analysis was performed on each using procedure on pg.

* Inspection of URAN*SYN*4 revealed the presence of dark particles in the yellow solid. These particles may be product of corrosion of the thermocouple during the synthesis. It was noticed that the thermocouple in "uranophane synthesis 4" was highly corroded at the end of the experiment. This solid should not be used in upcoming experiments.

7/11/95

SEM analysis of synthesized uranophane

Obj - determine morphology & gross chemistry of synthesized uranophane

Method - scanning electron microscope / energy dispersive X-ray microanalysis

Equipment -

Amray SEM in Div 06
Aluminum stubs
Carbon tape

Procedure -

- 1) Portions of URAN*SYN*3 and URAN*SYN*4 were attached to aluminum stubs using carbon tape
- 2) Samples were delivered to Jim Spencer in Div 06 for iridium coating.
- 3) Analyses were performed by Jim Spencer.

7/14/95 JP

Photos showing morphology & EDS showing chemistry of samples were taken.

These photos are kept in a 3-ring binder entitled "Nau Field Project Photos".

7/13/55

Chemical analysis of synthetic
uranophane by ICP

Obj - determine chemistry of synthetic
uranophane & other minerals (ie, standards
& clays used in sorption project).

Method - ICP analysis in Div 01

Procedure -

1) Portions of the following samples
+ standards were placed in
glass vials and labeled as
follows:

label	Sample	
20A	URAN*SYN	1.98g
20B	RGM-1	2.04g
20C	URAN*SYN*3	1.87g
20D	URAN*SYN	1.81g
20E	STM-1	2.00g
20F	278	1.97g
20G	URAN*SYN*3	1.63g
20H	Na-SAZ-1	< 1g
20I	Cheto	2.15g
20J	SAZ-Na	1.45g
20K	Cheto	2.01g
20L	SAZ-Na	1.56g
20M	SCo-1	~2g
20N	SCo-1	~2g

2) Samples were delivered to Div 01
for ICP and mass analysis

7/17/95 JP The synthesis of soddyite will be attempted using the procedure outlined below.

SODDYITE SYNTHESIS

WRITTEN BY: J.D. Prikrýl
REVISION NO.: 0

DATE WRITTEN: July 3, 1995
DATE REVISED:

OBJECTIVE: to synthesize soddyite $[(\text{UO}_2)_2\text{SiO}_4 \cdot 2\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/ $^{\circ}\text{C}$ meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 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 944234)
Sodium metasilicate nonahydrate $[\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}]$; MW=284.20 (lot 942853A)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume $4\text{H}_2\text{O}$ - MW = 132 (lot 951303)
9 M HCl - diluted from lot 945500

PROCEDURE:

The procedure for uranophane synthesis is based on Nguyen et al., 1992. Reagents will be mixed in the stoichiometric ratio U:Si = 1:2.

- Two batches will be synthesized but reagent for Si will be different to see if Na can be removed from system.
(1) Place 42 g U-acetate, and 56 g Na-metasilicate in a 2000 ml teflon liner and place liner in reaction vessel.
(2) Place 42 g U-acetate, and 26 g silicic acid in a 2000 ml teflon liner and place liner in reaction vessel.
- Cap and seal vessel and evacuate using a vacuum pump to remove $\text{CO}_2(\text{g})$.
- Transfer approximately 2000 ml of ultrapure H_2O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the H_2O at a pH of 1.0 by boiling on a hotplate. After degassing cap erlenmeyer flask with a rubber stopper so that air cannot enter the flask.

JP 7/17/95
319617/1 194

- After cooling record weight of the erlenmeyer flask with the degassed H_2O .
- Transfer approximately 1300 g of the degassed H_2O into the vessel by pulling in by vacuum. After the transfer, record weight of the erlenmeyer flask with remaining degassed H_2O to determine actual weight of H_2O introduced.
- Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller and allow the reaction to proceed for one week or longer.

Batch 1 - using Na-metasilicate

'Soddyite synthesis 1'

Wt erlenmeyer flask with degassed H_2O = 2289.7g
before transfer to vessel

Wt erlenmeyer flask with degassed H_2O = 896.0g
after transfer to vessel

Wt degassed H_2O transferred to vessel = 1393.7g

Batch 2 - using silicic acid

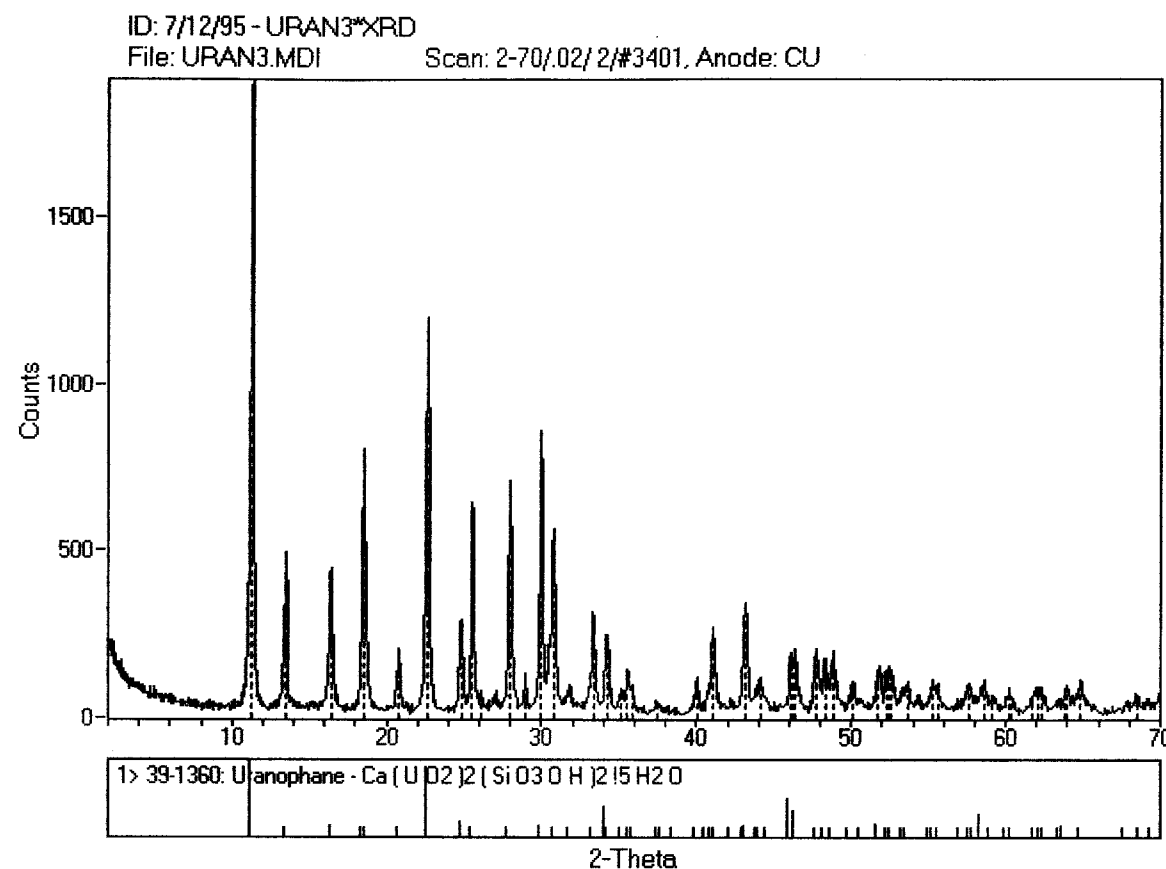
'Soddyite Synthesis 2'

Wt erlenmeyer flask with degassed H_2O = 2547.8g
before transfer to vessel

Wt erlenmeyer flask with degassed H_2O = 1157.6g
after transfer to vessel

Wt degassed H_2O transferred to vessel = 1390.2g

7/7/95 XRD Results - URAN*SYN*3



7/19/95 JP

AA analysis of Ca, Na, and Si
in uranophane dissolved in
0.1 M HClO_4 .

Obj - determine content of Ca, Na,
and Si in "URAN*SYN Dissolved
in 0.1 M HClO_4 " (see p 39).

Method - atomic absorption analysis (AA)

Equipment + Materials

Perkin Elmer 3100 AA spectrometer

Ca, Na, & Si AA lamps

1000 ppm Ca standard (9409823-24)

1000 ppm Na standard (lot 936266-29)

1000 ppm Si standard

0.1 M HClO_4 - dissolve JP 7/19/95 (lot 901940)

HCl (lot 915032A)

KCl (lot 885967)

Volumetric Flask

Plasticware + glassware as needed.

Procedure:

Ca analysis

1) Make up matrix solution of
0.1 M HClO_4 by adding 8.5 ml HClO_4
to about 500 ml H_2O in a 1L vol.
flask. Add 5g KCl and 5g HCl to
reduce interference. Make up to mark
with H_2O .

- 2) Make a 10 ppm Ca standard by adding 10 ml of Ca standard (1000 ppm) to 500 ml H_2O in a 1L Vol Flask. Make up to mark with H_2O .
- 3) Make Ca calibration standards for the AA using the 10 ppm Ca standard and the 0.1 M $HClO_4$ matrix. Following cal. standards were made in 10 ml vol flasks.

Ca ppm	10 ppm Ca standard (ml)	0.1 M $HClO_4$ matrix (ml)
0.5	0.5	9.5
2.0	2.0	8.0
5.0	5.0	5.0

- 4) Samples of "URAN*SYN dissolved in 0.1 M $HClO_4$ " were prepared by taking 1 ml and diluting to 10 ml with the 0.1 M $HClO_4$ matrix. 2 samples were prepared. Cal + Ca2

- 5) AA spectrometer was calibrated using calibration standards and then samples were measured. Results are shown below

$$Cal = 1.86 \text{ ppm} \times \text{dilution factor (10)} = 18.6 \text{ ppm}$$

$$Ca2 = 1.87 \text{ ppm} \times \text{dilution factor (10)} = 18.6 \text{ ppm}$$

Na analysis

- 1) Make up matrix solution of 0.1 M $HClO_4$ by adding 8.5 ml $HClO_4$ to ~500 ml H_2O in a 1L vol. flask. Add 1 g of KCl to reduce interference from other elements. Make up to mark with H_2O .

- 2) Make a 10 ppm Na standard by adding 10 ml of Na standard (1000 ppm) to 500 ml H_2O in a 1L vol flask. Make up to mark with H_2O .

- 3) Make Na calibration standards for AA using the 10 ppm Na standard & the 0.1 M $HClO_4$ matrix. Following cal. standards were made in 50 ml vol flasks.

Na ppm	10 ppm Na standard (ml)	0.1 M $HClO_4$ matrix (ml)
0.05	250 μ l	49.75 ml
0.2	1 ml	49 ml
1.0	5 ml	45 ml

- 4) Samples of "URAN*SYN dissolved in 0.1 M $HClO_4$ " were prepared by taking .5 ml & diluting to 10 ml with 0.1 M $HClO_4$ matrix. 2 samples were prepared Na1 + Na2

- 5) AA spectrometer was calibrated using cal standards & then samples were measured. Results are shown below.

$$Na1 = .516 \text{ ppm} \times \text{dilution factor (20)} = 10.32 \text{ ppm}$$

$$Na2 = .547 \text{ ppm} \times \text{dilution factor (20)} = 10.94 \text{ ppm}$$

Si Analysis

1) Make up matrix solution of 0.1 M HClO_4 by adding 8.5 ml HClO_4 to 2500 ml H_2O in a 12 vol. flask. Make up to mark with H_2O .

2) Make Si calibration standards using a 1000 ppm Si AA standard solution and the 0.1 M HClO_4 solution matrix. Following cal. standards were made in 50 ml volumetric flasks.

Si ppm	1000 ppm Si Std. (ml)	0.1 M HClO_4 matrix (ml)
10	.5	45.5
20	1.0	49.0
50	2.5	47.5

4) Samples of "URAN*SYN" dissolved in 0.1 M HClO_4 were prepared by taking 5 ml + diluting to 10 ml with "0.1 M HClO_4 matrix". 2 samples were prepared Si 1 and Si 2.

5) AA Spectrometer was calibrated using cal standards & then samples were measured. Results are shown below.

$$\begin{aligned}\text{Si 1} &= 33.67 \times \text{dilution factor (2)} = 67.34 \text{ ppm} \\ \text{Si 2} &= 33.62 \times \text{dilution factor (2)} = 67.24 \text{ ppm}\end{aligned}$$

Calculation of expected Ca + Si in "URAN*SYN" in "0.1 M HClO_4 " if only monophase is present.

$$\text{Ca ppm} = \frac{46.08 \text{ g Ca}}{1 \text{ mol uranophase}} \times \frac{1 \text{ mol uranophase}}{831 \text{ g uranophase}} \times \frac{.05 \text{ g uranophase}}{100 \text{ ml}} = 24.1 \text{ ppm}$$

$$\text{Si ppm} = \frac{56.00 \text{ g Si}}{1 \text{ mol uranophase}} \times \frac{1 \text{ mol uranophase}}{831 \text{ g uranophase}} \times \frac{.05 \text{ g uranophase}}{100 \text{ ml}} = 33.7 \text{ ppm}$$

The low Ca content in "URAN*SYN" in 0.1 M HClO_4 (18.6 as opposed to 24.1) with respect to calculated value and high Si content with respect to calculated value (67 ppm as opposed to 33.7 ppm) and presence of significant Na (~10 ppm) suggests that "URAN*SYN" contains not only synthesized uranophane but some (unreacted) reagent (probably Na-silicate).

7/27/95 JP

The pressure vessels for "Soddyite synthesis 1" and "Soddyite synthesis 2" were cooled and opened.

Contents of vessels included yellow solids + solution.

The pH of the solution in the vessels was measured:

Soddyite synthesis 1 - pH = 4.54

Soddyite synthesis 2 - pH = 1.89

⁵⁸
A portion of each solid was removed from container + placed in a glass beaker. The solid was washed several times with DI water and dried. The solid for "Soddyite synthesis 2" appeared to possibly be unreacted reagents; possibly pH was too low for soddyite growth.

The two solids were powdered and analyzed by XRD using procedure on p 6.

Inspection of solid in "Soddyite synthesis 2" indicated that it was unreacted silicic acid and on XRD was not perfluorinated. Materials were discarded.

7/28/95 JP

"Soddyite synthesis 3"

A 3rd soddyite synthesis experiment was initiated.

The procedure on p 44-45 was followed with the following modifications in the procedure.

a. 2. 21g U-acetate + 13g silicic acid were placed in a 2000 ml beaker liner.

c. Approximately 2000 ml of H₂O was placed in enlarged flask + pH was lowered to 5.0 by addition of 0.1 M HCl.

JP 7/28/95
F. Transfer about 650 ml of degassed H₂O to vessel.

This experiment was conducted to determine if soddyite can be synthesized from silicic acid using a higher start pH (5.0). In the previous experiment - "soddyite synthesis 2" silicic acid did not dissolve.

"Soddyite synthesis 3"

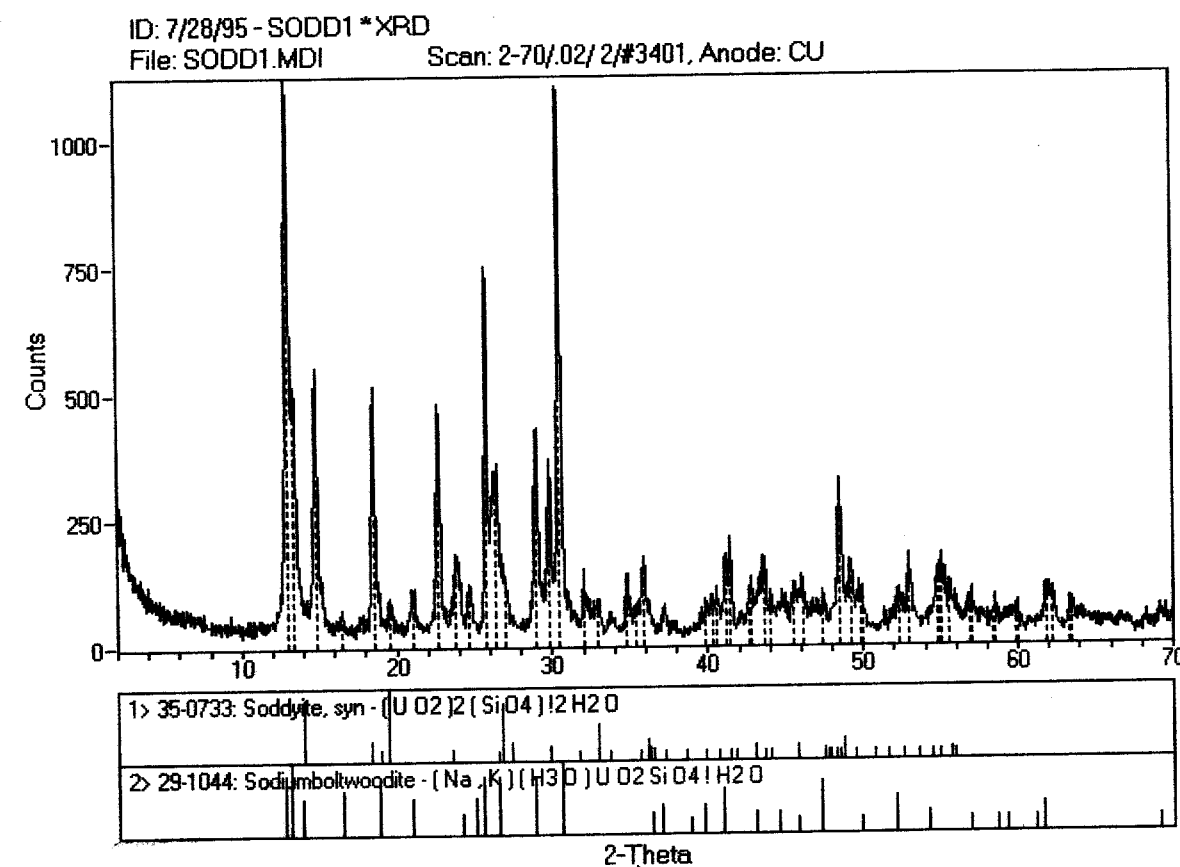
Wt erlenmeyer flask with degassed H_2O = 2331.9 g
before transfer to vessel.

Wt erlenmeyer flask with degassed H_2O = 1675.0 g
after transfer to vessel

Wt degassed H_2O transferred to vessel = 656.9 g

4/28/95 JF

XRD Results of "Soddyite synthesis 1"
solid



Solid does not have structure of
soddyite. It more closely
resembles Na-boltwoodite.

8/7/95 JF

Pressure vessel for "Soddyite synthesis 3"
experiment was cooled + opened

Contents included solution + yellow
solid.

The pH of the solution was measured:

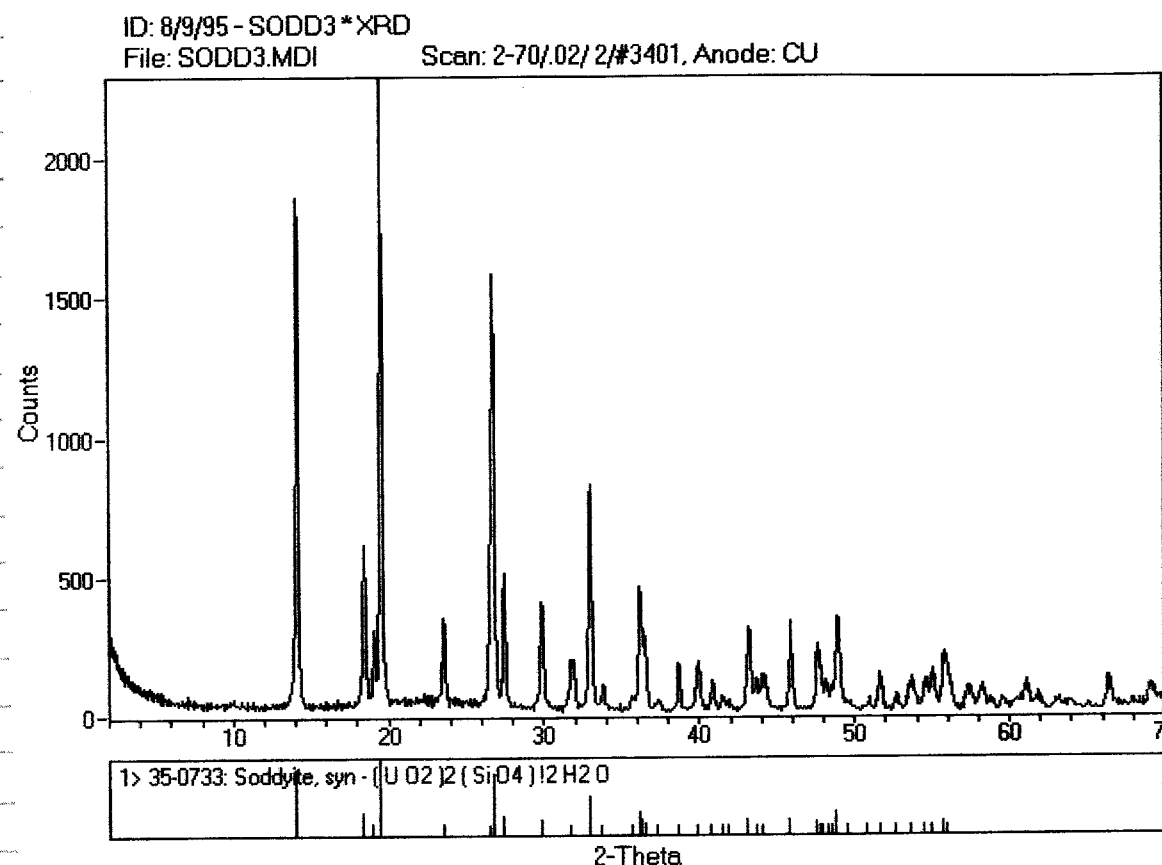
pH = 3.07

A portion of the solid was removed
from the beaker container + placed
in a glass beaker. The solid was
washed with DI water + dried.

The solid was powdered + analyzed by
XRD using procedure ~ p6.

Remaining yellow solid was filtered,
dried, and placed in a glass
container. The glass container was
labeled "SODD* SYN* 3".

Results of XRD analysis of "soddyite
synthesis 3" solid.



XRD indicates presence of soddyite.
However, inspection of solid under a
binocular microscope showed that
silicic acid was also present. Therefore,
soddyite will be separated from
the starting silicic acid reagent by
density separation.

8/9/95

uranophane synthesis using silicic acid - an experiment was initiated to attempt to synthesize uranophane using silicic acid instead of Na-silicates.

This experiment will be referred to as "uranophane Synthesis 5".

The procedure on the following page was used.

8/10/95
0800h
JP

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikrýl
REVISION NO.: 1

DATE WRITTEN: March 7, 1995
DATE REVISED: Aug 9, 1995

OBJECTIVE: to synthesize uranophane $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 2.5\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 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 ~~944231~~)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume $4\text{H}_2\text{O}$ - MW = 132 (lot 951303)
Calcium acetate monohydrate $[\text{Ca}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$; MW=176.19
(lot ~~319617/1~~ 194) 944231 JP
9 M HCl - diluted from lot 945500 9/7/95

PROCEDURE:

The procedure for uranophane synthesis is based on Cesbron et al., 1993. Reagents will be mixed in the stoichiometric ratio $\text{Ca}:\text{U}:\text{Si} = 2:1:1$. Although Cesbron et al. do not give information on the amount of solid and H_2O used, they state that good crystallinity is obtained if pH is less than 5.

- Place 17.5 g Ca-acetate, 21 g U-acetate, and 6.5 g silicic acid in a 2000 ml teflon liner and place liner in reaction vessel.
- Cap and seal vessel and evacuate using a vacuum pump to remove $\text{CO}_2(\text{g})$.
- Transfer approximately 1000 ml of ultrapure H_2O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the H_2O by boiling on a hotplate. After degassing cap erlenmeyer flask with a rubber stopper so that air cannot enter the flask.

- e) After cooling record weight of the erlenmeyer flask with the degassed H₂O.
- f) Transfer approximately 650 g of the degassed H₂O into the vessel by pulling in by vacuum. After the transfer, record weight of the erlenmeyer flask with remaining degassed H₂O to determine actual weight of H₂O introduced.
- g) Raise and set the temperature of the solution in the reaction vessel to 150C using the temperature controller and allow the reaction to proceed for one week or longer.

3/18/95 JP

"Soddyite Synthesis 4"

A 4th soddyite synthesis experiment was initiated

The procedure on p 44-45 was modified as follows.

a) 21 g U acetate & 8.8 g silicic acid were placed in a 2000 ml teflon liner.

c) Approximately 1000 ml of H₂O was placed in an erlenmeyer flask. pH was not modified

f) Transfer about 650 ml of degassed H₂O to vessel.

8/18/95 JP

Vessel for "Uranophane Synthesis 5" was cooled + opened.

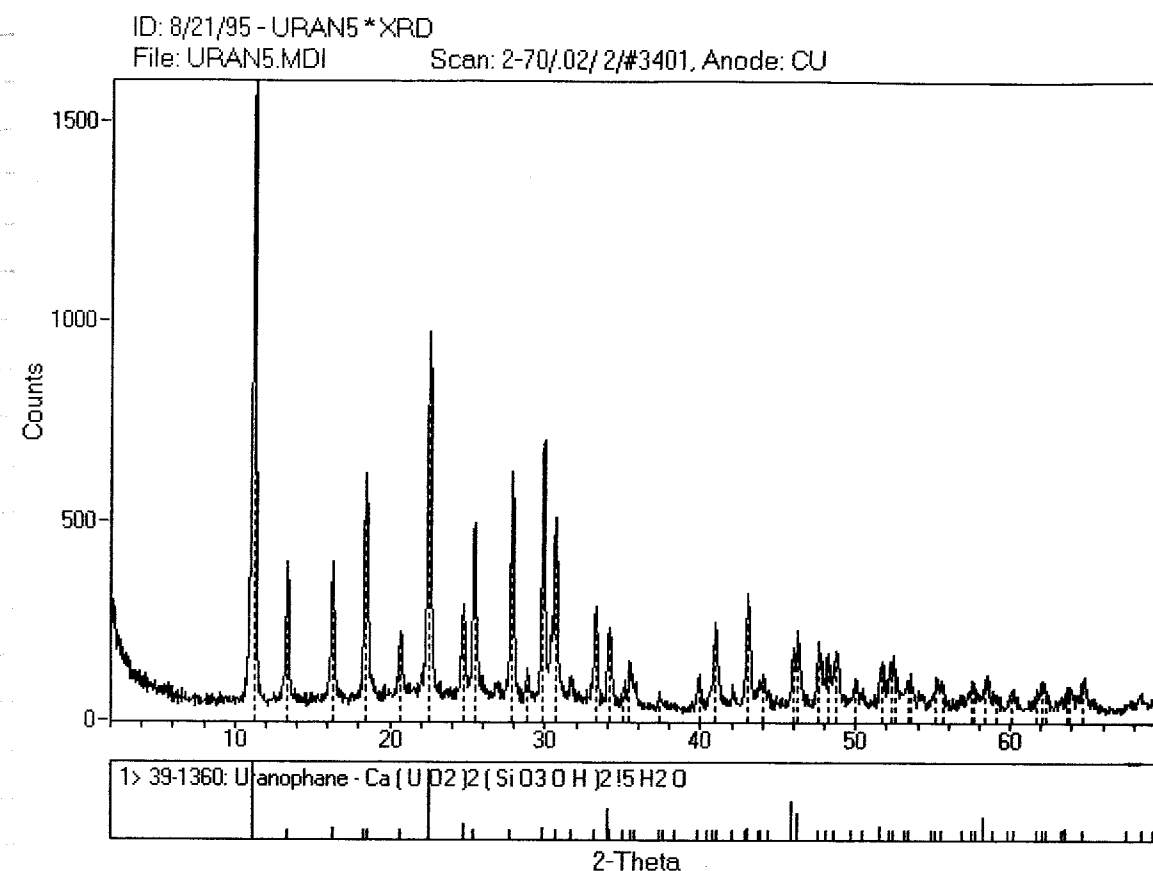
pH of solution in vessel = 4.13

A portion of the solid was removed, washed with DI water a few times + dried.

Inspection of solid using binocular microscope indicated the presence of silicic acid (undissolved).

Solid was powdered and analyzed by XRD using powder - μ PD To determine presence of U minerals.

8/28/95 Results of XRD of URANOPHANE synthesis 5 experiment



XRD pattern shows presence of Uranophane. However, visual inspection of solid also shows presence of undissolved silicic acid.

The solid was filtered, dried, and placed in a plastic vial. The sample was labeled URAN*SYN*5

8/28/95 JP

Results of chemical analysis of
minerals sent to Div 01
are shown below

Sample ID	Analysis	Sample Result
20A	Si as SiO ₂	16.6%
	Mg as MgO	0.300%
	Ca as CaO	6.31%
	P as P ₂ O ₅	0.179%
	LOI (Loss @ 1000°C)	13.0%
	U as U ₃ O ₈	60.0%
	TOTAL	96.3%

Sample ID	Analysis	Duplicate Result
20A	Si as SiO ₂	16.8%
	Mg as MgO	0.318%
	Ca as CaO	6.32%
	P as P ₂ O ₅	0.142%
	LOI (Loss @ 1000°C)	13.0%
	U as U ₃ O ₈	60.1%
	TOTAL	96.6%

20A = URAW * SYN

20A - URAW * SYN

Analysis	Sample Result (ug/g)	Duplicate Result (ug/g)	Detection Limit (ug/g)
Aluminum	<10	<10	10
Antimony	<10	<10	10
Arsenic	<25	<25	25
Barium	<4	<4	4
Beryllium	<2	<2	2
Boron	<20	<20	20
Cadmium	<4	<4	4
Chromium	<4	<4	4
Cobalt	<10	<10	10
Copper	<4	<4	4
Iron	154	177	10
Lead	<10	<10	10
Lithium	<4	<4	4
Manganese	<4	<4	4
Molybdenum	<4	<4	4
Nickel	<12	<12	12
Potassium	<40	<40	40
Selenium	<10	<10	10
Silver	<10	<10	10
Sodium	<25	<25	25
Strontium	<10	<10	10
Thallium	<10	<10	10
Tin	<20	<20	20
Titanium	<8	<8	8
Vanadium	<20	<20	20
Zinc	<20	<20	20

20A - URAW * SYN

20B - RGM-1

Sample ID	Analysis	Sample Result
20B	Si as SiO ₂	74.7%
	Al as Al ₂ O ₃	12.6%
	Fe as Fe ₂ O ₃	1.72%
	Mg as MgO	0.251%
	Ca as CaO	1.06%
	Na as Na ₂ O	4.36%
	K as K ₂ O	4.58%
	Ti as TiO ₂	0.248%
	LOI (Loss @ 1000°C)	1.11%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	866	4
Beryllium	2	2
Boron	<20	20
Cadmium	<4	4
Chromium	12	4
Cobalt	<10	10
Copper	16	4
Lead	25	10
Lithium	64	4
Manganese	282	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	126	76
Selenium	<10	10
Silver	<10	10
Strontium	106	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	32	20

20C - U R A N * S Y N * 3

Sample ID	Analysis	Sample Result
20C	Si as SiO ₂	17.5%
	Mg as MgO	0.417%
	Ca as CaO	6.53%
	P as P ₂ O ₅	0.176%
	LOI (Loss @ 1000°C)	12.5%
	U as U ₃ O ₈	62.2%
	TOTAL	99.3%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Aluminum	<10	10
Antimony	<10	10
Arsenic	<25	25
Barium	<4	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	<4	4
Cobalt	<10	10
Copper	<4	4
Iron	120	10
Lead	<10	10
Lithium	<4	4
Manganese	<4	4
Molybdenum	<4	4
Nickel	<12	12
Potassium	<40	40
Selenium	<10	10
Silver	<10	10
Sodium	<25	25
Strontium	<10	10
Thallium	<10	10
Tin	<20	20
Titanium	<8	8
Vanadium	<20	20
Zinc	<20	20

20D - URAN * SYW

Sample ID	Analysis	Sample Result
20D	Si as SiO ₂	17.5%
	Mg as MgO	0.392%
	Ca as CaO	6.47%
	P as P ₂ O ₅	0.167%
	LOI (Loss @ 1000°C)	13.0%
	U as U ₃ O ₈	61.1%
	TOTAL	98.6%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Aluminum	<10	10
Antimony	<10	10
Arsenic	<25	25
Barium	<4	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	<4	4
Cobalt	<10	10
Copper	<4	4
Iron	221	10
Lead	<10	10
Lithium	<4	4
Manganese	<4	4
Molybdenum	<4	4
Nickel	<12	12
Potassium	<40	40
Selenium	<10	10
Silver	<10	10
Sodium	<25	25
Strontium	<10	10
Thallium	<10	10
Tin	<20	20
Titanium	<8	8
Vanadium	<20	20
Zinc	<20	20

20E - STM-1

Sample ID	Analysis	Sample Result
20E	Si as SiO ₂	67.2%
	Al as Al ₂ O ₃	18.7%
	Fe as Fe ₂ O ₃	5.35%
	Ca as CaO	1.11%
	Na as Na ₂ O	10.6%
	K as K ₂ O	5.07%
	Ti as TiO ₂	0.135%
	Mn as MnO	0.248%
	P as P ₂ O ₅	0.195%
	LOI (Loss @ 1000°C)	1.70%
	TOTAL	110%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	588	4
Beryllium	9	2
Boron	<20	20
Cadmium	<4	4
Chromium	11	4
Cobalt	<10	10
Copper	4	4
Lead	16	10
Lithium	38	4
Magnesium	588	19
Molybdenum	<4	4
Nickel	<12	12
Selenium	<10	10
Silver	<10	10
Strontium	706	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	260	20

20F - 278

Sample ID	Analysis	Sample Result
20F	Si as SiO ₂	74.6%
	Al as Al ₂ O ₃	13.0%
	Fe as Fe ₂ O ₃	1.96%
	Mg as MgO	0.223%
	Ca as CaO	0.900%
	Na as Na ₂ O	5.23%
	K as K ₂ O	4.48%
	Ti as TiO ₂	0.226%
	Ba as BaO	0.106%
	LOI (Loss @ 1000°C)	0.691%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	6	4
Lead	15	10
Lithium	48	4
Manganese	403	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	115	80
Selenium	<10	10
Silver	<10	10
Strontium	65	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	59	20

20G - URAW * SYN * 3

Sample ID	Analysis	Sample Result
20G	Si as SiO ₂	17.5%
	Mg as MgO	0.378%
	Ca as CaO	6.22%
	P as P ₂ O ₅	0.159%
	LOI (Loss @ 1000°C)	12.8%
	U as U ₃ O ₈	58.5%
	TOTAL	95.6%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Aluminum	<10	10
Antimony	<10	10
Arsenic	<25	25
Barium	<4	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	<4	4
Cobalt	<10	10
Copper	<4	4
Iron	104	10
Lead	<10	10
Lithium	<4	4
Manganese	<4	4
Molybdenum	<4	4
Nickel	<12	12
Potassium	<40	40
Selenium	<10	10
Silver	<10	10
Sodium	<25	25
Strontium	<10	10
Thallium	<10	10
Tin	<20	20
Titanium	<8	8
Vanadium	<20	20
Zinc	<20	20

20H - Na - SA 2-1

Sample ID	Analysis	Sample Result
20H	Si as SiO ₂	59.6%
	Al as Al ₂ O ₃	16.0%
	Fe as Fe ₂ O ₃	1.41%
	Mg as MgO	5.35%
	Ca as CaO	0.803%
	Na as Na ₂ O	2.83%
	K as K ₂ O	0.144%
	Ti as TiO ₂	0.195%
	Mn as MnO	0.180%
	LOI (Loss @ 1000°C)	14.9%
	TOTAL	101%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	266	4
Beryllium	4	2
Boron	<20	20
Cadmium	<4	4
Chromium	25	4
Cobalt	<10	10
Copper	<4	4
Lead	41	10
Lithium	256	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Strontium	112	10
Thallium	<10	10
Tin	<20	20
Vanadium	62	20
Zinc	75	20

20I - Cheto

Sample ID	Analysis	Sample Result
20I	Si as SiO ₂	46.7%
	Al as Al ₂ O ₃	12.5%
	Fe as Fe ₂ O ₃	1.11%
	Mg as MgO	4.43%
	Ca as CaO	1.99%
	K as K ₂ O	0.153%
	Ti as TiO ₂	0.161%
	LOI (Loss @ 1000°C)	25.5%
	TOTAL	92.6%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	299	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	<4	4
Lead	26	10
Lithium	212	4
Manganese	562	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Sodium	605	25
Strontium	286	10
Thallium	<10	10
Tin	<20	20
Vanadium	57	20
Zinc	53	20

20J - SAZ - Na

Sample ID	Analysis	Sample Result
20J	Si as SiO ₂	53.5%
	Al as Al ₂ O ₃	14.3%
	Fe as Fe ₂ O ₃	1.27%
	Mg as MgO	4.84%
	Na as Na ₂ O	3.44%
	Ti as TiO ₂	0.185%
	LOI (Loss @ 1000°C)	23.4%
	TOTAL	101%

Sample ID	Analysis	Duplicate Result
20J	Si as SiO ₂	55.0%
	Al as Al ₂ O ₃	14.8%
	Fe as Fe ₂ O ₃	1.31%
	Mg as MgO	5.00%
	Na as Na ₂ O	3.61%
	Ti as TiO ₂	0.190%
	LOI (Loss @ 1000°C)	23.4%
	TOTAL	103%

20J - SAZ - Na

Analysis	Sample Result (ug/g)	Duplicate Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	<10	10
Arsenic	<25	<25	25
Barium	53	53	4
Beryllium	4	3	2
Boron	<20	<20	20
Cadmium	<4	<4	4
Calcium	253	251	20
Chromium	8	12	4
Cobalt	<10	<10	10
Copper	<4	<4	4
Lead	26	29	10
Lithium	226	229	4
Manganese	263	263	4
Molybdenum	<4	<4	4
Nickel	<12	<12	12
Phosphorus	<80	<80	80
Potassium	530	550	40
Selenium	<10	<10	10
Silver	<10	<10	10
Strontium	<10	<10	10
Thallium	<10	<10	10
Tin	<20	<20	20
Vanadium	<20	<20	20
Zinc	63	61	20

20K - Cheto

Sample ID	Analysis	Sample Result
20K	Si as SiO ₂	58.1%
	Al as Al ₂ O ₃	15.3%
	Fe as Fe ₂ O ₃	1.37%
	Mg as MgO	5.46%
	Ca as CaO	2.49%
	K as K ₂ O	0.191%
	Ti as TiO ₂	0.199%
	LOI (Loss @ 1000°C)	25.5%
	TOTAL	109%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	315	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Chromium	9	4
Cobalt	<10	10
Copper	<4	4
Lead	21	10
Lithium	221	4
Manganese	594	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Selenium	<10	10
Silver	<10	10
Sodium	629	25
Strontium	299	10
Thallium	<10	10
Tin	<20	20
Vanadium	61	20
Zinc	59	20

20L - SAZ - Na

Sample ID	Analysis	Sample Result
20L	Si as SiO ₂	61.4%
	Al as Al ₂ O ₃	16.7%
	Fe as Fe ₂ O ₃	1.47%
	Mg as MgO	5.63%
	Na as Na ₂ O	4.07%
	Ti as TiO ₂	0.213%
	LOI (Loss @ 1000°C)	24.4%
	TOTAL	114%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	52	4
Beryllium	3	2
Boron	<20	20
Cadmium	<4	4
Calcium	261	20
Chromium	8	4
Cobalt	<10	10
Copper	<4	4
Lead	20	10
Lithium	230	4
Manganese	258	4
Molybdenum	<4	4
Nickel	<12	12
Phosphorus	<80	80
Potassium	560	40
Selenium	<10	10
Silver	<10	10
Strontium	<10	10
Thallium	<10	10
Tin	<20	20
Vanadium	<20	20
Zinc	58	20

20M-SCo-1

Sample ID	Analysis	Sample Result
20M	Si as SiO ₂	68.6%
	Al as Al ₂ O ₃	14.4%
	Fe as Fe ₂ O ₃	5.47%
	Mg as MgO	2.81%
	Ca as CaO	2.64%
	Na as Na ₂ O	1.12%
	K as K ₂ O	3.29%
	Ti as TiO ₂	0.578%
	P as P ₂ O ₅	0.238%
	LOI (Loss @ 1000°C)	10.0%
	TOTAL	109%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	613	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	74	4
Cobalt	16	10
Copper	29	4
Lead	28	10
Lithium	48	4
Manganese	419	4
Molybdenum	<4	4
Nickel	29	12
Selenium	<10	10
Silver	<10	10
Strontium	174	10
Thallium	<10	10
Tin	<20	20
Vanadium	142	20
Zinc	119	20

20N - SC0-1

Sample ID	Analysis	Sample Result
20N	Si as SiO ₂	74.7%
	Al as Al ₂ O ₃	14.8%
	Fe as Fe ₂ O ₃	5.62%
	Mg as MgO	2.93%
	Ca as CaO	2.74%
	Na as Na ₂ O	1.19%
	K as K ₂ O	3.43%
	Ti as TiO ₂	0.613%
	P as P ₂ O ₅	0.271%
	LOI (Loss @ 1000°C)	10.0%
	TOTAL	116%

Analysis	Sample Result (ug/g)	Detection Limit (ug/g)
Antimony	<10	10
Arsenic	<25	25
Barium	582	4
Beryllium	<2	2
Boron	<20	20
Cadmium	<4	4
Chromium	66	4
Cobalt	19	10
Copper	28	4
Lead	23	10
Lithium	48	4
Manganese	388	4
Molybdenum	<4	4
Nickel	28	12
Selenium	<10	10
Silver	<10	10
Strontium	165	10
Thallium	<10	10
Tin	<20	20
Vanadium	130	20
Zinc	108	20

8/29/95 JP

Vessel for soddyite synthesis 4" was cooled + opened

pH of solution in vessel = 3.17

A portion of the solid was removed, washed with DI water + dried.

Inspection of solid indicated presence of unreacted silicic acid.

The solid was filtered, dried, and placed in a plastic vial. The sample was labeled SODD*SYW*4.

9/7/95

JP

A 5th soddyite synthesis experiment was initiated using the following procedure.

SODDYITE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 1

DATE WRITTEN: July 3, 1995
DATE REVISED: Sept 7, 1995

OBJECTIVE: to synthesize soddyite $[(\text{UO}_2)_2\text{SiO}_4 \cdot 2\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/ $^{\circ}\text{C}$ meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Hot plate (Thermolyne Type 13100)
Spectra Mesh teflon (70 micron openings)
Teflon beading
Teflon centrifuge tubes (50 ml)

SUPPLIES: pH buffers (pH = 1, 2, 4)
ultrapure water
glassware and plasticware as needed
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 944231)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume no H_2O ; MW = 60 (lot 951303)
9 M HCl - diluted from lot 945500

PROCEDURE:

- 1400hr JP
- Reagents for soddyite synthesis will be mixed in the stoichiometric ratio U:Si = 1:2.
- Place 21 g U-acetate in a 2000 ml teflon liner.
 - Place 6 g silicic acid (coarse fraction separated by density settling) in a 50 ml teflon centrifuge tube. Cover tube opening with spectra mesh and secure with teflon beading. Place tube in the teflon liner.
 - Transfer approximately 650 g of ultrapure H_2O to the teflon liner and place in the pressure vessel. Make sure H_2O enters centrifuge tube and then seal vessel.
 - Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller.
 - Degas the pressure vessel once it reaches about $>140^{\circ}\text{C}$ by opening the air intake valve attached to the pressure gauge assembly.
 - Close intake valve after degassing and allow the reagents to react for about 10 days.

9/8/95 JF

A sixth uranophane synthesis experiment was initiated using the following procedure.

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 2

DATE WRITTEN: March 7, 1995
DATE REVISED: Sept 8, 1995

OBJECTIVE: to synthesize uranophane $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 2.5\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/ $^{\circ}\text{C}$ meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)
Spectra mesh teflon (70 micron openings)
Teflon beading

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
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 (analysis no. 337419/1 1094)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume no H_2O ; MW = 60 (lot 951303)
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 945500

PROCEDURE:

JF

0900 hr The procedure for uranophane synthesis is based on Cesbron et al., 1993. Reagents will be mixed in the stoichiometric ratio $\text{Ca}:\text{U}:\text{Si} = 2:1:1$. Although Cesbron et al. do not give information on the amount of solid and H_2O used, they state that good crystallinity is obtained if pH is less than 5.

- Place 17.5 g Ca-acetate and 21 g U-acetate in a 2000 ml teflon liner.
- Place 3 g silicic acid (coarse fraction separated by density settling) in spectra mesh. Wrap mesh and secure with teflon beading. Place mesh in teflon liner.
- Transfer approximately 1000 ml of ultrapure H_2O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.

- d) Transfer approximately 650 g of the degassed H₂O to the teflon liner and place in pressure vessel.
- e) Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller.
- f) Degas the pressure vessel once it reaches >140°C by opening the air intake valve attached to the pressure gauge assembly.
- g) Close intake valve after degassing and allow reagents to react for about 10 days.

9/18/95 JP

Vessel for 'soddyite synthesis 5' was cooled + opened.

pH of solution = 3.13

A portion of the solid was removed, washed with DI water + dried.

Inspection of dried solid indicated a yellow solid; no silicic acid was visually present.

The solid that formed outside the tube containing silicic acid was filtered, dried, and labeled 'SODD*SYN*SA'. The solid inside the tube was filtered + washed thru a 10 µm mesh and then dried. This solid was labeled 'SODD*SYN*SB'.

9/20/95 JP

'SODD*SYN*SA' was washed with boiling DI water several times to remove any remaining starting reagents.

Portions of SODD*SYN*SA + SODD*SYN*SB were powdered and analyzed by XRD using procedure on p6.

9/18/95 JP

Vessel for 'uranophane synthesis 6' was cooled + opened.

pH of solution = 3.67

A portion of the solid was removed, washed with DI water + dried.

Inspection of the dried solid indicated that no silicic acid was present.

The solid that found outside the mesh was filtered, dried, and labeled URAN*SYN*6A

The solid inside the mesh was filtered and washed thru a 10 μ m mesh then dried + labeled "URAN*SYN*6B".

9/20/95 JP

URAN*SYN*6A was washed several times with boiling DI water to remove any starting reagents.

URAN*SYN*6A and URAN*SYN*6B were analyzed by XRD using procedure on p6.

Visual inspection of URAN*SYN*6A indicates presence of a orange red material; possibly goethite.

9/21/95

Two additional uranophane synthesis experiments were initiated: 'uranophane synthesis 7' and 'uranophane synthesis 8'.

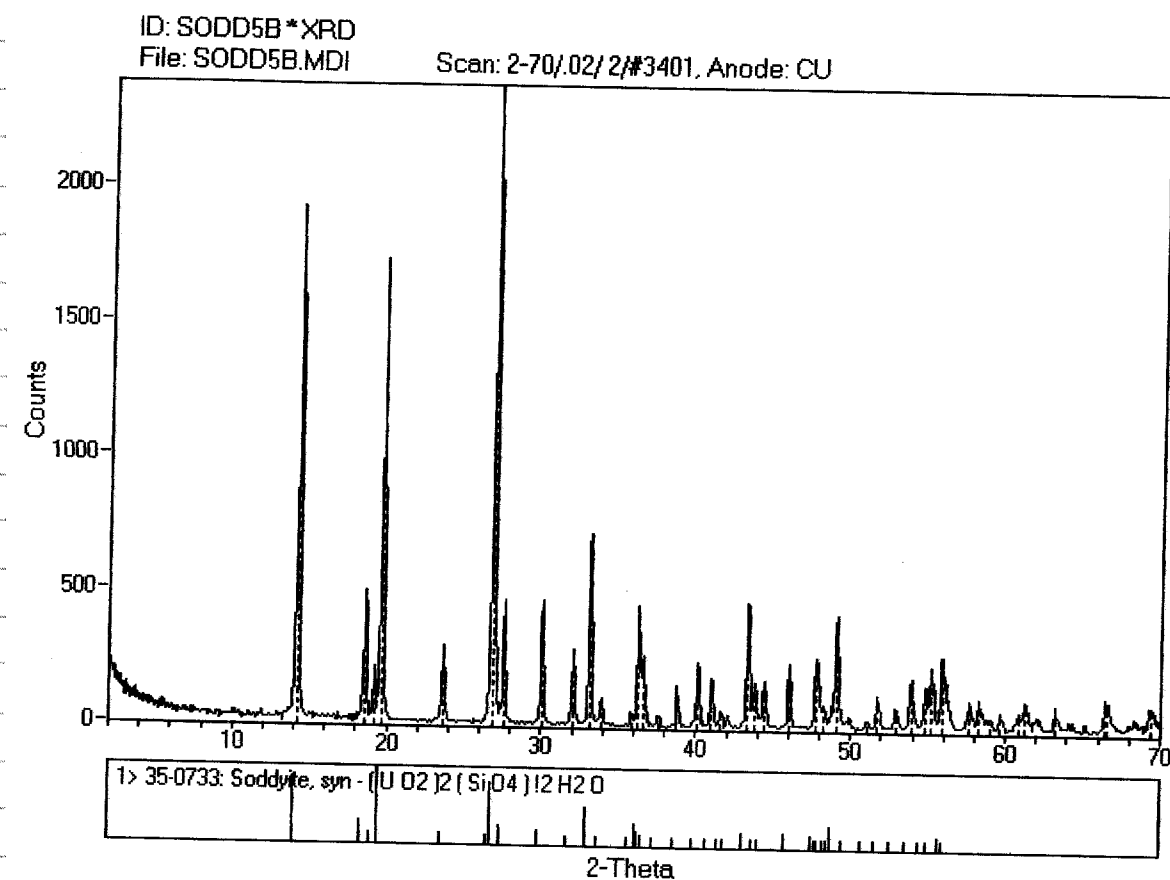
These experiments will follow procedure on p 95; however the amount of reagents + solution added to the vessel will be doubled.

42 g U acetate, 35 g Ca-acetate, and 6 g of silicic acid will be added to Teflon liner and 1300 g H_2O will be added.

In 'uranophane synthesis 7' silicic acid was wrapped in spectra mesh while in 'uranophane synthesis 8' silicic acid was placed in a teflon tube + covered with spectra mesh.

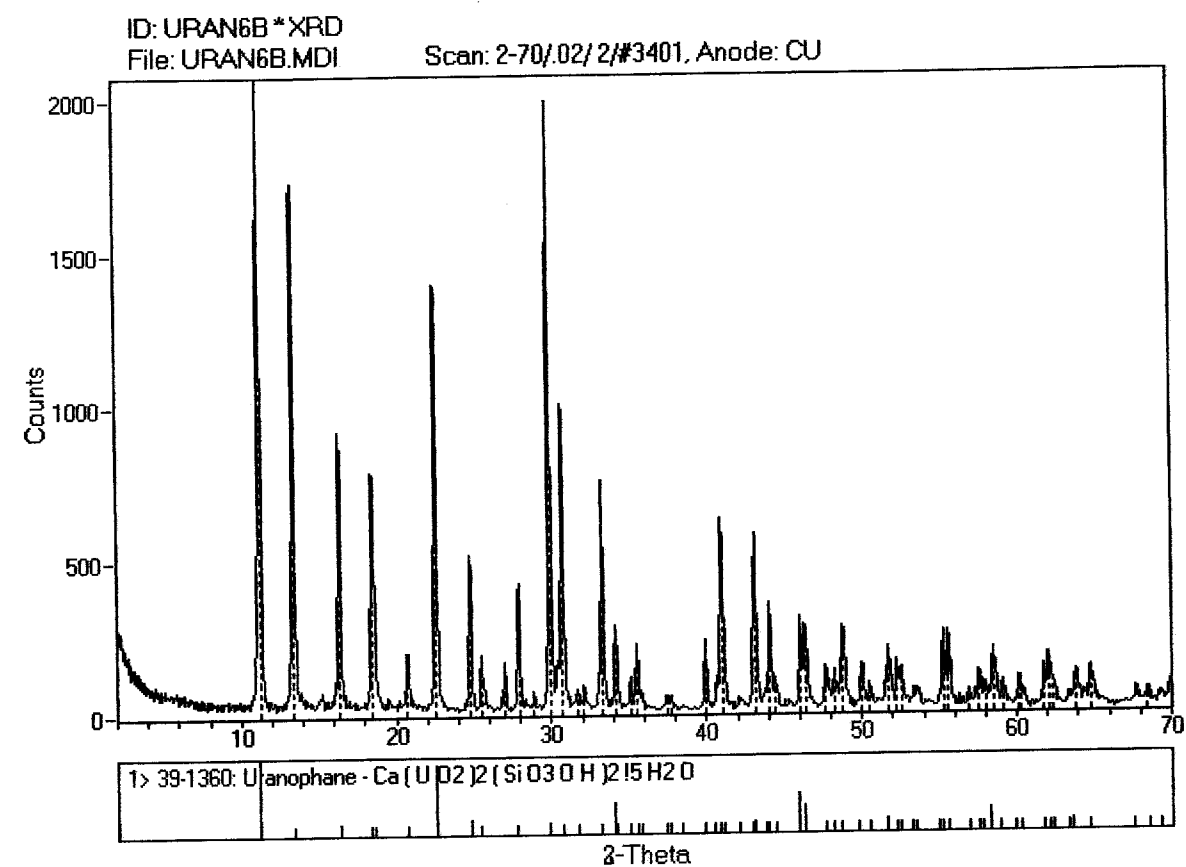
9/21/95 JP

Results of XRD analysis of SODD*SYN*5B

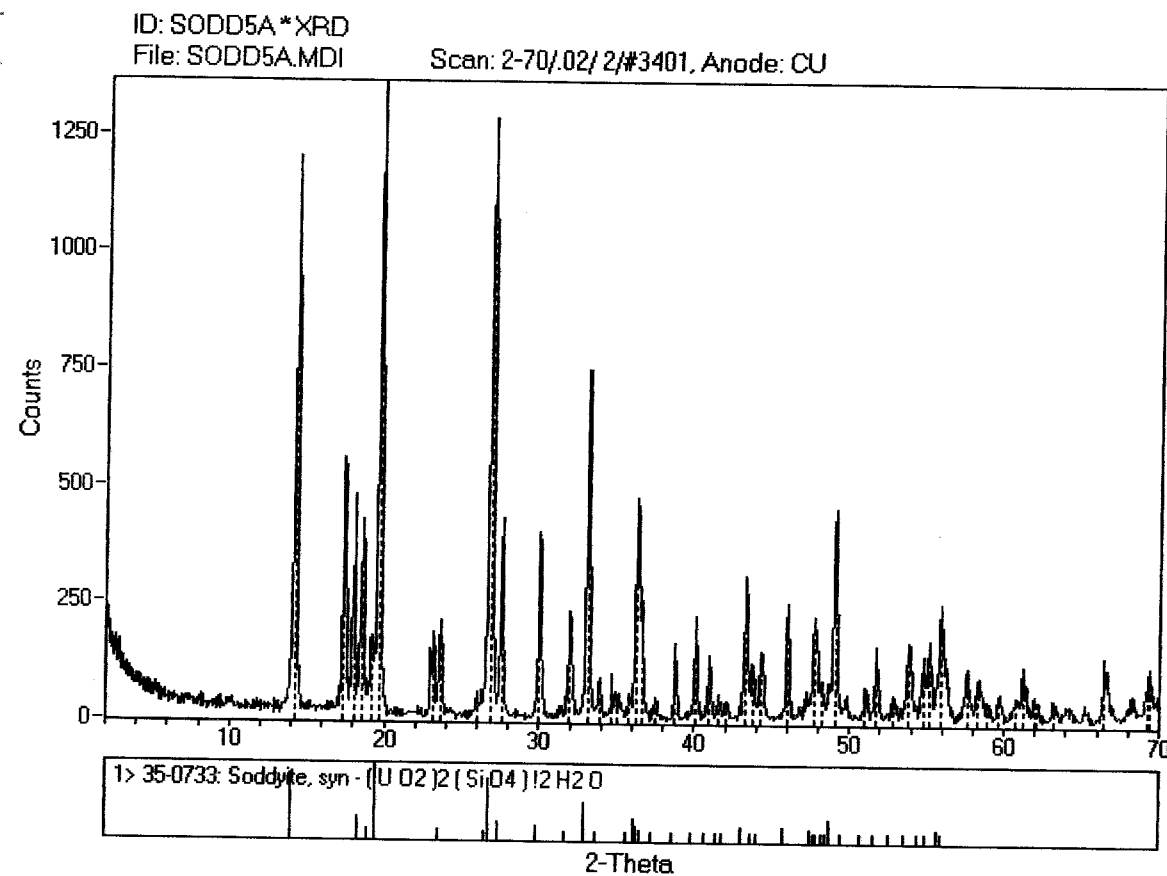


9/21/95

Results of XRD analysis of URAN*SYN*6B



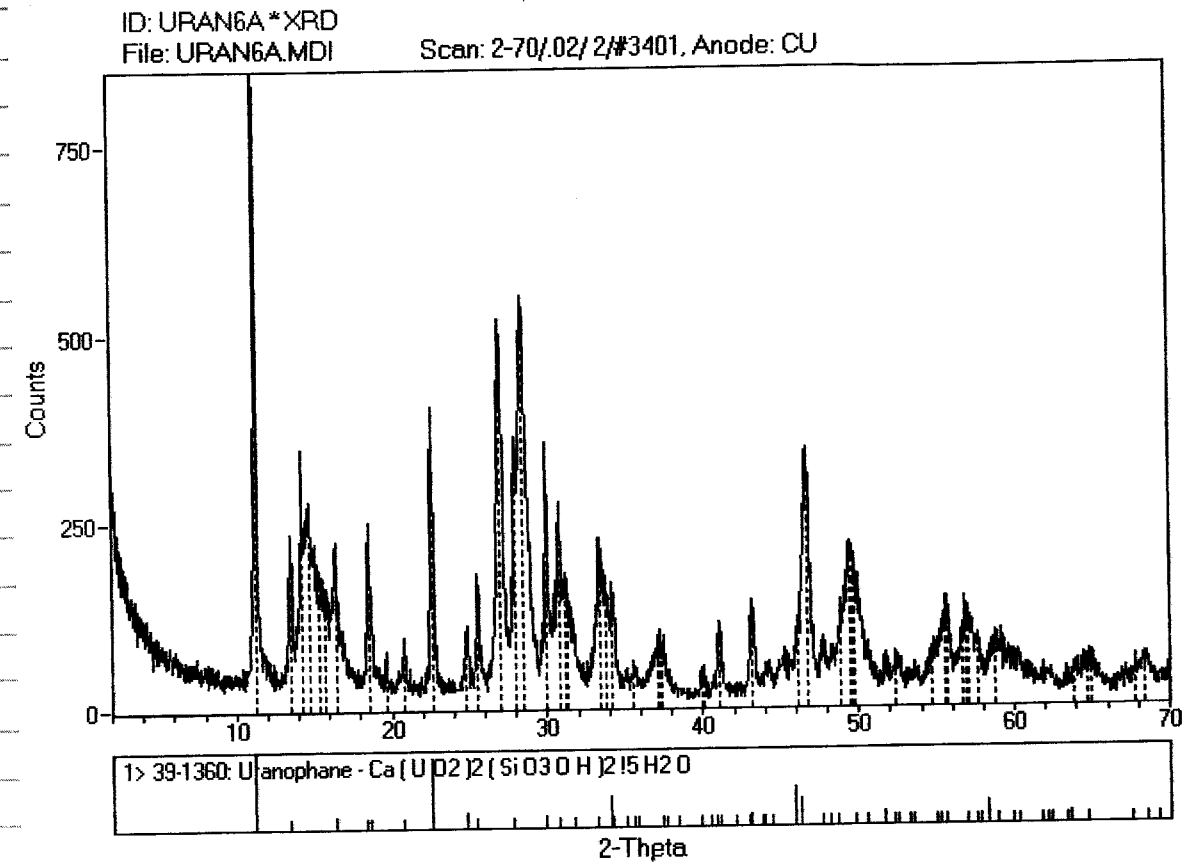
9/25/95 JH

Results of XRD analysis of SODD5A*SYN*5A
SODD5A JH 9/25

Pattern shows presence of peaks other than soddite; therefore other phases are present.

9/25/95 JH

Results of XRD analysis of URAN*SYN*6A.



Pattern shows presence of peaks other than uranophane + other phases are present.

10/2/95 JP

Vessels for 'uranophane synthesis 7' and 'uranophane synthesis 8' were cooled and aged.

pH of solutions

uranophane synthesis 7 = 4.14

uranophane synthesis 8 = 4.13

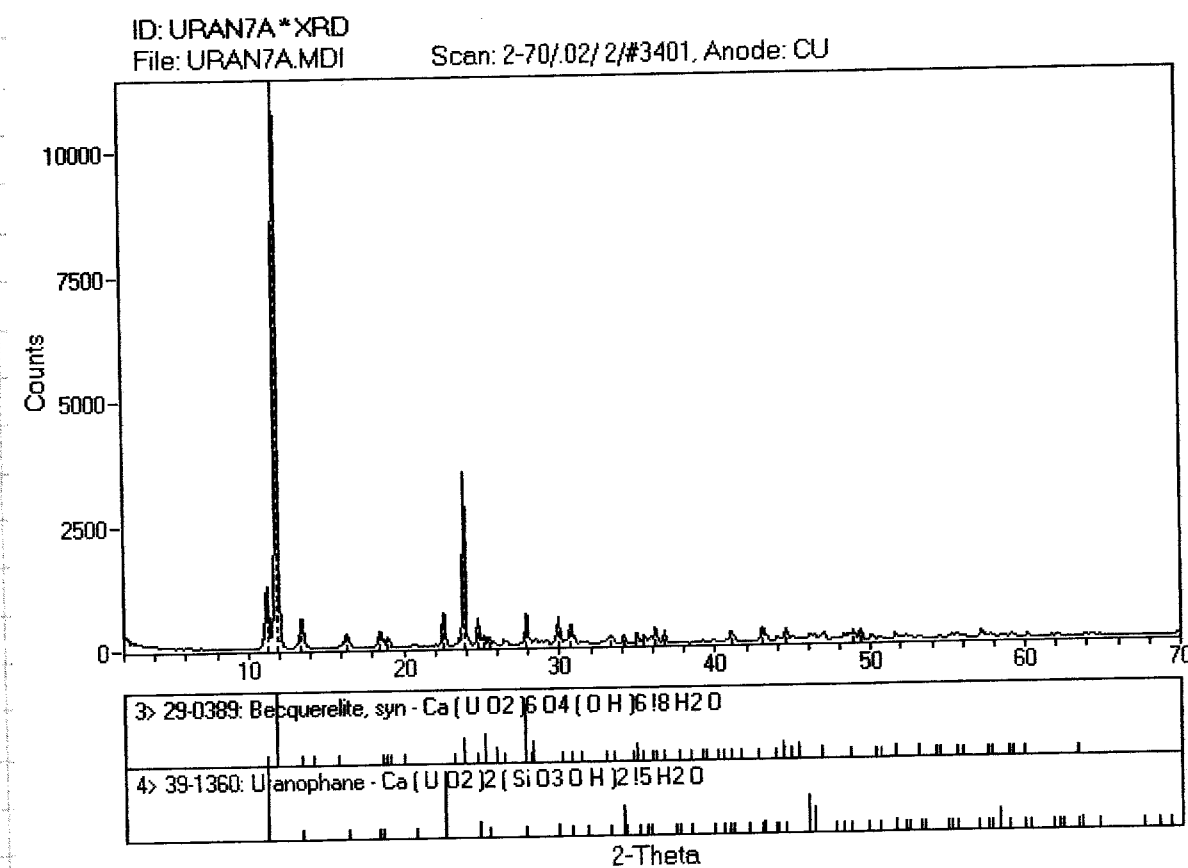
The solids that formed outside the mesh in 'uranophane synthesis 7' and outside the teflon tube in 'uranophane synthesis 8' were filtered, dried, placed in plastic vials, and labeled URAN*SYN*7A and URAN*SYN*8A.

The solids that formed inside the mesh and inside the teflon tube were filtered thru a 10 μ m nylon mesh, then dried, placed in plastic vials, and labeled URAN*SYN*7B and URAN*SYN*8B.

The above 4 samples were analyzed by XRD using procedure on p6.

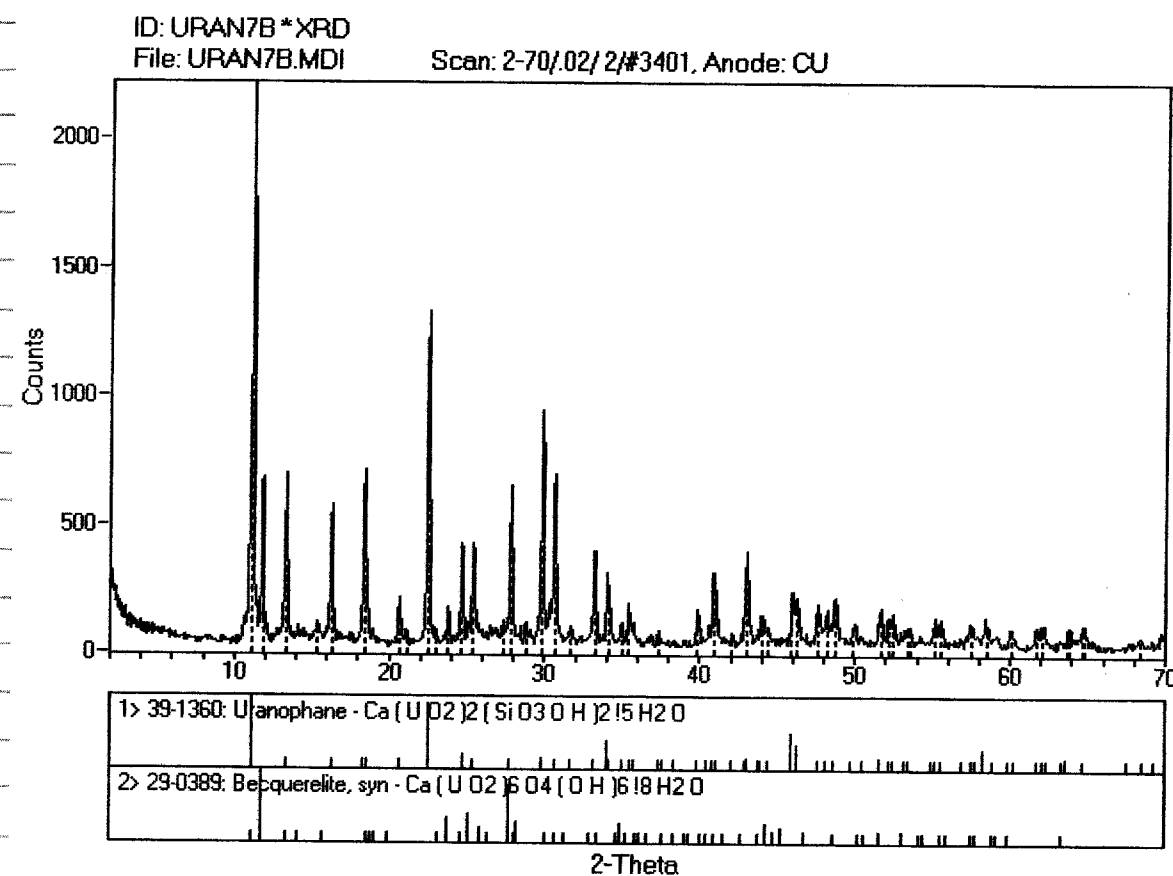
10/4/95

Results of XRD analysis URAN*SYN*7A



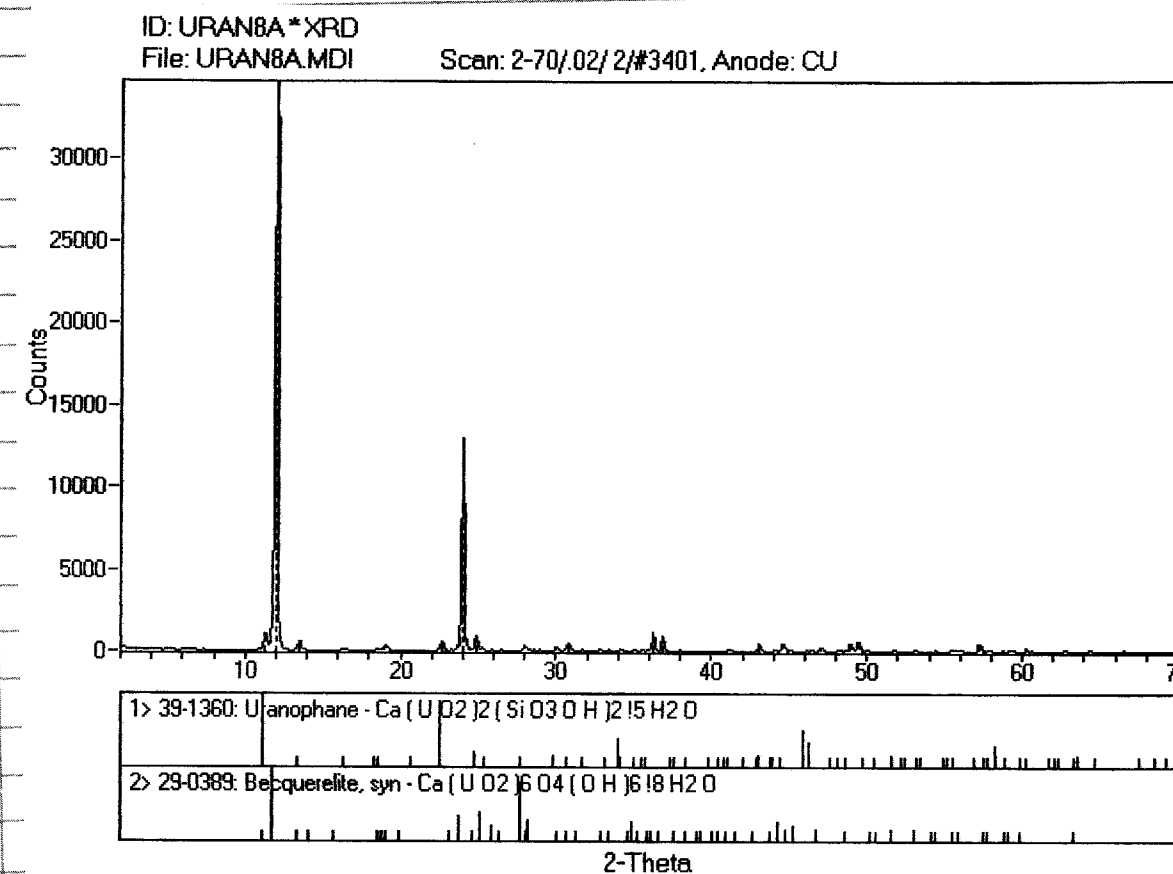
Pattern shows presence of ^{another major} phase other than uranophane. The phase appears to be bequerelite.

Results of XRD analysis of URAN*SYN*7B



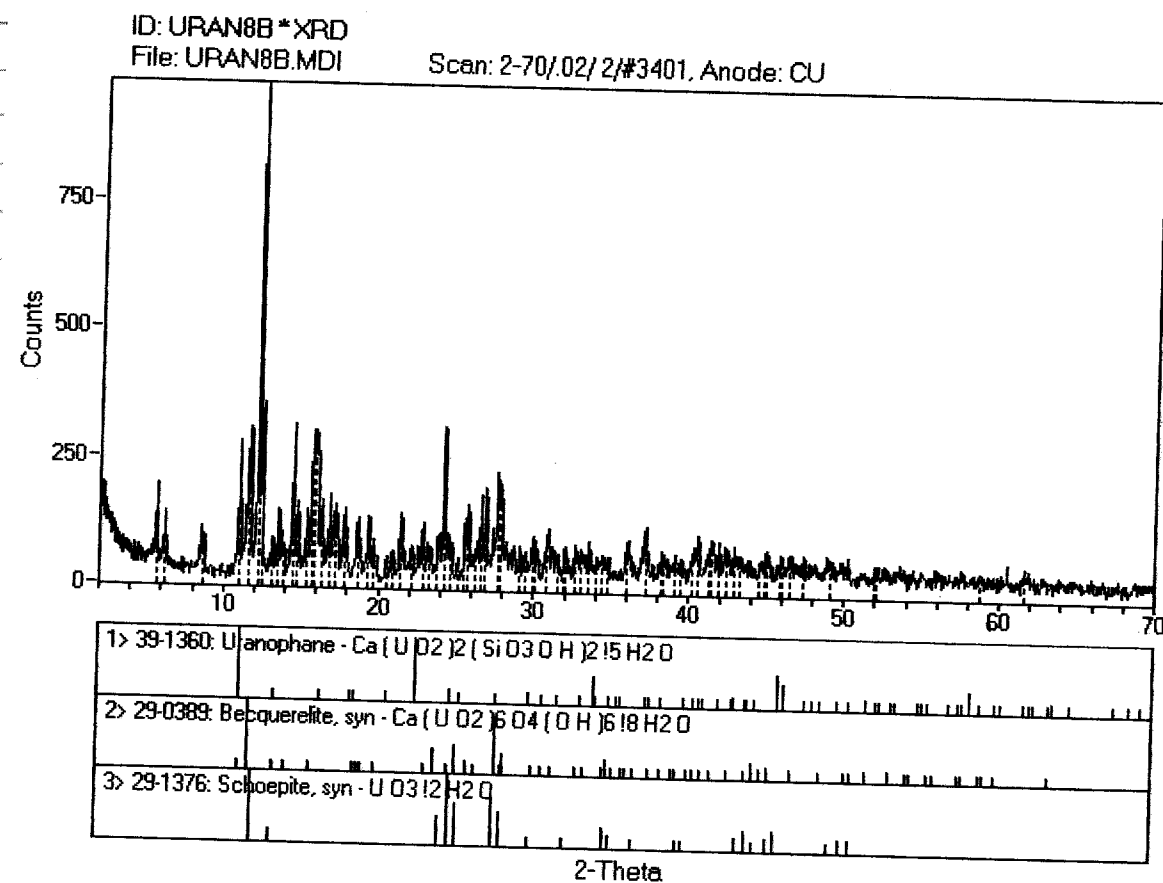
Both uranophane and becquerelite
are present.

Results of XRD analysis of URAN*SYN*8A



XRD pattern indicates major phase to
be becquerelite.

XRD results of URAN*SYN#8B



Major phases seem to be uranyl
oxide hydrates.

10/4/95

In experiments 'uranophane synth 7' and
'uranophane synth 8' uranyl oxide
hydrates (e.g. becquerelite) formed in
addition to uranophane. Apparently the
silicic acid within the mesh and
teflon tube became isolated from
the solution due to uranophane
precipitation. It was noted that
yellow uranyl minerals adorned
the silicic acid when the solid
was removed from the mesh
& tube.

To alleviate this occurrence teflon
mesh & tubes will not be used
to isolate the silicic acid in
subsequent experiments.

10/4/95

Uranophane synthesis experiments were initiated
using the following procedure.

These experiments will be referred to as
'Uranophane synthesis 9' + 'Uranophane synthesis 10'.

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 3

DATE WRITTEN: March 7, 1995
DATE REVISED: Oct 4, 1995

OBJECTIVE: to synthesize uranophane $[\text{Ca}(\text{UO}_2)_2(\text{SiO}_3\text{OH})_2 \cdot 2.5\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/ $^{\circ}\text{C}$ meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 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 (analysis no. 337419/1 1094)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume no H_2O ; - MW = 60 (lot 951303)
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 945500

PROCEDURE:

The procedure for uranophane synthesis is based on Cesbron et al., 1993. Reagents will be mixed in the stoichiometric ratio $\text{Ca}:\text{U}:\text{Si} = 2:1:1$. Although Cesbron et al. do not give information on the amount of solid and H_2O used, they state that good crystallinity is obtained if pH is less than 5.

- Place 35 g Ca-acetate, 42 g U-acetate, and 6 g silicic acid in a 2000 ml teflon liner and place liner in reaction vessel.
- Transfer approximately 1500 ml of ultrapure H_2O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the H_2O by boiling on a hotplate.

*use further
separated by
distillation*

- d) Transfer approximately 1300 g of the degassed H₂O to the teflon liner and place in pressure vessel.
- e) Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller.
- f) Degas the pressure vessel once it reaches >140°C by opening the air intake valve attached to the pressure gauge assembly.
- g) Close intake valve after degassing and allow reagents to react for about 10 days.

10/16/95 JP

Vessels for 'monophase synthesis 9' +
'monophase synthesis 10' were cooled
& opened

pH of solutions
effluent 9 = 3.93
effluent 10 = 4.03

Solids were filtered thru a 10 µm
mesh.

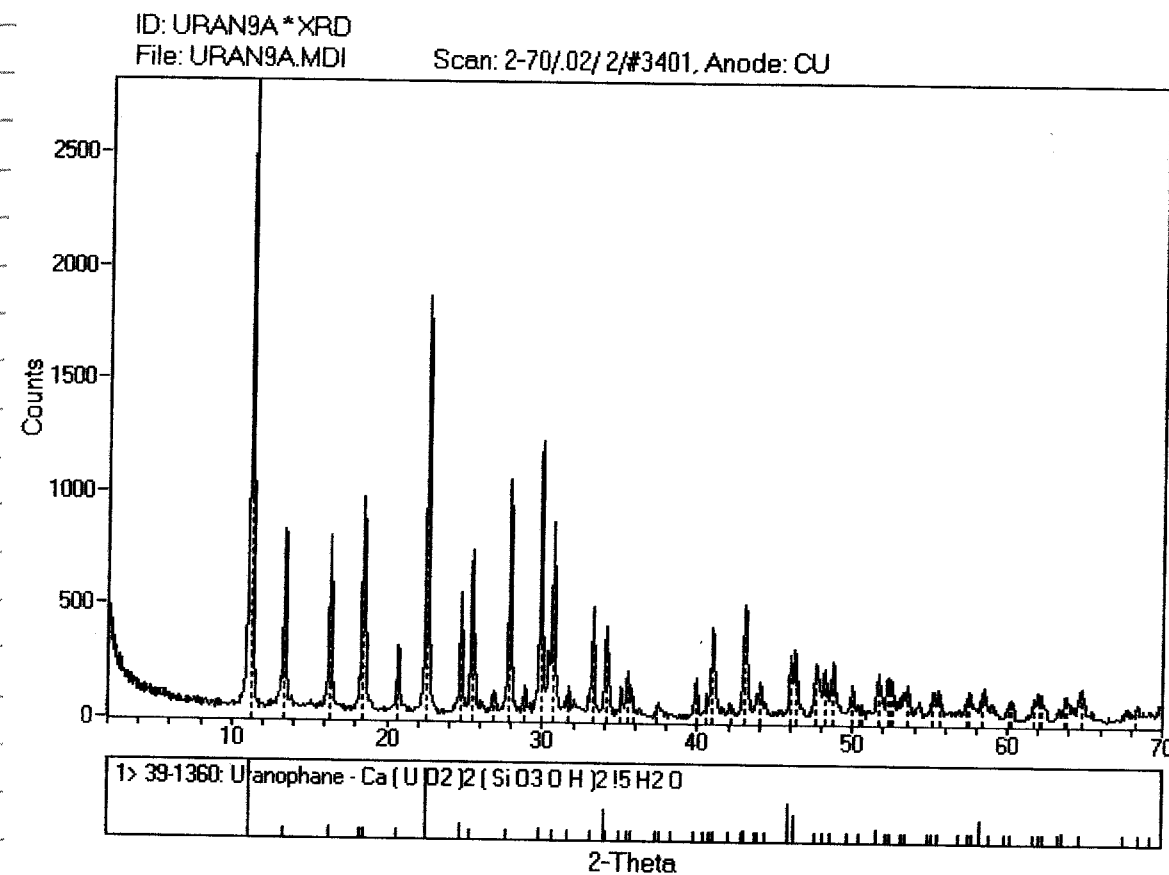
Filtered solids were dried + placed in
plastic vials. Vials were labeled
URAN*SYN*9 and URAN*SYN*10.

Portion of solids were analyzed by XRD
using procedure in p. 6.

10/23/95 gp

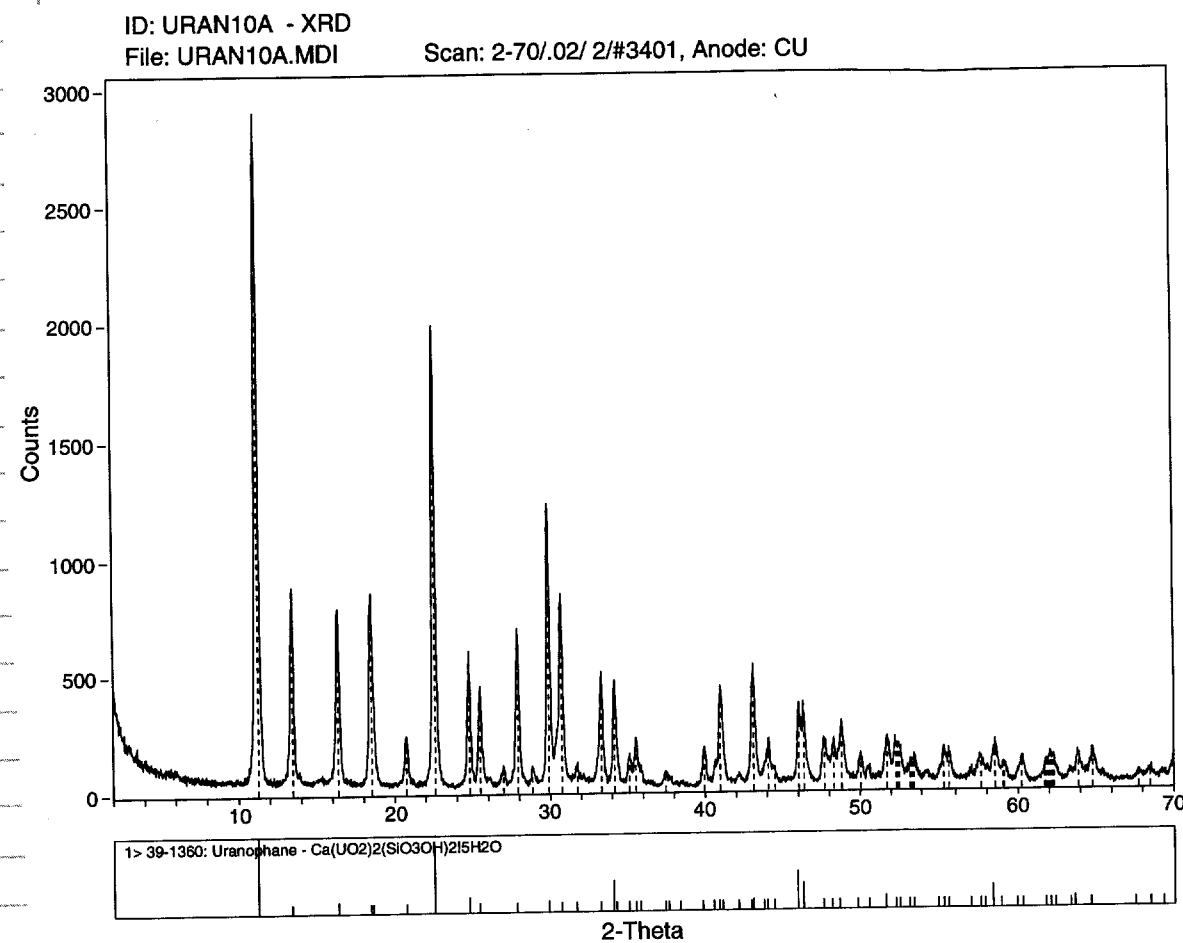
Results of XRD analyses.

URAN*SYN*9



Uranophane is only phase present

URAN*SYN*10



Uranophane is only phase present.

10/26/95

Preparation of U and Si standards for chemical analysis.

Obj - prepare standards with U contents similar to monophane for QA purposes.

Reagents - Uanyl acetate (lot 337419/1 1094)
sodium metasilicate (lot 942853A).

Supplies + Materials + Equipment

- Teflon beakers
- nanopure water
- mettler PA 4600 balance
- weighing paper.
- drying oven (Blue M).

Procedure

- ① Three standards will be prepared.
Standard 1 will contain 50% by weight Uanyl acetate + 50% Na-silicate.
Standard 2 - 80% U-acetate + 20% Na-silicate
and Standard 3 - 90% Uanyl acetate + 10% Na-silicate.
- ② To teflon beaker 2.5g Uanyl acetate and 2.5 Na-silicate were added for standard 1.
4g U-Acetyl + 1g Na-silicate were mixed for standard 2.
4.5g U-Acetyl + 0.5g Na-silicate were mixed for standard 3.

③ About 100 ml of water were added to each beaker.

④ The solid was mixed in the solution to achieve homogeneity.

⑤ The 3 beakers were placed in a drying oven at 60°C and the solution was allowed to evaporate.

⑥ The solid was then scrapped from the beaker + placed in plastic vials.

Vials were labeled as follows.

U-Ac/Na-silicate 50/50

U-Ac/Na-silicate 80/20

U-Ac/Na-silicate 90/10. of 10/26/95

JP 10/31/95 Sodyite synthesis experiments were initiated using the following procedure.

The experiments will be referred to as 'sodyite synthesis 6' and 'sodyite synthesis 7'.

SODDYITE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 2

DATE WRITTEN: July 3, 1995
DATE REVISED: Oct 31, 1995

OBJECTIVE: to synthesize sodyite $[(\text{UO}_2)_2\text{SiO}_4 \cdot 2\text{H}_2\text{O}]$ for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH = 1, 2, 4)
ultrapure water
glassware and plasticware as needed
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 337419/1 1094)
Silicic acid $[\text{SiO}_2 \cdot n\text{H}_2\text{O}]$; MW depends on H_2O content; assume no H_2O ; MW = 60 (lot 951303)
9 M HCl - diluted from lot 945500

PROCEDURE:

Reagents for sodyite synthesis will be mixed in the stoichiometric ratio U:Si = 1:2.

- Place 42 g U-acetate and 12 g silicic acid (coarse fraction separated by density settling in a 2000 ml teflon liner.
- Transfer approximately 1500 g of ultrapure H_2O to a 2000 ml erlenmeyer flask and degas the H_2O by boiling on a hotplate.
- Transfer approximately 1300 g of the degassed H_2O to the teflon liner and place in pressure vessel.
- Raise and set the temperature of the solution in the reaction vessel to 150°C using the temperature controller.
- Degas the pressure vessel once it reaches about >140°C by opening the air intake valve attached to the pressure gauge assembly.
- Close intake valve after degassing and allow the reagents to react for about 10 days.

JP 11/1/95 Chemical analyses of monophane

Obj - determine chemistry of superheated monophane + standards.

Method - ICP analysis in Div 01.

Procedure:

- Portions of the following samples + standards were placed in glass vials + labeled as follows:

Label	Sample
20P	URAN*SYN*9
20Q	URAN*SYN*10
20R	U/AC/Na-silicate 80/20
20S	URAN*SYN*9
20T	U/AC/Na-silicate 50/50
20U	U/AC/Na-silicate 90/10
20V	URAN*SYN*10
20W	URAN*SYN*9

- Samples were delivered to Div 01 for analysis of major + trace elements

4/13/95 JF

Vessels for 'soddyite synthesis 6' and
'soddyite synthesis 7' were cooked
and opened

pH of solutions
Experiment 6 = 3.33
Experiment 7 = 3.31

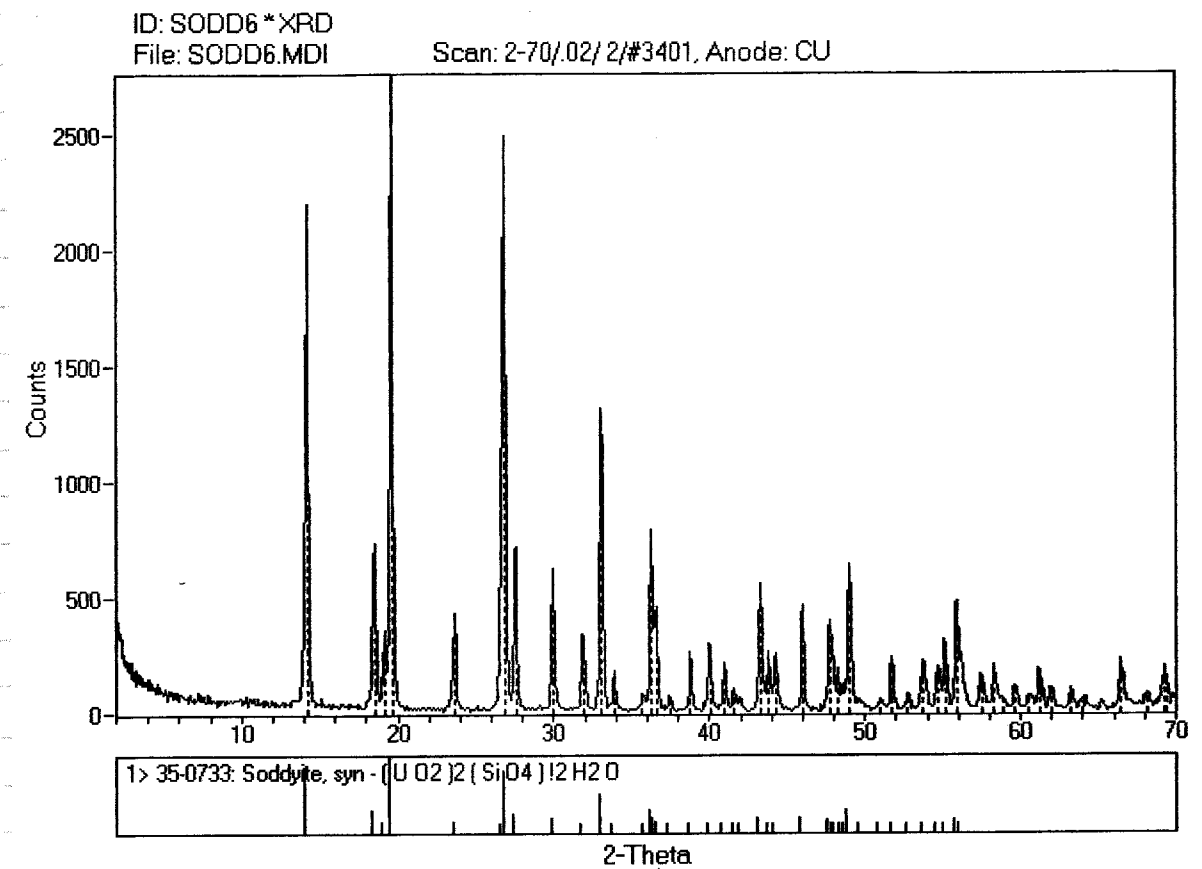
Solids were filtered thru a 10 μ m
Nylon mesh.

Filtered solids were dried + placed in
plastic vials. Vials were labeled
SODD*SYN*6 + SODD*SYN*7.

Portions of solids were analyzed by
XRD using procedure in p6.

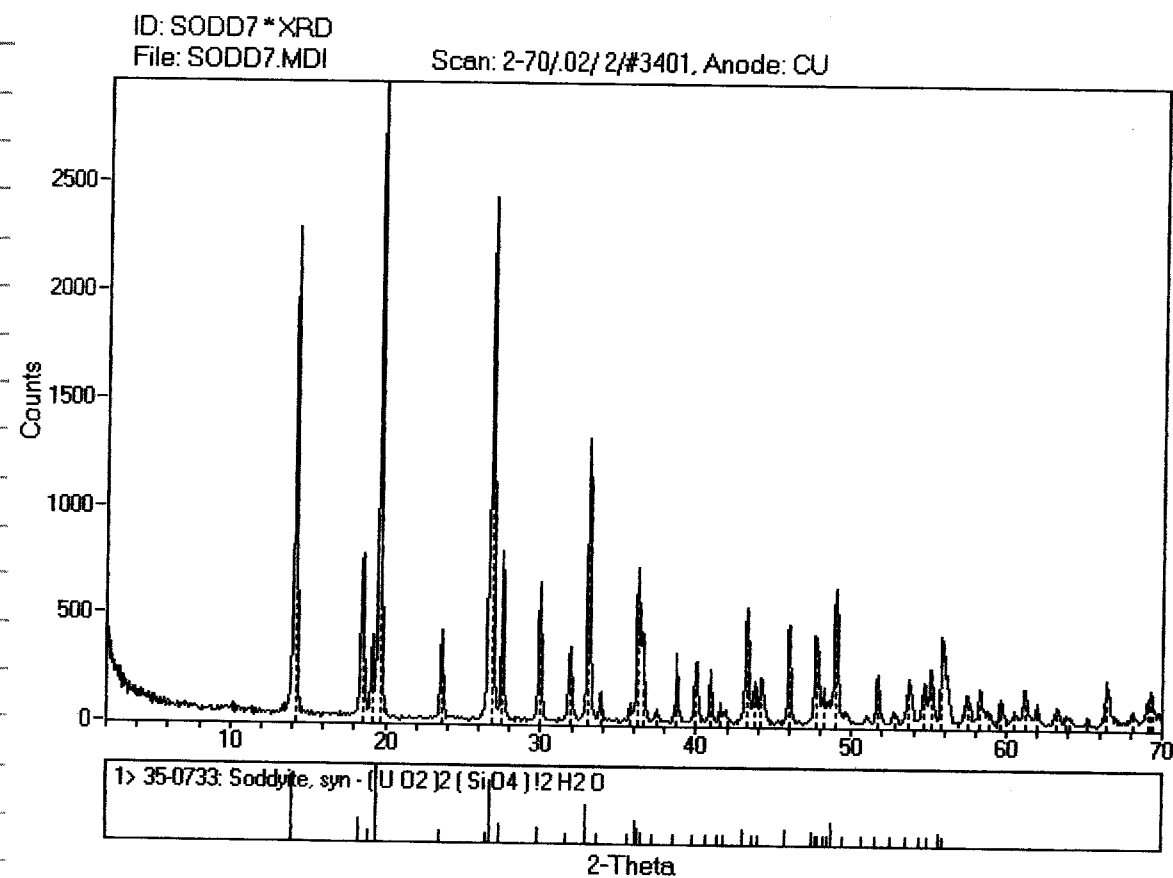
4/15/95

Results of XRD analysis of SODD*SYN*6



Soddyite is only phase seen.

Results of XRD analysis of SODD# SYN# 7



Soddyite is only phase seen.

11/17/95 JG

0300
JG

Two additional soddyite synthesis experiments were initiated using procedure on p 118.

The experiments will be referred to as 'soddyite syn# 8' and 'soddyite syn# 9'

11/29/95 JP

Vessels for 'soddyite synthesis 8' and 'soddyite synthesis 9' were cooled + opened.

pH of solution:

Experiment 8 = 2.98

" 9 = 3.05

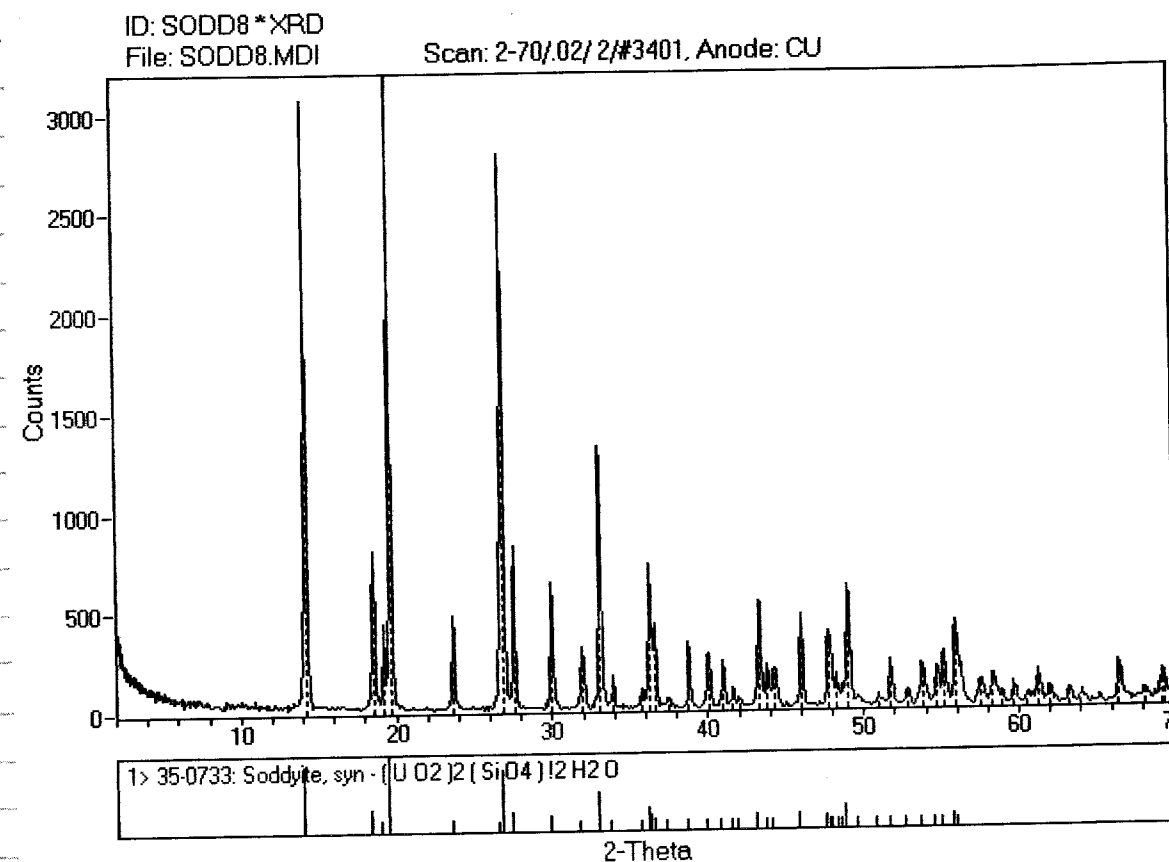
Solids were filtered thru a 10 μ m nylon mesh.

Filtered solids were dried + placed in plastic vials. Vials were labeled SODD8*SYN*8 + SODD8*SYN*9

Portions of solids were analyzed by XRD using procedure in p. 6.

11/30/95 JP

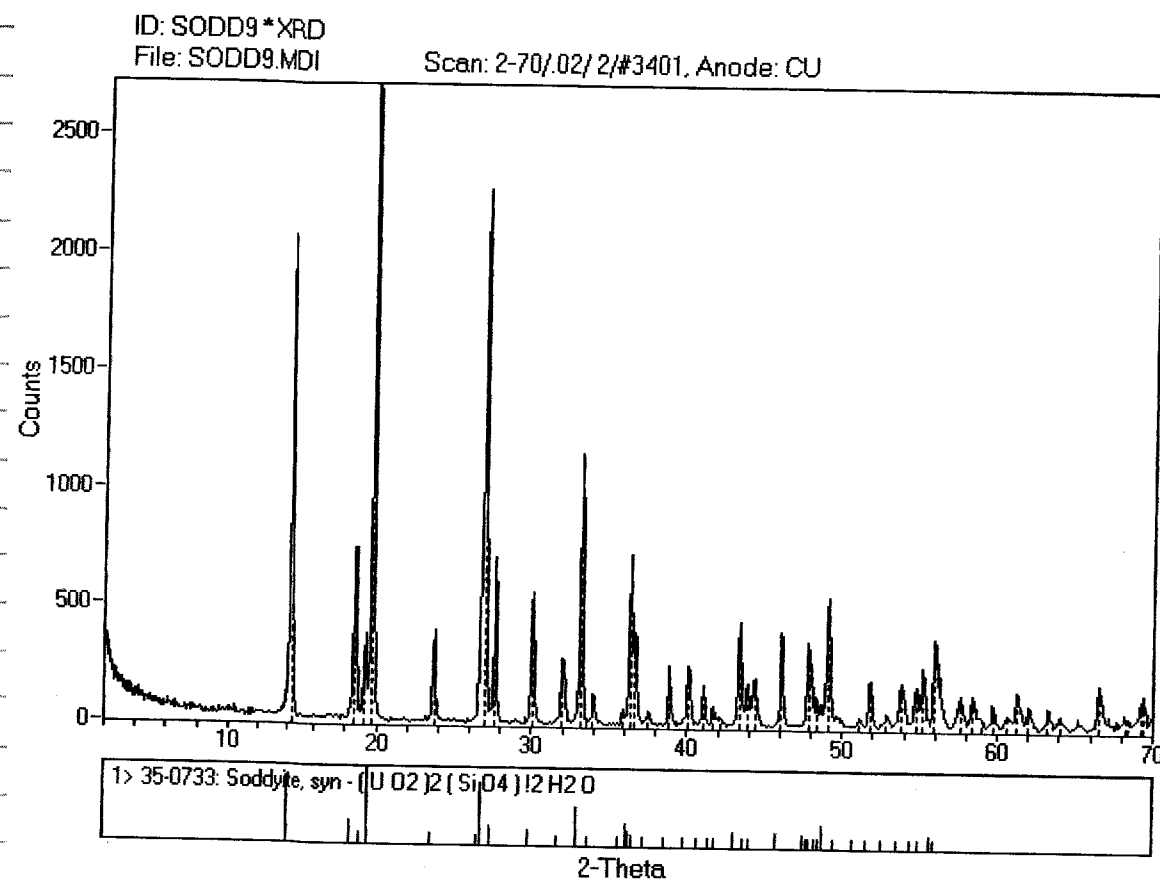
Results of XRD analysis of SODD8*SYN*8



Soddyite is only phase seen.

11/30/95pp

Results of XRD analysis of SODD*SYN*9



Soddyite is only phase seen.

2/5/95

SEM/EDS analyses of synthesized
monophase & SoddyiteObj - determine morphology & chemistry
of synthesized U mineralsMethod - Scanning electron microscope/
energy dispersive x-ray analysis.

Procedure:

1) Portions of URAW*SYN*9,
URAW*SYN*10, SODD*SYN*7, and
SODD*SYN*9 were placed on
aluminum stubs w/ carbon
tape.

2) Samples were delivered to Div 01
for coating.

3) Samples will be analyzed by
SEM/EDS using the AMRAY
SEM and Tracor Northern EDS
systems in Div 01.

4) Results will be entered in the
~~notebook~~ ^{2/18/95} when analyses are
completed. Results are entered
in 3 ring binder entitled "New-
Field Project Photos".

2/6/95 JP

Results of chemical analysis of synthesized
maopline by Div 01 are shown below.
(see p119).

SOUTHWEST RESEARCH INSTITUTE

6220 CULEBRA ROAD • POST OFFICE DRAWER 28510 • SAN ANTONIO, TEXAS, USA 78228-0510 • (210) 684-5111 • TELEX 244846

December 5, 1995

Mr. James Prikyrl
Division 20
Southwest Research Institute

Subject: Project 20-5704-152
Metals by ICP and Total Uranium Analyses
Work Order Number: 7939
Samples Received: November 01, 1995

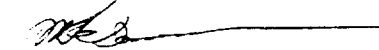
Dear Mr. Prikyrl:

Enclosed please find the sample results and QC sample results for the above referenced project. If you should have any questions, please contact me at 210-522-2169.

Sincerely,

Jo Ann Boyd
Jo Ann Boyd
QAU Group Leader

TECHNICAL APPROVAL:



Mike Dammann
Inorganic Group Leader

JAB:



SAN ANTONIO, TEXAS
HOUSTON, TEXAS • DETROIT, MICHIGAN • WASHINGTON, DC

Division 20
Southwest Research Insitute
SDG: 20P
SwRI Work Order Number: 7939
December 5, 1995
page 1

SwRI Case Narrative
Division 20 SDG:20P

1. Fourteen (8) Samples for ICP and U-Total Analysis:

SwRI ID	Customer ID
62301	20P
62302	20Q
62303	20R
62304	20S
62305	20T
62306	20U
62307	20V
62308	20W

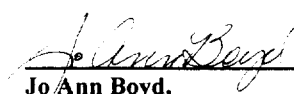
2. Samples were received at SwRI on November 01, 1995 for a 14 day turnaround time from Validated Time of Sample Receipt (VTSR).

METHODOLOGY OF ANALYSIS:

The major oxides were analyzed using a "whole rock" procedure that entails a complete dissolution of the samples followed by ICAP analysis for the major species. The chemical dissolution of samples was done by an HCL/HNO₃/HF acid digestion in a teflon bomb. The digestion products were brought to a known volume using a 4% Boric acid solution to complex any excess HF and were heated again to dissolve any fluoride salts. The final solution was analyzed for major constituents utilizing a simultaneous ICAP with an internal standard. Calibration was performed using NBS certified rock standards, NIST SRM278 and NIST SRM688. The loss on ignition was done by gravimetry at 1000 C. The result for the major species were reported as their oxides.

Division 20
Southwest Research Insitute
SDG: 20P
SwRI Work Order Number: 7939
December 5, 1995
page 2

"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Director or his designee, as verified by the following signature."

 12/5/95
Jo Ann Boyd, Date
Group Leader
Quality Assurance Unit,
Division 01

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 11/01/95

Lab System ID: 62301

Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20P	Si as SiO ₂	15.658%
20P	Al as Al ₂ O ₃	<0.001%
	Fe as Fe ₂ O ₃	0.009%
	Mg as MgO	0.155%
	Ca as CaO	6.593%
	Na as Na ₂ O	<0.001%
	K as K ₂ O	0.001%
	Ti as TiO ₂	0.054%
	Mn as MnO	0.001%
	Ba as BaO	0.002%
	Cr as Cr ₂ O ₃	<0.001%
	Cu as CuO	0.004%
	Li as Li ₂ O	0.001%
	Mo as MoO ₃	0.007%
	Ni as NiO	<0.001%
	P as P ₂ O ₅	0.152%
	Pb as PbO	<0.001%
	Sr as SrO	<0.001%
	V as V ₂ O ₅	<0.001%
	Zn as ZnO	0.001%
	Co as CoO	0.002%
	U as U ₃ O ₈	58.974%
	LOI(Loss at 1000°C)	12.510%
	TOTAL	94.124%

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 11/01/95

Lab System ID: 62302

Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20Q	Si as SiO ₂	15.945 %
URAN*SYN*ID	Al as Al ₂ O ₃	<0.001 %
	Fe as Fe ₂ O ₃	0.011 %
	Mg as MgO	0.190 %
	Ca as CaO	6.562 %
	Na as Na ₂ O	<0.001 %
	K as K ₂ O	<0.001 %
	Ti as TiO ₂	0.052 %
	Mn as MnO	0.001 %
	Ba as BaO	<0.001 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.004 %
	Li as Li ₂ O	0.001 %
	Mo as MoO ₃	0.007 %
	Ni as NiO	0.003 %
	P as P ₂ O ₅	0.167 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	0.001 %
	Co as CoO	<0.001 %
	U as U ₃ O ₈	58.478 %
	LOI(Loss at 1000°C)	12.750 %
	TOTAL	94.172 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 11/01/95

Lab System ID: 62303

Project No.: 20-5704-152

Sample ID	Analysis	Sample Result	Duplicate Result
20R	Si as SiO ₂	5.683 %	5.774 %
Wacetate 30%	Al as Al ₂ O ₃	<0.001 %	<0.001 %
Wn silicate 20%	Fe as Fe ₂ O ₃	0.003 %	0.001 %
	Mg as MgO	0.165 %	0.113 %
	Ca as CaO	<0.001 %	<0.001 %
	Na as Na ₂ O	4.731 %	4.982 %
	K as K ₂ O	<0.001 %	<0.001 %
	Ti as TiO ₂	0.045 %	0.045 %
	Mn as MnO	0.001 %	0.001 %
	Ba as BaO	0.012 %	<0.001 %
	Cr as Cr ₂ O ₃	<0.001 %	<0.001 %
	Cu as CuO	0.004 %	0.004 %
	Li as Li ₂ O	0.001 %	<0.001 %
	Mo as MoO ₃	0.007 %	0.008 %
	Ni as NiO	<0.001 %	0.002 %
	P as P ₂ O ₅	0.015 %	0.140 %
	Pb as PbO	<0.001 %	<0.001 %
	Sr as SrO	<0.001 %	<0.001 %
	V as V ₂ O ₅	<0.001 %	<0.001 %
	Zn as ZnO	<0.001 %	0.003 %
	Co as CoO	0.003 %	<0.001 %
	U as U ₃ O ₈	54.361 %	55.865 %
	LOI	25.840 %	25.840 %
	TOTAL	91.005 %	92.779 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20
Lab Code: SWRI Date Received: 11/01/95
Lab System ID: 62304 Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20S	Si as SiO ₂	15.455 %
WEANTSYN 9	Al as Al ₂ O ₃	<0.001 %
	Fe as Fe ₂ O ₃	0.006 %
	Mg as MgO	0.265 %
	Ca as CaO	6.436 %
	Na as Na ₂ O	<0.001 %
	K as K ₂ O	0.003 %
	Ti as TiO ₂	0.052 %
	Mn as MnO	0.001 %
	Ba as BaO	0.001 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.004 %
	Li as Li ₂ O	0.001 %
	Mo as MoO ₃	0.001 %
	Ni as NiO	0.001 %
	P as P ₂ O ₅	0.173 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	<0.001 %
	Co as CoO	<0.001 %
	U as U ₃ O ₈	59.099 %
	LOI(Loss at 1000°C)	13.020 %
	TOTAL	94.517 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 11/01/95

Lab System ID: 62305

Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20T	Si as SiO ₂	15.092 %
58% vacante	Al as Al ₂ O ₃	<0.001 %
50% Na silicate	Fe as Fe ₂ O ₃	0.002 %
	Mg as MgO	0.181 %
	Ca as CaO	0.001 %
	Na as Na ₂ O	14.481 %
	K as K ₂ O	0.002 %
	Ti as TiO ₂	0.032 %
	Mn as MnO	<0.001 %
	Ba as BaO	<0.001 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.003 %
	Li as Li ₂ O	<0.001 %
	Mo as MoO ₃	0.005 %
	Ni as NiO	0.005 %
	P as P ₂ O ₅	0.122 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	0.003 %
	Co as CoO	0.005 %
	U as U ₃ O ₈	39.324 %
	LOI(Loss at 1000°C)	25.410 %
	TOTAL	94.667 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20
Lab Code: SWRI Date Received: 11/01/95
Lab System ID: 62306 Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20U	Si as SiO ₂	3.240 %
90% Uantale	Al as Al ₂ O ₃	<0.001 %
10% Na silicate	Fe as Fe ₂ O ₃	0.002 %
	Mg as MgO	0.255 %
	Ca as CaO	0.008 %
	Na as Na ₂ O	2.630 %
	K as K ₂ O	<0.001 %
	Ti as TiO ₂	0.053 %
	Mn as MnO	0.001 %
	Ba as BaO	0.002 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.005 %
	Li as Li ₂ O	0.001 %
	Mo as MoO ₃	0.014 %
	Ni as NiO	0.001 %
	P as P ₂ O ₅	0.193 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	0.001 %
	Co as CoO	<0.001 %
	U as U ₃ O ₈	65.497 %
	LOI(Loss at 1000°C)	24.740 %
	TOTAL	96.642 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20
Lab Code: SWRI Date Received: 11/01/95
Lab System ID: 62307 Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20V	Si as SiO ₂	16.732 %
URAN*SYN*10	Al as Al ₂ O ₃	<0.001 %
	Fe as Fe ₂ O ₃	0.003 %
	Mg as MgO	0.231 %
	Ca as CaO	6.087 %
	Na as Na ₂ O	<0.001 %
	K as K ₂ O	0.002 %
	Ti as TiO ₂	0.051 %
	Mn as MnO	0.001 %
	Ba as BaO	0.005 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.003 %
	Li as Li ₂ O	0.001 %
	Mo as MoO ₃	0.012 %
	Ni as NiO	0.001 %
	P as P ₂ O ₅	0.170 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	0.001 %
	Co as CoO	0.001 %
	U as U ₃ O ₈	57.416 %
	LOI(Loss at 1000°C)	12.740 %
	TOTAL	93.457 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 11/01/95

Lab System ID: 62308

Project No.: 20-5704-152

Sample ID	Analysis	Sample Result
20W	Si as SiO ₂	16.163 %
URANUSYN*9	Al as Al ₂ O ₃	<0.001 %
	Fe as Fe ₂ O ₃	0.008 %
	Mg as MgO	0.173 %
	Ca as CaO	6.225 %
	Na as Na ₂ O	<0.001 %
	K as K ₂ O	<0.001 %
	Ti as TiO ₂	0.052 %
	Mn as MnO	0.001 %
	Ba as BaO	<0.001 %
	Cr as Cr ₂ O ₃	<0.001 %
	Cu as CuO	0.004 %
	Li as Li ₂ O	0.001 %
	Mo as MoO ₃	0.010 %
	Ni as NiO	0.002 %
	P as P ₂ O ₅	0.195 %
	Pb as PbO	<0.001 %
	Sr as SrO	<0.001 %
	V as V ₂ O ₅	<0.001 %
	Zn as ZnO	<0.001 %
	Co as CoO	0.005 %
	U as U ₃ O ₈	58.151 %
	LOI(Loss at 1000°C)	13.070 %
	TOTAL	94.059 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20
Lab Code: SWRI Date Received: 11/01/95
Lab System ID: NA Project No.: 20-5704-152

Sample ID	Analysis	Beginng of Run	End of Run
278	Si as SiO ₂	74.222 %	72.831 %
	Al as Al ₂ O ₃	13.831 %	14.880 %
	Fe as Fe ₂ O ₃	2.106 %	2.120 %
	Mg as MgO	0.230 %	0.252 %
	Ca as CaO	0.983 %	0.952 %
	Na as Na ₂ O	4.684 %	5.274 %
	K as K ₂ O	4.044 %	4.546 %
	Ti as TiO ₂	0.241 %	0.247 %
	Mn as MnO	0.050 %	0.050 %
	Ba as BaO	0.101 %	0.105 %
	Cr as Cr ₂ O ₃	0.001 %	0.001 %
	Cu as CuO	0.001 %	0.001 %
	Li as Li ₂ O	0.010 %	0.011 %
	Mo as MoO ₃	0.001 %	0.001 %
	Ni as NiO	0.002 %	<0.001 %
	P as P ₂ O ₅	0.072 %	0.057 %
	Pb as PbO	0.005 %	0.003 %
	Sr as SrO	0.004 %	0.004 %
	V as V ₂ O ₅	0.002 %	0.002 %
	Zn as ZnO	0.007 %	0.007 %
	Co as CoO	0.001 %	0.002 %
	U as U ₃ O ₈	<0.001 %	<0.001 %
	LOI(Loss at 1000°C)	<0.001 %	<0.001 %
	TOTAL	100.596 %	101.347 %

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20
Lab Code: SWRI Date Received: 11/01/95
Lab System ID: NA Project No.: 20-5704-152

Sample ID	Analysis	Beginning of Run	End of Run
688	Si as SiO ₂	50.783 %	49.463 %
	Al as Al ₂ O ₃	17.425 %	19.243 %
	Fe as Fe ₂ O ₃	10.389 %	10.304 %
	Mg as MgO	8.389 %	8.924 %
	Ca as CaO	12.270 %	11.455 %
	Na as Na ₂ O	2.010 %	2.442 %
	K as K ₂ O	0.177 %	0.215 %
	Ti as TiO ₂	1.172 %	1.194 %
	Mn as MnO	0.160 %	0.158 %
	Ba as BaO	0.018 %	0.019 %
	Cr as Cr ₂ O ₃	0.046 %	0.044 %
	Cu as CuO	0.013 %	0.014 %
	Li as Li ₂ O	0.001 %	0.001 %
	Mo as MoO ₃	<0.001 %	<0.001 %
	Ni as NiO	0.020 %	0.017 %
	P as P ₂ O ₅	0.198 %	0.184 %
	Pb as PbO	0.006 %	<0.001 %
	Sr as SrO	0.010 %	0.011 %
	V as V ₂ O ₅	0.052 %	0.052 %
	Zn as ZnO	0.010 %	0.009 %
	Co as CoO	0.010 %	0.014 %
	U as U ₃ O ₈	<0.001 %	<0.001 %
	LOI(Loss at 1000°C)	<0.001 %	<0.001 %
	TOTAL	103.159 %	103.762 %

2/22/96 gp

Work on the previously synthesized many silicate minerals will now be conducted under the "Container Life and Source Term KTI"

The charge no. for these experiments is 20-5708-073.

2/22/96 gp

Dissolution of synthesized monophane

Obj. dissolve synthesized monophane (URAN+SYN*9) and (URAN+SYN*10) in dilute HCl so that major element chemical analysis can be done in the CNWRA labs.

Reagent & supplies:

- 0.1 M HCl
- Volumetric flasks (100 ml + 1L)
- PP bottles (125 ml)
- Mettler analytical balance (Model AE240)
- Teflon stir bars
- Corning stirrer / hot plate

Procedure:

- 1) Make a 0.1 M HCl solution by diluting 8.2 ml conc HCl (lot 956110) in 500 ml ultrapure H₂O in a 1L volumetric flask. Make up to mark with H₂O.
- 2) Placed the following amounts of URAN+SYN*9 and URAN+SYN*10 in 100 ml volumetric flasks.

$$\begin{aligned} \text{URAN} + \text{SYN} * 9 &= .5056 \text{ g} \\ \text{URAN} + \text{SYN} * 10 &= .5027 \text{ g} \end{aligned}$$

The 0.1M HCl solution was added to the flasks up to the mark. Teflon stir bars were added to the flasks and flasks were placed on stirrers to dissolve the monophane.

3.) After the monophane had dissolved the solutions were transferred to 125 ml PP bottles.

Bottles were labeled as follows:

0.5g URAW*SYN*9 dissolved in 0.1M HCl
and
0.5g URAW*SYN*10 dissolved in 0.1M HCl.

02/23/96 JP

AA analysis of synthesized monophane

Obj - determine concentration of Si, Ca, and Na in synthesized monophanes.

Method - atomic absorption spectrometry

Supplies + Reagents.

- 50 ml volumetric flasks (100 ml and 1 L)
- 1000 ppm Ca standard, Fisher certified, lot 950440-24
- 1000 ppm Si standard, Fisher certified, lot 951765-12
- 1000 ppm Na standard, Fisher certified, lot 951760-24
- 1.5 ml HNO_3 in 1 L H_2O solution - matrix solution
- Perkin Elmer AA Model 3100
- Ca hollow cathode lamp (Fisher)
- Si " " " "
- Na " " " "
- ~0.2 M HCl solution - prepared by dissolving 35 g HCl (lot 915032A) in 500 ml H_2O .

Procedure.

Ca analysis -

- 1) Make a series of Ca standards by diluting the 1000 ppm certified AA standard solution with the 1.5 ml HNO_3 in 1000 ml H_2O matrix solution. Used 100 ml volumetric flasks to make these standards. Concentrations of standards made were 5, 10, 12, and 20 ppm Ca.
- 2) Placed 5 ml of URAN*SYN*9 in 0.1 M HCl and URAN*SYN*10 in 0.1 M HCl in 100 ml volumetric flasks and made up to mark using the matrix solution. These are the solutions that will be analyzed for Ca.
- 3) Perkin-Elmer AA was setup using conditions outlined in 'Perkin Elmer Analytical Methods for AA Spectrometry' for Ca. ($\lambda = 422.7 \text{ nm}$, 0.7 nm slit, air-acetylene flame). Lanthanum solution was added to both standards + samples to reduce interferences.
- 4) Perkin Elmer AA was calibrated using standards and then samples were analyzed.

5) Results of Ca analysis are given below:

URAN*SYN*9 - duplicate samples

Conc. reading	Dilution Factor	Ca (ppm)
12.46	20x	249.2
12.51	20x	250.3

URAN*SYN*10 - duplicate samples

Conc. reading	Dilution Factor	Ca (ppm)
12.64	20x	252.8
12.57	20x	251.4

Si analysis -

- 1) Make a series of Si standards by diluting the 1000 ppm certified AA standard with the 1.5 ml HNO_3 in 1000 ml H_2O matrix solution. Concentrations of standards were 25, 50, and 100 ppm Si. $\text{PP}_{21/22/16}$
- 2) Placed 10 ml of URAN*SYN*9 in 0.1 M HCl and URAN*SYN*10 in 0.1 M HCl in 50 ml volumetric flasks + made up to mark using matrix solution.
- 3) Perkin Elmer AA was setup using conditions outlined in 'Perkin Elmer Analytical Methods for AA Spectrometry'.

for Si. ($\lambda = 251.6 \text{ nm}$, 0.2 nm slit, acetylene-nitrogen oxide flame).

4) Perkin Elmer AA was calibrated using standards + then samples were read.

5) Results of Si analysis are given below:

URAN*SYN*9 duplicate samples

Conc. ready	Dilution factor	Si ppm
72.3	5X	361.5
70.65	5X	353.25

URAN*SYN*10 duplicate samples

Conc. ready	Dilution factor	Si ppm
76.35	5X	381.75
73.9	5X	369.5

Na analysis

1) Made a series of Na standards by diluting the 1000 ppm Na certified AA standard with matrix solution.
Concentration of standards was 0.5 and 1.0 ppm Na.

2) Placed ^{10 g} 5 ml of URAN*SYN*9 in 0.1 M HCl + 0.1 M URAN*SYN*10 in 0.1 M HCl in 100 ml volumetric flask + made up to mark with water

3) Perkin Elmer AA was setup using conditions outlined in 'Perkin Elmer Analytical Methods for AA Spectrometry' for Na ($\lambda = 589 \text{ nm}$, 0.2 nm slit, air-acetylene flame).

4) Perkin Elmer AA was calibrated using standard + then samples were measured.

5) Results of Na analysis are given below:

URAN*SYN*9 duplicate samples

Conc. ready	Dilution factor	Na ppm
.19	5X	.95
.19	5X	.95

URAN*SYN*10 duplicate samples

Conc. ready	Dilution factor	Na ppm
.04	5X	.20
.04	5X	.20

3/6/96 JP

Gravimetric analysis of synthesized monophase
to determine H_2O content.

Obj: To determine water content of synthesized
monophase.

Method: gravimetry

Equipment + supplies

Muffle Furnace; Fisher Isotemp Model 134A

Quartz crucible

Mettler analytical balance; Model AE240

URAN*SYN*9

URAN*SYN*10

Procedure

- 1) Place weighed quantity of synthesized
monophase in quartz crucibles.
Weights were as follows:

Wt of quartz crucible	= 12.7124 g
Wt of crucible + URAN*SYN*9	= 12.9644 g
Wt of URAN*SYN*9	= .2520 g

Wt of quartz crucible	= 11.6722 g
Wt of crucible + URAN*SYN*10	= 11.9226 g
Wt of URAN*SYN*10	= .2504 g

- 2) Place crucibles containing synthesized monophase
in muffle furnace set at $900^\circ C$ for
2 hours.

- 3) Cool crucibles + reweigh.

Wt of crucible + URAN*SYN*9 = 12.9322 g

Wt of water loss in URAN*SYN*9 =

$$12.9644 - 12.9322 = .0322 \text{ g}$$

$$\% \text{ loss} = .0322 \div .252 \times 100 = 12.78 \%$$

Wt of crucible + URAN*SYN*10 = 11.8897

Wt of water loss in URAN*SYN*10 =

$$11.9226 - 11.8897 = .0329 \text{ g}$$

$$\% \text{ loss} = .0329 \div .2504 \times 100 = 13.14 \%$$

3/21/96 JP

Polarographic analysis of synthetic uranophane

Obj - determine conc of U in synthetic uranophane

Method - polarographic analysis

Supplies, Equipment, + reagents:

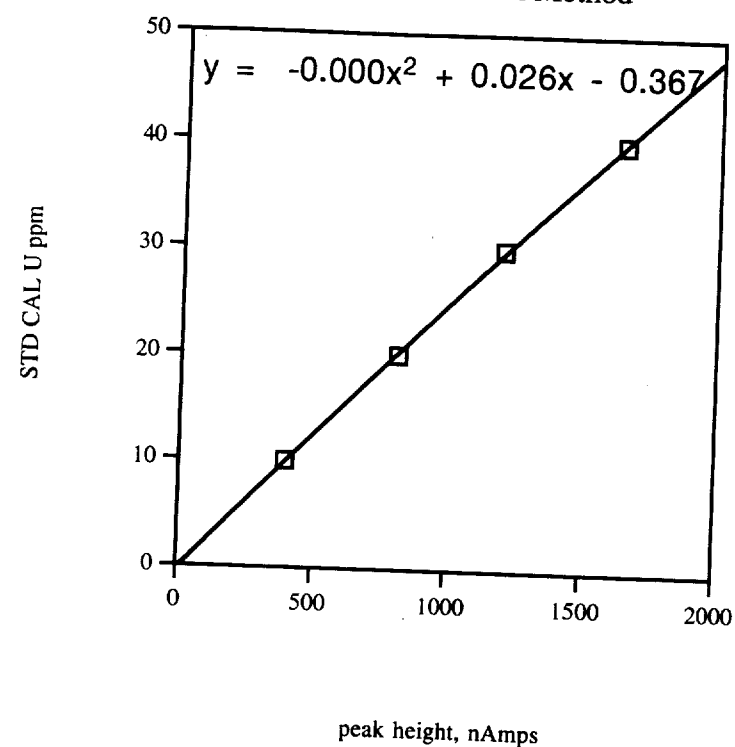
- volumetric Flasks
- 0.1 M HCl (lot 956110)
- Model EG+G 384B Polarographic Analyzer
- EG+G Model 303A Mercury Drop Electrode
- 1000 ppm U standard - made by dissolving 1.0545 g uranyl nitrate (lot 8440 R.H.R.) in 500 ml of 0.1 M HCl.

Procedure

A. Standard Calibration metal

- 1) Made a series of U standards by diluting the 1000 ppm U standard. 0.1 M HCl was used to make these standards. Concentration of standards made were 10, 20, 30, + 40 ppm U.
- 2) Placed 1 ml of URAN+SYN+9 in 0.1 M HCl and URAN+SYN+10 in 0.1 M HCl in 100 ml volumetric flasks + make up to mark with 0.1 M HCl solution. These are solutions that will be analyzed for U.
- 3) Polarographic analyzer + mercury drop electrode were setup for square wave voltammetry using conditions outlined in Model 384B Polarographic Analyzer Instruction Manual (pp U-34 to U-38). Conditions are listed in 3-ring binder entitled "Polarographic Analysis of Synthetic Uranophane".
- 4) Standards were analyzed and a calibration curve was built. Data for these calibrations is kept in 3-ring binder entitled "Polarographic Analysis of Synthetic Uranophane".

Calibration curve is shown below.
Standard Calibration Method



5). Samples were then analyzed. Data + plots are kept in 3 ring binder.

U contents in samples are shown below

	peak height nAmps	U conc
URAN*SYN*9	985.5	2525.6
URAN*SYN*10	941.5	2411.2

$$U = 0.026(\text{peak height, nAmps}) - 0.367 \times 100$$

B. Standard Addition's Method

1) 10 ml of URAN*SYN*9 in 0.1 M HCl was analyzed. p

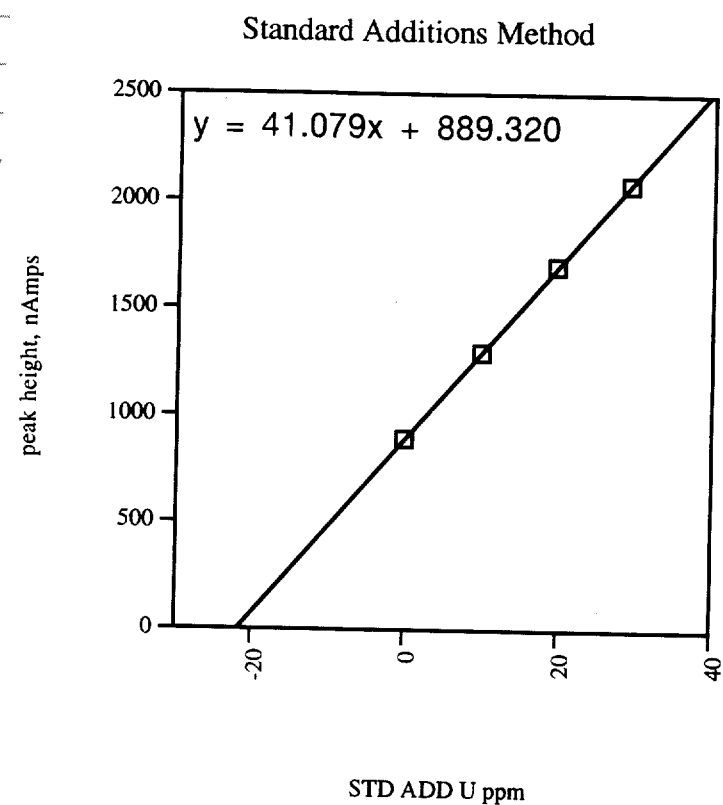
2) 100 μ l of 1000 ppm U in 0.1 M HCl was added to the sample and the sample was reanalyzed.

3) Another 100 μ l (total 200 μ l) of 1000 ppm U was added and the sample was reanalyzed.

4) Another 100 μ l (total 300 μ l) of 1000 ppm U was added + the sample reanalyzed.

5) Data for all three analyses are kept in the 3 ring binder "Polarographic analysis of synthesized uranylplac".

6) Data for U was plotted + the U concentration calculated (see next page).



URAN * SYN * 9 peak height = 888.5 nAmps

$$U = \frac{889.32}{41.079} \times 100 = 2164.9$$

3/26/96

Dissolution of synthesized morphine

Followed procedure outlined on p 147-148

The following amounts of morphine were dissolved in 100 ml 0.1 M HCl

URAN * SYN * 9 = .1010 g
 URAN * SYN * 10 = .1017 g
 URAN * SYN * 3 = .1034 g

Samples were labeled as below:

0.1g URAN * SYN * 9 in 0.1 M HCl
 0.1g URAN * SYN * 10 in 0.1 M HCl
 0.1g URAN * SYN * 3 in 0.1 M HCl

3/27/96

AA analysis of synthesized uranophane

Ca + Si contents of following solutions were determined:

0.1g URAN*SYN*9 in 0.1M HCl

0.1g URAN*SYN*10 in 0.1M HCl

0.1g URAN*SYN*3 in 0.1M HCl

Procedure for AA analysis on p 149-152 was followed:

Results of Ca analysis:

URAN*SYN*9 (duplicate samples)

Conc reading	Dilution factor	Ca (ppm)
2.44	20x	48.8
2.44	20x	48.8

URAN*SYN*10

Conc reading	dil factor	Ca (ppm)
2.47	20x	49.4
2.49	20x	49.8

URAN*SYN*3

Conc reading	dil factor	Ca (ppm)
2.53	20x	50.6
2.55	20x	51.0

Results of Si analysis

URAN*SYN*9 (duplicate samples)

Conc reading	Dil factor	Si ppm
73.8 74.2	1x	74.2
74.0	1x	74.0

URAN*SYN*10

Conc reading	Dil factor	Si ppm
74.3	1x	74.3
74.1	1x	74.1

URAN*SYN*3

Conc reading	Dil factor	Si ppm
75.8	1x	75.8
75.3	1x	75.3

3/27/96 JF

Polarographic analysis of synthesized uranophane

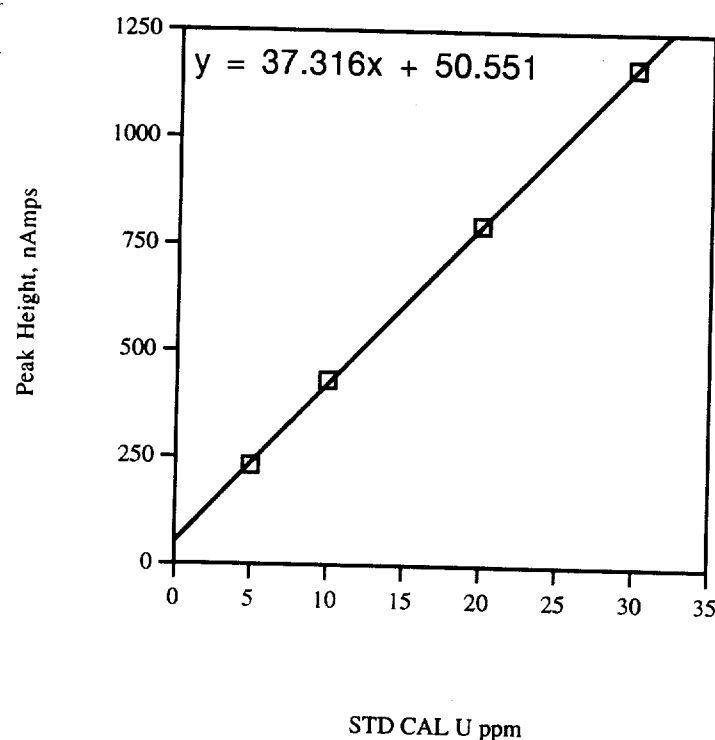
U content of Solvay solution was determined using procedure on p 156-158

0.1g URANTSYN*9 in 0.1 M HCl

0.1g URANTSYN*10 in 0.1 M HCl

0.1g URANTSYN*3 in 0.1 M HCl

Standard calibration Method Results

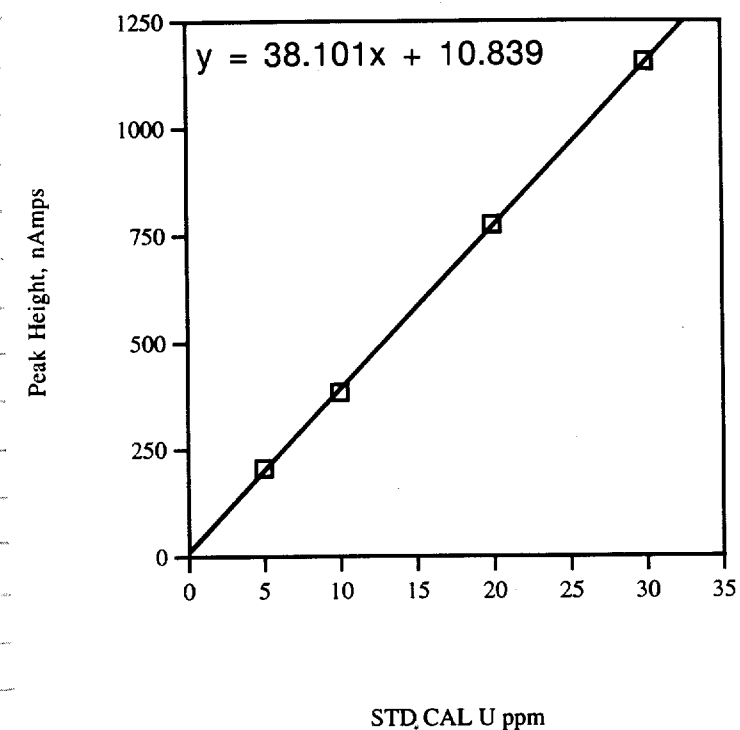


Samples were diluted by 20x.

Results

	peak height, nAmps	U conc. ppm
URANTSYN*9	1021	2600 ^{90 3/27/96} 2600 + 520.2
URANTSYN*10	982.5	2497 499.4
URANTSYN*3	875.5	^{90 3/27/96} 2244 2244 442.2

Duplicate standard calibration

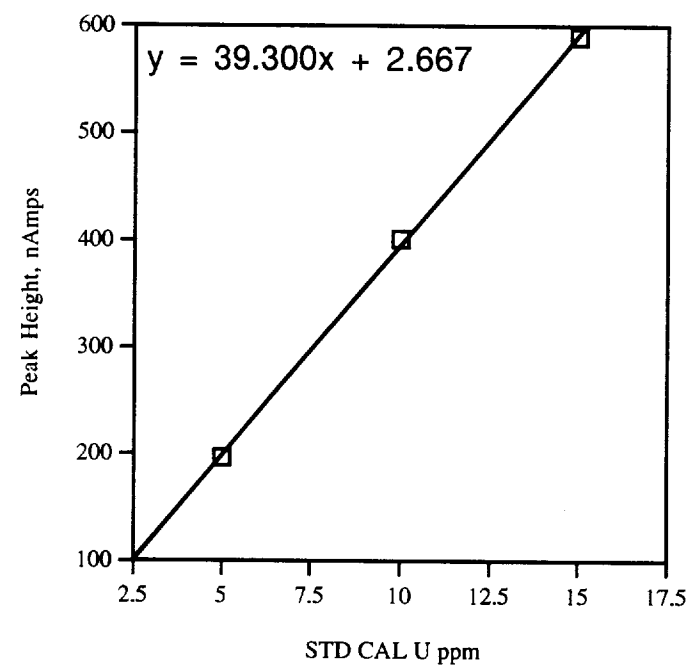


Results.

	peak height, nAmps	U conc ppm
URANTSYN*9	1000.5	2597 ^{90 3/27/96} 2597 519.4
URANTSYN*10	945	2452 490.4
URANTSYN*3	996.5	2587 ^{90 3/27/96} 2587 517.4

3/29/96 gp

Another standard calibrate for U but
samples are diluted by 50x.



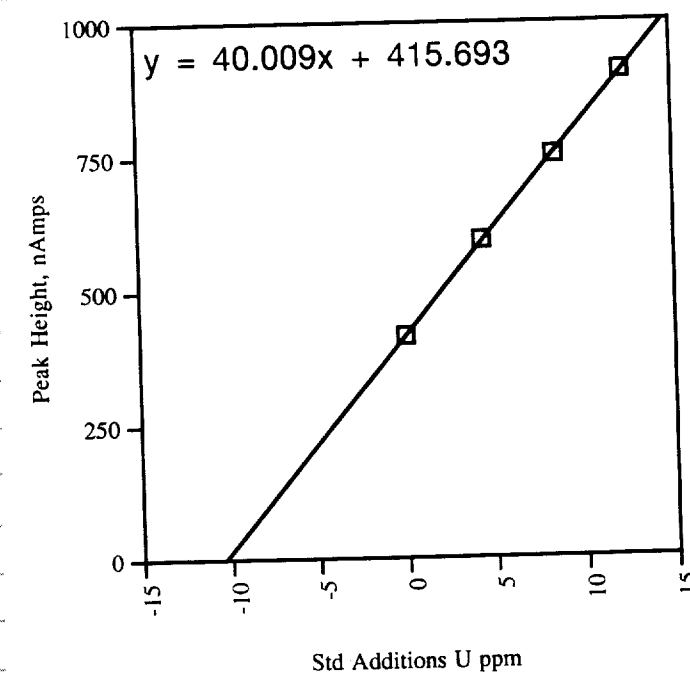
Results

	peak height, nAmps	U conc ppm
URAN*SYN*9	391.95	495.3
URAN*SYN*10	421	532.2
URAN*SYN*3	456	576.8

Standard Addition Method

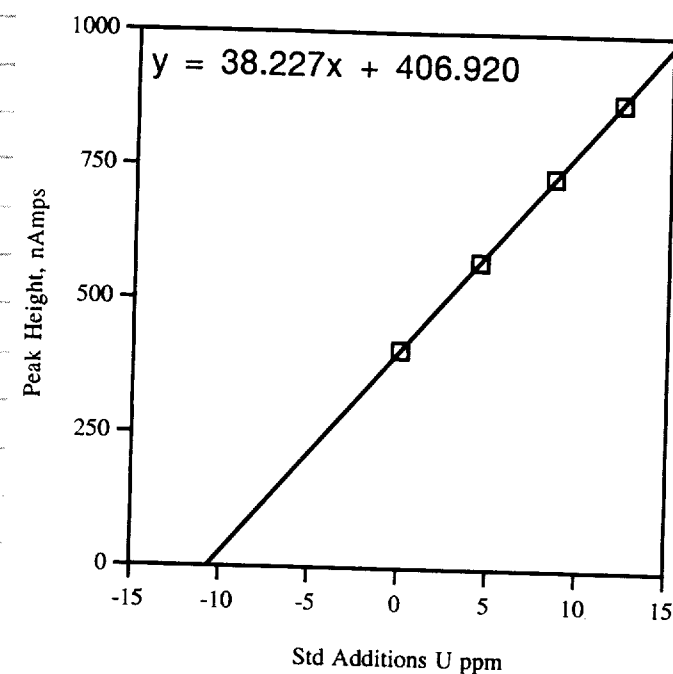
In these analyses ²³⁵U
solution was added to an initial sample
volume of 10 ml

URAN*SYN*10

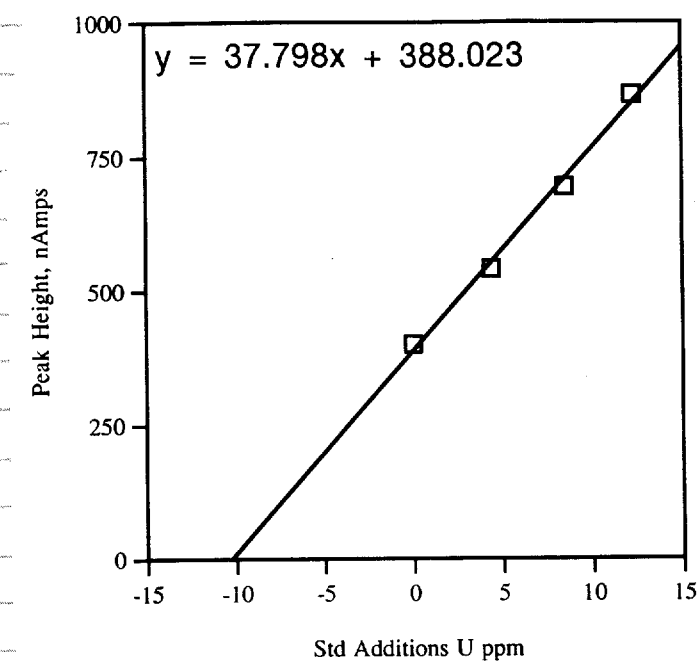


U = 519.5 ppm

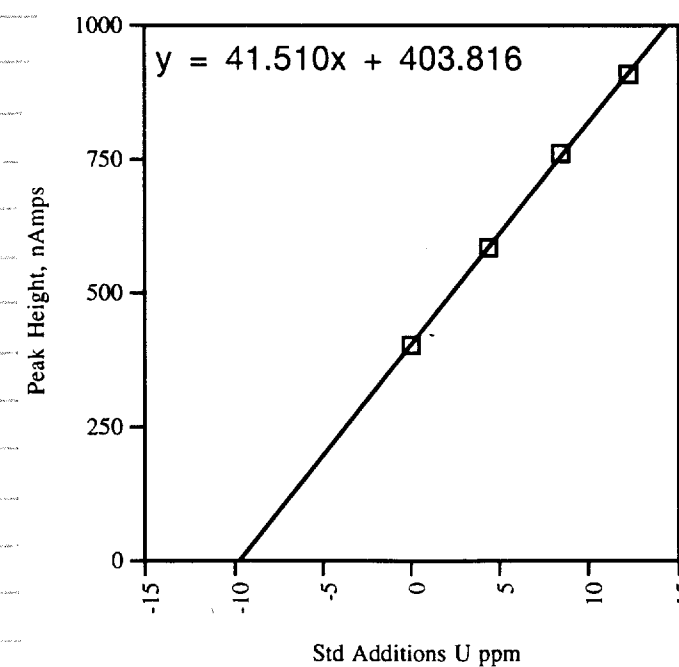
URAN*SYN*10

 $\mu = 532.2 \text{ ppm}$

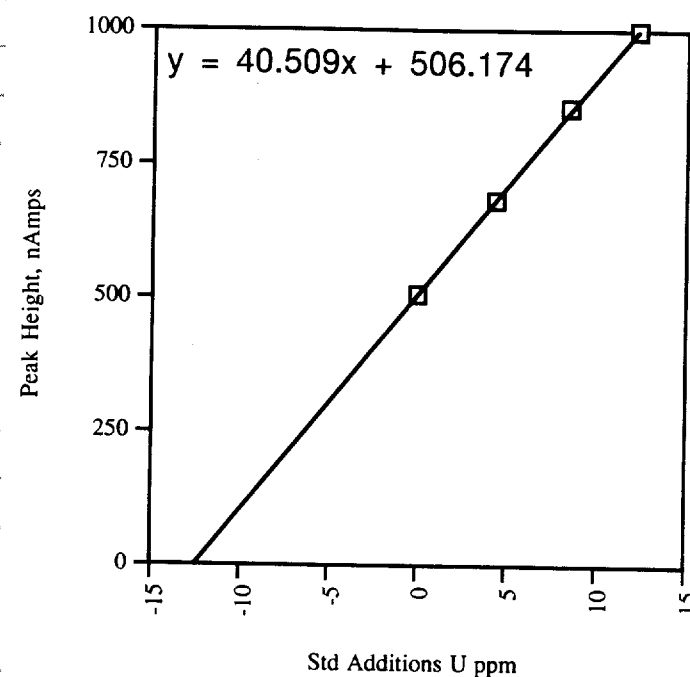
URAN*SYN*9

 $\mu = 513.3$

URAN*SYN*9

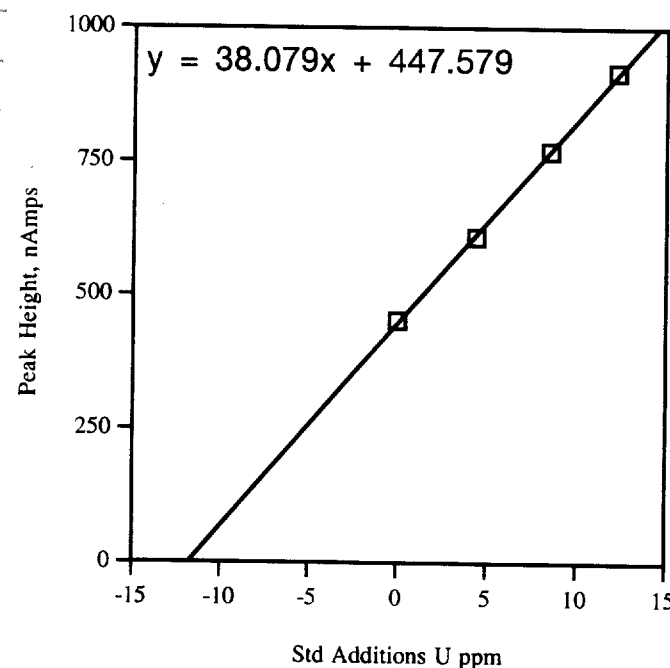
 $\mu = 486.4$

URAN*SYN*3



$$U = 624.8$$

URAN*SYN*3



$$U = 587.7$$

4/9/96 gp

Gravimetric analysis of silicic acid used
in wrong silicate synthesis

Obj - determine water content of silicic acid

Method - gravimetry

Equipment + supplies:

Muffle Furnace; Fisher Isotemp 184A

Quartz crucible

Mettler analytical balance; Model AE240

Silicic acid - lot 951303

Procedure:

- 1) place weighed amount of silicic acid
in a quartz crucible.
Weight is as follows.

$$\begin{aligned} \text{Wt of crucible} &= 12.6466 \text{ g} \\ \text{Wt of crucible + silicic acid} &= 13.2388 \text{ g} \\ \text{Wt of silicic acid} &= .5922 \text{ g} \end{aligned}$$

- 2) Place crucible in furnace set at 900°C
for 2 hr.

3) (cool crucible & reweigh)

wt of crucible + silicic acid = 13.0715 g

wt of H₂O loss = 13.2388 - 13.0715 = .1673 g

% loss = $.1673 \div .5922 \times 100 = 28.25\%$

4/15/96 JP

A new uranophane synthesis experiment was initiated using the following procedure. This procedure was modified from previous experiments to ensure that all the silicic acid reagent used is dissolved in the volume of solution used.

This experiment will be referred to as URANOPHANE SYNTHESIS.

URANOPHANE SYNTHESIS

WRITTEN BY: J.D. Prikryl
REVISION NO.: 4

DATE WRITTEN: March 7, 1995
DATE REVISED: April 15, 1996

OBJECTIVE: to synthesize uranophane [Ca(UO₂)₂(SiO₃OH)₂·2.5H₂O] for use in thermodynamic experiments of uranyl silicate minerals for the near-field geochemistry project.

EQUIPMENT: Parr stainless steel reaction vessels (Model 4913EB; 2000 ml capacity)
Teflon liners for reaction vessels
Parr Model 4841 or 4843 temperature controllers
Type J thermocouples (teflon coated)
ORION pH/mV/ISE/°C meter
Combination pH electrode
Automatic temperature compensator probe
Analytical balance (Mettler PM 4600)
Welsh vacuum pump (Model 8910)
Hot plate (Thermolyne Type 13100)

SUPPLIES: pH buffers (pH= 1,2,4)
ultrapure water
glassware and plasticware as needed
1000 ml erlenmeyer flasks with stoppers
Fittings, valves, and tubing as necessary

REAGENTS: Uranyl acetate dihydrate [UO₂(CH₃COO)₂·2H₂O; MW=424.15 (analysis no. 337419/1 1094)
Silicic acid [SiO₂·nH₂O]; MW depends on H₂O content; assume no H₂O; - MW = 60 (lot 951303)
Calcium acetate monohydrate [Ca(CH₃COO)₂·H₂O; MW=176.19 (lot 944231)
9 M HCl - diluted from lot 945500

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 H₂O used, they state that good crystallinity is obtained if pH is less than 5.

- Place 10.2 g Ca-acetate, 12.25 g U-acetate, and 1.75 g silicic acid in a 2000 ml teflon liner and place liner in reaction vessel.
- Transfer approximately 1500 ml of ultrapure H₂O to a 2000 ml erlenmeyer flask and lower pH of the fluid to 1.0 by dropwise addition of 9M HCl.
- Degas the H₂O by boiling on a hotplate.
- Transfer approximately 1350 g of the degassed H₂O to the teflon liner and place in pressure vessel.
- Raise and set the temperature of the solution in the reaction vessel to 150C using the temperature controller.
- Degas the pressure vessel once it reaches >110C by opening the air intake valve attached to the pressure gauge assembly.
- Close intake valve after degassing and allow reagents to react for about 10 days.

4/22/96 gp

Vessel for "URAN#SYN#11" was cooled and opened.

Solids were filtered through a 10 µm nylon mesh.

Filtered solids were dried + placed in a plastic vial labeled URAN#SYN#11.

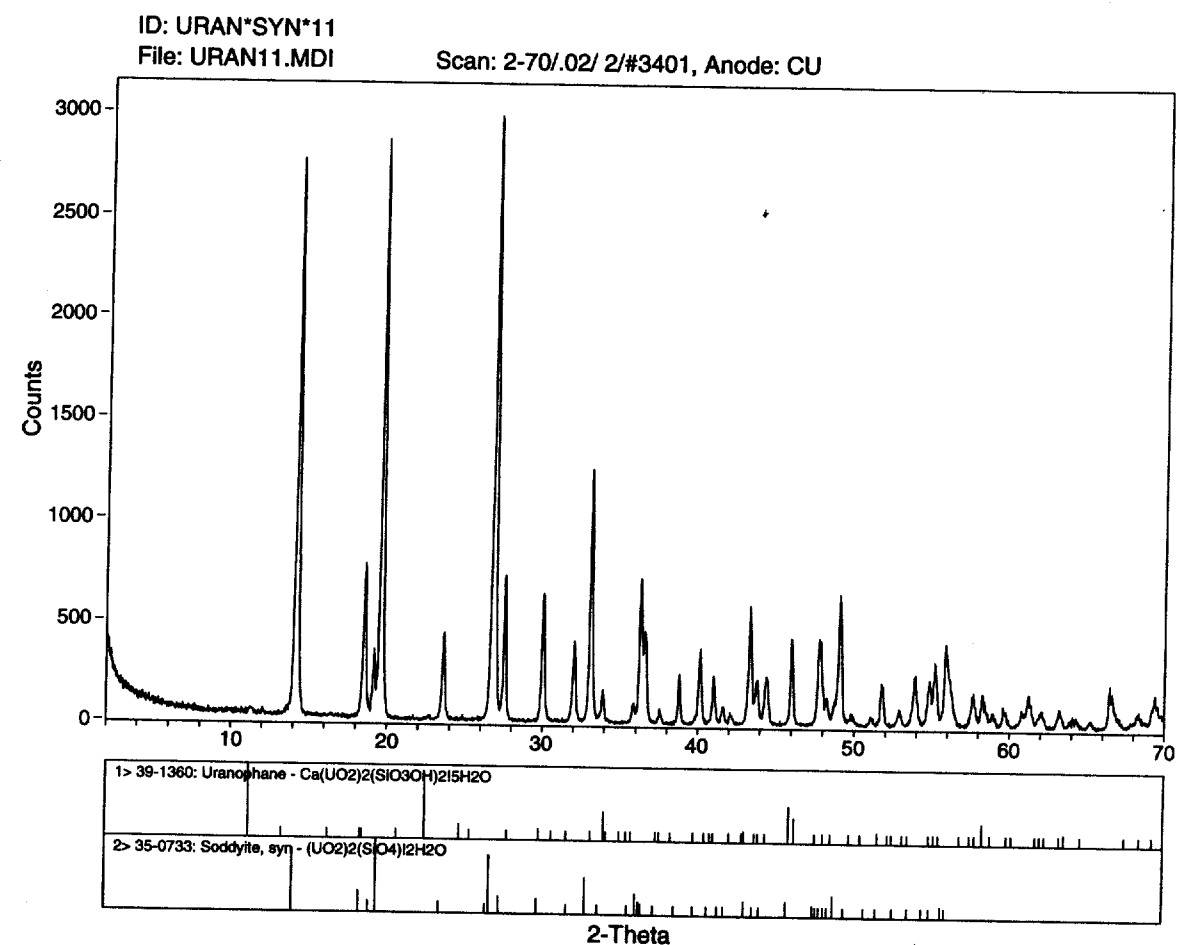
Portion of solid was analyzed by XRD using procedure in pl.

removed note
next page

gp
4/24/96

4/24/96

Results of XRD analysis



Results indicate that material synthesized was soddyite rather than uranophane. This was probably due to undercalcination with Ca.

Since the material synthesized was soddyite the sample was renamed SODD*SYN*11.

4/25/96

Dissolution of synthesized soddyite

Followed procedure outlined for uranophane dissolution on p 147-148.

The following amount of SODD*SYN*11 was dissolved in 100 ml 1M HCl.

Wt of SODD*SYN*11 = .1065 g

Sample was placed in PP bottle + labeled

0.1 g SODD*SYN*11 in 1M HCl

#/25/96

Prepared aqueous samples of dissolved synthetic uranophane & soddyite for analysis by ICP for U, Si, Ca, & Na in Div 01.

Previously dissolved uranophane & soddyite samples were diluted for these analyses. All samples were prepared in a 0.1 M HCl matrix.

Standards were also prepared using AA standards. These samples were also prepared in a 0.1 M HCl matrix.

Not NO for standards used are shown below:

Si	1000 ppm AA standard	-	951765A-12
Ca	" " " "	-	950440-24
Na	" " " "	-	ORION

The 1000 ppm U standard prepared on p 156 was used for U.

Samples and their dilution factors and standards were labeled as below:

Sample/standard	Solution used or prepared	Dilution Factor
20A1	0.1g URAN + SYN*9 in 0.1 M HCl	10x
20A2	U-50ppm, Ca-10ppm, Si-15ppm, Na-0ppm	
20A3	0.5g URAN + SYN*10 in 0.1 M HCl	20x
20A4	0.1g SODD + SYN*11 in 0.1 M HCl	10x
20A5	0.1g URAN + SYN*10 in 0.1 M HCl	10x
20A6	U-90ppm, Ca-8ppm, Si-7ppm, Na-1ppm	
20A7	0.5g URAN + SYN*9 in 0.1 M HCl	20x
20A8	0.1g URAN + SYN*3 in 0.1 M HCl	10x
20A9	0.1g URAN SODD + SYN*8 in 0.1 M HCl	10x
20AA	U-140ppm, Ca-5ppm, Si-10ppm, Na-3ppm	
20AB	0.1g URAN + SYN*9 in 0.1 M HCl	10x
20AC	0.1g URAN + SYN*3 in 0.1 M HCl	10x

4/25/96

Two new uranophane synthesis experiments were initiated. These two experiments will be referred to as 'uranophane synthesis 12' and 'uranophane synthesis 13'.

The experimental procedure on p 173-174 was followed but the amounts of reagents were modified.

In 'uranophane synthesis 12' 17.5g calcium acetate, 1.80g silicic acid, and 21.0g methyl acetate were used in 650ml H₂O.

In 'uranophane synthesis 13' 17.5g calcium acetate, 1.80g silicic acid, and 21.0g methyl acetate were used.

5/8/96

Results of chemical analyses of synthesized uranophane by Div 01 were received (see p. 179 for description of samples).

Results are summarized below:

SOUTHWEST RESEARCH INSTITUTE SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 04/25/96

Matrix: Liquid

Date Analyzed: 05/08/96

Project No.: 20-5708-573

Sample ID	Lab System ID	Calcium Result (ug/mL)	Sodium Result (ug/mL)	Silicon Result (ug/mL)	Uranium Result (ug/mL)
20 A1	70230	4.76	<0.1	7.18	56.1
Duplicate	70230	4.55	<0.1	7.13	55.4
RPD	70230	4.5%	0.0%	0.7%	1.3%
Spike	70230	14.1	10.3	17.7	109
Spike Added	70230	10.0	10.0	10.0	50.0
Recovery	70230	93.4%	103.0%	105.2%	105.8%
20 A2	70231	10	23.3	15.6	60
20 A3	70232	11.3	<0.1	17.5	135
20 A4	70233	<0.1	<0.1	4.07	74
20 A5	70234	4.72	<0.1	7.02	55.5
20 A6	70235	8.03	12.1	7.2	87.7
20 A7	70236	11.5	<0.1	17.2	135
20 A8	70237	4.8	<0.1	7.21	57.1
20 A9	70238	<0.1	<0.1	4.06	72.8
20 AA	70239	4.94	18.6	10.3	137
20 AB	70240	4.57	<0.1	6.91	54.1
20 AC	70241	4.65	<0.1	7.2	55.4

Detection Limit: 0.1 ug/mL 0.1 ug/mL 0.1 ug/mL 0.1 ug/mL

5/9/96 gp

Gravimetric analysis of synthesized monophase and soddyite

Obj - determine water content of synthesized monophase + soddyite

Method - gravimetry

Equipment -

Muffle Furnace

Ohaus crucible

Mettler analytical balance Model AE240

Procedure

- 1) Place weighed amounts of synthesized monophase or soddyite in quartz crucible. Weights are as follows.

$$\begin{aligned} \text{Wt of crucible} &= 12.5063 \text{ g} \\ \text{Wt of crucible} + \text{SODD} * \text{SYN} * 7 &= 12.7701 \text{ g} \\ \text{Wt of SODD} * \text{SYN} * 7 &= .2638 \text{ g} \end{aligned}$$

$$\begin{aligned} \text{Wt of crucible} &= 12.2499 \text{ g} \\ \text{Wt of crucible} + \text{SODD} * \text{SYN} * 8 &= 12.5102 \text{ g} \\ \text{Wt of SODD} * \text{SYN} * 8 &= .2603 \text{ g} \end{aligned}$$

$$\begin{aligned} \text{Wt of crucible} &= 12.7085 \text{ g} \\ \text{Wt of crucible} + \text{SODD} * \text{SYN} * 11 &= 12.9632 \text{ g} \\ \text{Wt of SODD} * \text{SYN} * 11 &= .2547 \text{ g} \end{aligned}$$

$$\begin{aligned} \text{Wt of crucible} &= 12.2511 \text{ g} \\ \text{Wt of crucible} + \text{URAN} * \text{SYN} * 3 &= 12.5054 \text{ g} \\ \text{Wt of URAN} * \text{SYN} * 3 &= .2543 \text{ g} \end{aligned}$$

- 2) Place crucible in muffle furnace at 500°C for 2 hrs.

- 3) Cool crucible + weigh

$$\text{Wt of crucible} + \text{SODD} * \text{SYN} * 7 = 12.7515$$

$$\text{Wt of H}_2\text{O loss} = 12.7701 - 12.7515 = .0186 \text{ g}$$

$$\% \text{ loss} = .0186 \div .2638 \times 100 = 7.05\%$$

$$\text{Wt of crucible} + \text{SODD} * \text{SYN} * 8 = 12.4901$$

$$\text{Wt of H}_2\text{O loss} = 12.5102 - 12.4901 = .0201$$

$$\% \text{ loss} = .0201 \div .2603 \times 100 = 7.72\%$$

over

$$\text{Wt of crucible + SODD \& SYN \# 11} = 12.9443$$

$$\text{Wt of H}_2\text{O loss} = 12.9632 - 12.9443 = .0189\text{g}$$

$$\% \text{ loss} = .0189 \div .2547 \times 100 = 7.42\%$$

$$\text{Wt of crucible + KRAV \& SYN \# 3} = 12.4723\text{g}$$

$$\text{Wt of H}_2\text{O loss} = 12.5054 - 12.4723 = .0331$$

$$\% \text{ loss} = .0331 \div .2543 \times 100 = 13.02\%$$

5/10/96

Vessels for 'monophase synthesis 12' and 'monophase synthesis 13' were cooled and opened.

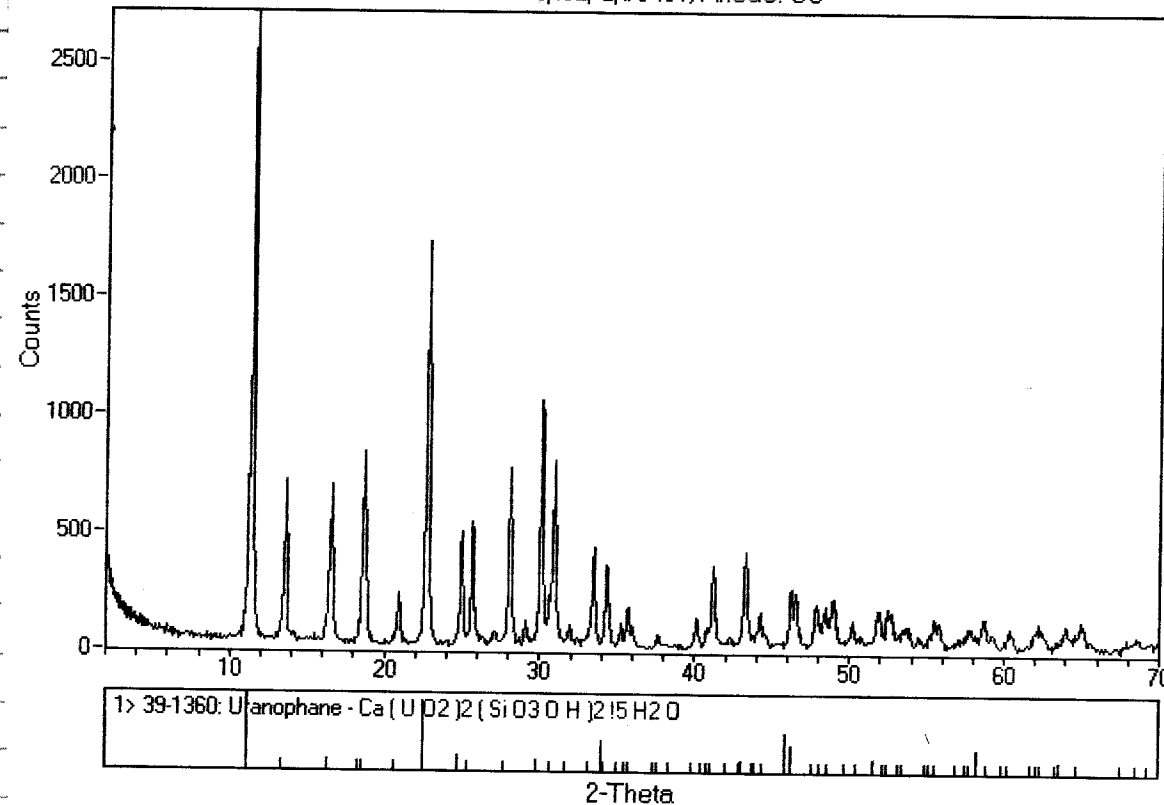
A portion of the solid in each vessel was removed, dried, and analyzed by ^{13}C NMR analysis using procedure on p6.

5/20/96

Results of XRD analysis

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

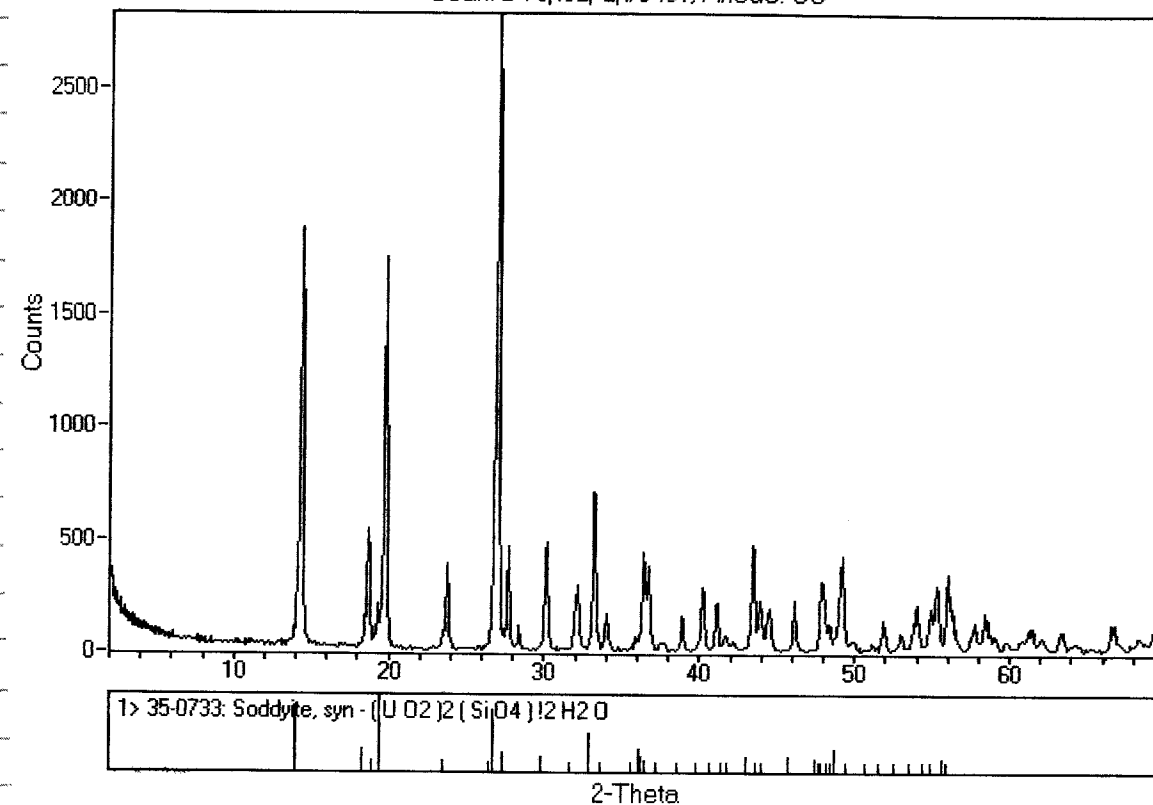
Scan: 2-70/.02/2/#3401, Anode: CU



Results indicate synthesized material is uranophane. The solid will be filtered thru a 10 μm mesh filter and labeled 'URAN*SYN*12'.

ID: URAN*SYN*13
File: URAN13.MDI

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



Results indicate that synthesized solid is soddyite. This solid will be filtered and labeled 'SODD*SYN*13'.

5/20/94

Dissolution of synthesized monophase
sample U₂AN + SiW + 12

Followed procedure in 147-148

The following amounts of U₂AN + SiW + 12 of 5/20/94
were dissolved in 100 ml 0.1 M HCl

.1003 g

.1004 g

.1999 g

Samples were placed in 125 ml PP
bottles and labeled as follows.

0.1 g	U ₂ AN + SiW + 12 in	0.1 M HCl	(.1003 g)
0.1 g	"	"	(.1004 g)
0.2 g	"	"	(.1999 g)

5/20/94

A new monophase synthesis experiment
was initiated and was entitled
'monophase synthesis 14'.

The experimental procedure on p173-174
was followed but the amounts of
reagents were modified as follows:

35 g	calcium acetate
42 g	urea acetate
1.8 g	silicic acid.

5/20/96

Prepared aqueous samples of dissolved
unmelted for analysis by ICP for
U, Si, Ca, Na in Div 01.

Previously dissolved URAW*SYN*12 samples
were diluted for these analyses. All
samples were prepared in a 0.1 M HCl
matrix.

Standards were also prepared in AA
standards and were also prepared
in a 0.1 M HCl matrix.

but no for standards are shown
below.

Si, 1000 ppm - 951765-A-12

Ca, 1000 ppm - 950440-24

Na

U, 1000 ppm standard prepared
from uranyl nitrate (p 156).

Samples and their dilute factors + standards
were labeled as below:

Sample/standard	Solution used or prepared	Dilution factor
20AD	0.1g URAW*SYN*12 in 0.1M HCl (.1004g)	10x
20AE	U-50ppm, Ca-10ppm, Si-15ppm, Na-0ppm	
20AF	0.2g URAW*SYN*12 in 0.1M HCl (.1999g)	10x
20AG	U-140ppm, Ca-5ppm, Si-10ppm, Na-3ppm	
20AH	0.1g URAW*SYN*12 in 0.1M HCl (.1003g)	10x
20AI	U-90ppm, Ca-8ppm, Si-7ppm, Na-1ppm	
20AJ	0.2g URAW*SYN*12 in 0.1M HCl (.1999g)	10x
20AK	0.1g URAW*SYN*12 in 0.1M HCl (.1004g)	10x
20AL	0.1g URAW*SYN*12 in 0.1M HCl (.1003g)	10x

5/28/96 gp

Results of chemical analysis on synthesized uranophane by Div 01 were received (see previous page for sample descriptions).

Results are shown below:

SOUTHWEST RESEARCH INSTITUTE
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SWRI

Date Received: 05/20/96

Matrix: Liquid

Project No.: 20-5708-573

Sample ID	Lab System ID	Calcium Result (ug/mL)	Silicon Result (ug/mL)	Sodium Result (ug/mL)	Uranium Result (ug/mL)
20AD	71369	4.80	6.91	<0.1	54.3
Duplicate	71369	4.82	6.74	<0.1	52.3
RPD	71369	0.4%	2.5%	0.0%	3.8%
Spike	71369	15.6	16.8	8.30	100
Spike Added	71369	10	10	10	50
Recovery	71369	108.0%	98.9%	83.0%	91.4%
20AE	71370	10.3	15.4	21.8	48.1
20AF	71371	9.33	13.5	<0.1	104
20AG	71372	5.16	10.4	17.2	133
20AH	71373	4.77	6.74	<0.1	52.8
20AI	71374	8.24	7.24	10.9	85.1
20AJ	71375	9.44	13.3	<0.1	103
20AK	71376	4.76	6.74	<0.1	51.8
20AL	71377	4.71	6.66	<0.1	51.7

Detection Limit: 0.1 ug/mL 0.1 ug/mL 0.1 ug/mL 0.1 ug/mL

5/28/96

Vessel for 'uranophane synthesis 14' was cooled + opened.

A portion of the solid was removed, dried and analyzed by XRD analysis using procedure in p. 6.

Remainder of solids were removed, filtered, placed in plastic vial and labeled URAN*SYN*14.

5/29/96.

Gravimetric analysis of URAN*SYN*12
and URAN*SYN*14 was conducted
using procedure on p. 182-183.

$$\begin{aligned} \text{wt crucible} &= 12.2510 \text{ g} \\ \text{wt of crucible + URAN*SYN*12} &= 12.4948 \text{ g} \\ \text{wt of URAN*SYN*12} &= .2438 \text{ g} \end{aligned}$$

$$\begin{aligned} \text{wt crucible} &= 11.5341 \text{ g} \\ \text{wt crucible + URAN*SYN*14} &= 11.7842 \text{ g} \\ \text{wt of URAN*SYN*14} &= .2501 \text{ g} \end{aligned}$$

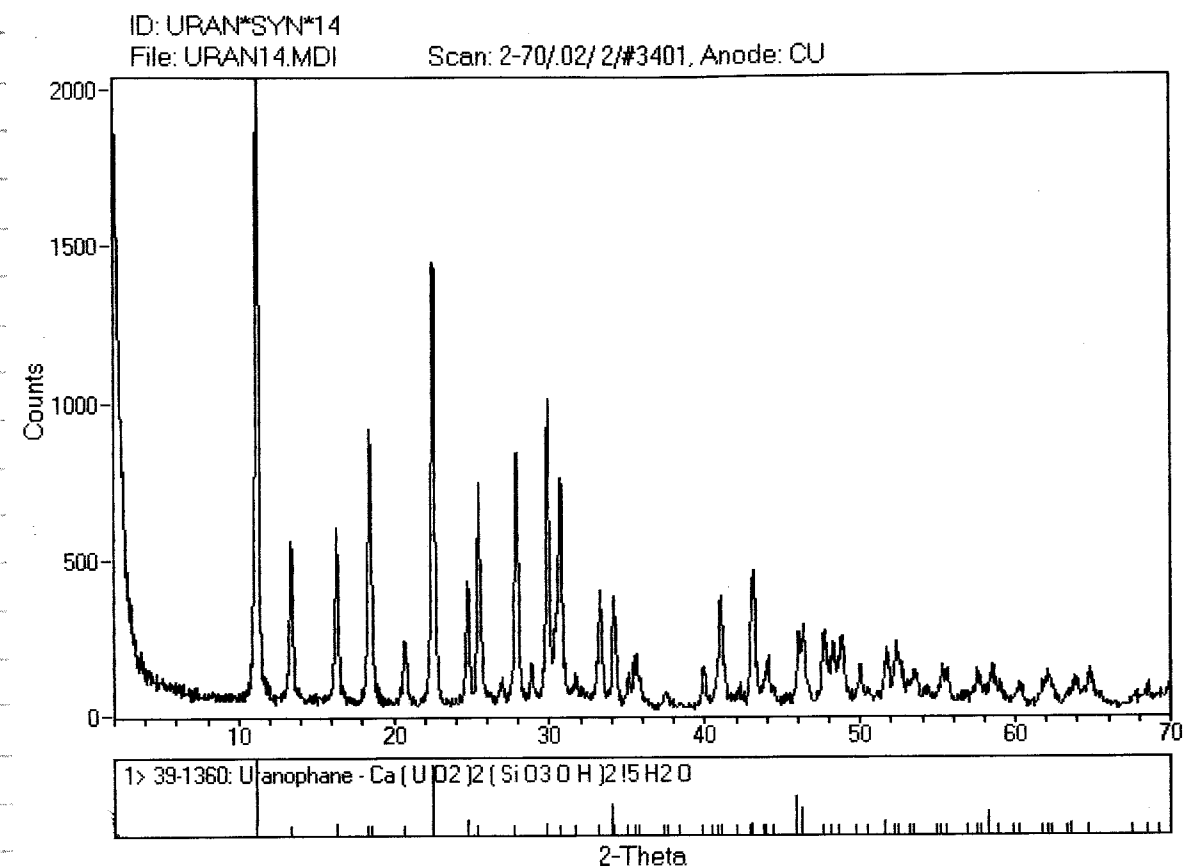
After heat to 900°C for 2 hr.

$$\begin{aligned} \text{wt of crucible + URAN*SYN*12} &= 12.4630 \text{ g} \\ \text{wt H}_2\text{O loss} &= 12.4948 - 12.4630 = .0318 \text{ g} \\ \% \text{ loss} &= .0318 \div .2438 \times 100 = 13.04\% \end{aligned}$$

$$\begin{aligned} \text{wt of crucible + URAN*SYN*14} &= 11.7508 \text{ g} \\ \text{wt H}_2\text{O loss} &= 11.7842 - 11.7508 = .0334 \text{ g} \\ \% \text{ loss} &= .0334 \div .2501 \times 100 = 13.35\% \end{aligned}$$

5/30/96

Results of XRD analysis of URAN*SYN*14



6/21/96 JP

Aliquots of the following samples were sent to Harry Mack at the Univ. of Texas at Austin for U analysis by isotope dilution mass spectrometry.

Each aliquot consisted of approximately 5ml of solution.

20A1

20A2

20A3

20A5

20A6

20A7

20A8

20AA

20AB

20AC

20AD

20AE

20AH

20AK

20AL

All these samples were prepared previously and consist of dissolved synthetic uranophane or standards. These samples have been analyzed for U, Ca, Si, and Na in Div 01 using ICP.

7/9/96

Preparation of matrix solutions for uranophane solubility studies.

Obj: prepare solutions containing accurate U, Ca, + SiO₂

Method: reagent addition

Materials + equipment

- ultrapure water
- CaCl₂ · 2H₂O (lot 913025)
- SiO₂ standard solution (lot 6064-6)
- U standard solution (lot 4-200u)
- 1, .1, and .01 M HCl
- polypropylene bottles (1L)
- Fisher Versabath 236
- Orion pH meter + pH electrode
- 4.0 + 7.0 pH buffers
- Mettler analytical balance AE240
- 0.45 μm syringe filters

Procedure

7/9/96 1000hr ① Into 3 1L volumetric flasks, the following amounts of reagents were added:

1.47 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$
 6ml 1000 ppm SiO_2

② Into individual flasks the following volume of U standard was added:

23.8 ml
 2.38 ml
 .238 ml

Each of the flasks was then filled to the mark with H_2O .

This will produce ^{7/9/96} solutions with the following makeup.

$\text{Ca} - 10^{-2} \text{ M}$
 $\text{SiO}_2 - 10^{-4} \text{ M}$
 $\text{Cl} - 2.012 \times 10^{-2} \text{ M}$

and U with either 10^{-4} , 10^{-5} , or 10^{-6} .

③ These solutions were transferred to PP bottles + labeled as follows:

USE Matrix 10^{-6} M U or
 USE Matrix 10^{-5} M U or
 USE Matrix 10^{-4} M U

④ pH of solutions

USE Matrix $10^{-6} \text{ M U} = 9.15$

USE Matrix $10^{-5} \text{ M U} = 3.23$

USE Matrix $10^{-4} \text{ M U} = 2.14$

⑤ pH of USE Matrix 10^{-4} M U was adjusted to pH of 4.00 by addition of HCl.

⑥ The solutions were placed in a shaking water bath at 25°C .

⑦ pH's will be measured periodically until solutions have come to equilibrium.

Date	pH		
	USE Matrix 10^{-4} M U	USE Matrix 10^{-5} M U	USE Matrix 10^{-6} M U
7/10/96	3.97	3.23	2.12
7/11/96	3.97	3.22	2.12

7/11/16

Uranophane Solubility Experiment

Obj: determine solubility of uranophane synthesized in the lab.

Method: approach uranophane solubility in solutions that are both saturated + undersaturated with uranophane

Materials + equipment

- URAN*SYN*12
- 1 L PP bottles
- Fisher Versabath Model 236
- Orion pH meter + pH electrode
- Mettler AE 240 analytical balance
- Mettler PM 4600 analytical balance
- Matrix solutions: USE Matrix 10^{-4} M U; 10^{-5} M U; 10^{-6} M U
- PP bottles + glassware as needed
- pipets + pipet tips
- pH buffer Fisher certified 7.00 + 4.00

Procedure

- ① label 6 1L PP bottles ^{without caps} and record their weights.

Label	Wt
USE1	91.51 g
USE2	91.84 g
USE3	92.41 g
USE4	92.01 g
USE5	91.51 g
USE6	91.49 g

- ② To each sample bottle the following amounts of URAN*SYN*12 were added.

USE1	.5008 g
USE2	.4998 g
USE3	.5002 g
USE4	.5004 g
USE5	.5011 g
USE6	.5002 g

- ③ To each sample bottle the following amounts ^{of} matrix solutions as indicated were added.

Matrix Solution			
USE1	USE Matrix 10^{-4} M U		100.02 g
USE2	" 10^{-4} M U		100.02 g
USE3	" 10^{-5} M U		100.03 g
USE4	" 10^{-5} M U		100.03 g
USE5	" 10^{-6} M U		100.02 g
USE6	" 10^{-6} M U		100.03 g

- ④ Total weight of sample bottles ^{without caps} + UREAN + SYD#12 + matrix solution were:

	wt
USE1	192.03 g
USE2	192.34 g
USE3	192.94 g
USE4	192.55 g
USE5	192.02 g
USE6	192.02 g

- ⑤ The bottles were capped and placed in a shaking water bath set at 25°C.

- ⑥ At 1 week intervals 5 ml - samples will be removed ^{using a 0.45 μ m syringe filter} from the bottles for Ca, SiO₂, and U analysis.

- ⑦ Two 5 ml samples of the matrix solutions (USE Matrix 10⁻⁴ mU; 10⁻⁵ mU, and 10⁻⁶ mU) were taken and placed in 15 ml PP bottles for later analysis. These samples were labeled as below and show the matrix solution sampled.

Label	Matrix solution
USEM1	USE Matrix 10 ⁻⁴ mU
USEM2	10 ⁻⁴ mU
USEM3	10 ⁻⁵ mU
USEM4	10 ⁻⁵ mU
USEM5	10 ⁻⁶ mU
USEM6	10 ⁻⁶ mU

These samples will show the initial Ca, SiO₂ & U contents before addition of monophase.

- ⑧ Below is shown the sampling data recorded at each sampling interval.

Date/Time	WT of bottle before sampling	USE1 WT of bottle after sampling	WT of solute removed	Sample label
7/18 - 0850	191.98	186.42	5.56	USE1-1
7/25 - 0815	186.33	180.77	5.56	USE1-2
8/1 - 0810	180.70	174.59	6.11	USE1-3
8/8 - 1247	174.53	168.93	5.60	USE1-4
8/15 - 0910	168.93	163.36	5.57	USE1-5
8/22 - 1310	163.31	156.59	6.72	USE1-6

		USE2		
7/18 - 0900	192.29	186.16	6.13	USE2-1
7/25 - 0820	186.11	180.56	5.55	USE2-2
8/1 - 0814	180.54	174.81	5.73	USE2-3
8/8 - 1250	174.79	169.15	5.64	USE2-4
8/15 - 0915	169.15	163.57	5.58	USE2-5
8/22 - 1315	163.60	156.98	6.62	USE2-6

		USE3		
7/18 - 0905	192.91	187.22	5.69	USE3-1
7/25 - 0825	187.15	181.74	5.41	USE3-2
8/1 - 0816	181.68	175.99	5.69	USE3-3
8/8 - 1252	175.99	169.79	6.20	USE3-4
8/15 - 0917	169.78	164.25	5.53	USE3-5
8/22 - 1317	164.24	158.64	5.60	USE3-6

Date/Time	wt of bottle before sample	USE 4 wt of bottle after sample	wt of solids removed	Sample Label
7/18 0905	152.51	186.69	5.82	USE 4-1
7/25 0825	186.66	181.13	5.53	USE 4-2
8/1 0820	181.09	174.85	6.24	USE 4-3
8/8 1255	174.85	169.29	5.56	USE 4-4
8/15 0920	169.28	163.67	5.61	USE 4-5
8/22 1320	148.66	158.19	5.47	USE 4-6

USE 5				
Date/Time	wt of bottle before sample	wt of bottle after sample	wt of solids removed	Sample Label
7/18 810	191.95	186.07	5.88	USE 5-1
7/25 0830	186.01	180.50	5.51	USE 5-2
8/1 0823	180.42	174.31	6.11	USE 5-3
8/8 1257	174.30	168.67	5.63	USE 5-4
8/15 0921	168.67	163.05	5.62	USE 5-5
8/22 1322	163.05	157.45	5.60	USE 5-6

USE 6				
Date/Time	wt of bottle before sample	wt of bottle after sample	wt of solids removed	Sample Label
7/18 0915	191.97	186.38	5.59	USE 6-1
7/25 0830	186.31	180.64	5.67	USE 6-2
8/1 0825	180.58	174.48	6.10	USE 6-3
8/8 1300	174.48	168.64	5.84	USE 6-4
8/15 0924	168.64	163.03	5.61	USE 6-5
8/22 1325	163.03	157.42	5.61	USE 6-6

7/18 ⑨ The pH of all samples taken will be adjusted to ~ 2.0 by addition of 6M HCl if needed. The table below shows the pH upon sampling, the adjusted pH, and the amount of 6M HCl added.

Sample	Initial pH	Adjusted pH	Amount of 6M HCl added
7/18 7/18 1000hr USE M 1	2.14	—	—
USE M 2	2.14	—	—
USE M 3	3.23	1.96	10 μ l
USE M 4	3.23	1.96	10 μ l
USE M 5	3.95	2.07	10 μ l
USE M 6	3.98	1.93	10 μ l
7/18 7/18 1030hr USE 1-1	3.85	2.01	10 μ l
USE 2-1	3.87	2.04	10 μ l
USE 3-1	4.58	1.98	10 μ l
USE 4-1	4.57	2.03	10 μ l
USE 5-1	4.93	2.03	10 μ l
USE 6-1	4.92	1.97	10 μ l
7/25 7/25 0830hr USE 1-2	3.88	1.98	10 μ l
USE 2-2	3.88	1.99	10 μ l
USE 3-2	4.58	2.00	10 μ l
USE 4-2	4.43	2.04	10 μ l
USE 5-2	4.91	1.96	10 μ l
USE 6-2	4.92	2.02	10 μ l
8/1 8/1 USE 1-3	3.91	2.03	10 μ l
USE 2-3	3.90	2.01	10 μ l
USE 3-3	4.58	2.00	10 μ l
USE 4-3	4.58	2.00	10 μ l
USE 5-3	4.53	2.00	10 μ l
USE 6-3	4.89	2.02	10 μ l

	Initial pH	Adjusted pH	Amount of 6M HCl added
7/8/97 USE1-4	3.89	2.25	10ul
USE2-4	3.65	1.98	10ul
USE3-4	4.55	2.03	10ul
USE4-4	4.74	1.90	10ul
USE5-4	4.77	1.98	10ul
USE6-4	4.85	1.96	10ul

8/15/97 USE1-5	3.88	2.00	10ul
USE2-5	3.88	2.01	10ul
USE3-5	4.58	1.99	10ul
USE4-5	4.55	2.01	10ul
USE5-5	4.85	1.99	10ul
USE6-5	4.82	1.96	10ul

8/22/97 USE1-6	3.91	2.11	10ul
USE2-6	3.11	2.06	10ul
USE3-6	4.54	2.00	10ul
USE4-6	4.54	1.99	10ul
USE5-6	4.84	2.00	10ul
USE6-6	4.82	1.99	10ul

7/19/96 gp

Results of U analysis by isotope dilution
mass spectrometry on samples sent to
Harley Mack at UT Austin are
shown below.

No.	ppm U
20A1	56.2
20A2	49.6
20A3	137.8
20A5	57.3
20A6	90.8
20A7	142.0
20A8	57.6
20AA	141.4
20AB	55.2
20AC	57.1
20AD	56.8
20AE	50.0
20AH	56.4
20AK	56.0
20AL	56.5

These values assume that your U has common isotope abundances: $^{235}\text{U} = 0.0072$, $^{238}\text{U} = .992743$.

7/23/96 JP

Preparation of standards for uranophane solubility experiments.

Obj - prepare a set of standards to be analyzed with samples taken in the uranophane solubility experiments for quality control.

Method - addition of reagents + solution standards to ultrapure H_2O

Materials - Equipment

- 7P bottles - 1L
- 1L Volumetric Flasks
- pipets
- $CaCl_2 \cdot 2H_2O$ (lot 913025)
- U standard solution (1000 ppm) (lot 4-200 U)
- SiO_2 standard solution (1000 ppm) (lot 6064-6)
- Mettler AE240 balance
- weighing boats

① A set of 6 standards will be prepared with U, Ca, + SiO_2 contents similar to those expected in samples from the uranophane solubility experiments.

② Into 6 1L volumetric flasks place about 500 ml H_2O . The flasks were labeled as follows:

USE51

USE52

USE53

USE54

USE55

USE56

③ The following amounts or volumes of reagents or standards were added to each flask

	U (ml)	SiO_2 (ml)	$CaCl_2 \cdot H_2O$ (g)
USE51	25	5.5	1.2842
USE52	2.5	6	1.6564
USE53	1	6.5	1.4687
USE54	15	6	1.5580
USE55	10	5	1.3794
USE56	5	7	1.4684

- ④ Below are shown the contents of U, Ca, + SiO_2 in each standard

	U (ppm)	SiO_2 (ppm)	Ca (ppm)
USE51	25	5.5	350
USE52	2.5	6	451.5
USE53	1	6.5	400.4
USE54	15	6	424.7
USE55	10	5	375.7
USE56	5	7	400.3

- ⑤ Standards were transferred to 1L PP bottles and labeled.

- ⑥ 5 ml of each standard was placed in a 15 ml PP bottle. These bottles were labeled as follows. The pH were measured + adjusted to a pH of about 2.0 if needed with 6M HCl.

	Initial pH	Adjusted pH	Volume of 6M HCl added
USE51-1	2.13	—	—
USE52-1	3.22	2.01	10 μL
USE53-1	3.84	1.96	10 μL
USE54-1	2.35	2.08	10 μL
USE55-1	2.53	1.83	10 μL
USE56-1	2.87	1.91	10 μL

7/25/96 gp
0900hr

Chemical analysis of monophase solubility
Samples + standards.

Obj - determine U, Ca, + SiO_2 in samples + standards

Method - ICP Analysis by Div 01

Materials + Equipment

- monophase solubility samples + standards
- PP bottles (15 ml)
- Eppendorf pipets + tips

Procedure

- ① Approximately 3 ml of sample + standard solutions were transferred to 15 ml PP bottles and sent to Div 01 for ICP analysis for Ca, U, and SiO_2 .

② The bottles were labeled as follows:

Sample	Label for KP analysis
USEM1	20u1
USEM2	20u2
USEM3	20u3
USEM4	20u4
USEM5	20u5
USEM6	20u6
USE1-1	20u7
USE2-1	20u8
USE3-1	20u9
USE4-1	20u10
USE5-1	20u11
USE6-1	20u12
USE1-2	20u13
USE2-2	20u14
USE3-2	20u15
USE4-2	20u16
USE5-2	20u17
USE6-2	20u18
USE51-1	20u19
USE52-1	20u20
USE53-1	20u21
USE54-1	20u22
USE55-1	20u23
USE56-1	20u24

8/2 gp
1346 hr Preparation of new standards set
for ICP analysis of Ca, SiO₂, + U
in uraniferous solubility experiments.

5 ml of the standards prepared
previously (p 208-210) were placed in
15 ml pp bottles and labeled as below.
The pH was measured & adjusted
to about 2.0 if needed with 6M HCl.

	Initial pH	Adjusted pH	Volume of 6M HCl added
USES1-2	2.15	-	-
USES2-2	3.22	2.08	10 μ l
USES3-2	3.83	1.98	10 μ l
USES4-2	2.37	1.93	10 μ l
USES5-2	2.54	1.86	10 μ l
USES6-2	2.86	1.94	10 μ l

8/8 gp Preparation of samples for ICP analysis
(see procedure on p 211-212)
3 ml of the following samples + standards
were transferred to 15 ml PP bottles,
labeled as follows, and were sent to
Div 01 for ICP analysis of Ca, SiO₂ + U.

Sample	Label for ICP analysis
USE1-3	20U25
USE2-3	20U26
USE3-3	20U27
USE4-3	20U28
USE5-3	20U29
USE6-3	20U30
USE1-4	20U31
USE2-4	20U32
USE3-4	20U33
USE4-4	20U34
USE5-4	20U35
USE6-4	20U36
USE51-2	20U37
USE52-2	20U38
USE53-2	20U39
USE54-2	20U40
USE55-2	20U41
USE56-2	20U42

8/22/96 gp Results of ICP analysis

SAMPLE ANALYSIS RESULTS

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Project No.: 20-570R-573

Client: Division 20

Date Received: 07/25/96

08/09/96

Date Analyzed: 08/18/96

08/20/96

Sample ID	Lab System ID	Calcium Result (ug/mL)	Silicon Result (ug/mL)	Uranium Result (ug/mL)
FB	---	<0.1	<0.05	<0.001
20U1	75397	453	3.12	26.4
Duplicate	75397	451	3.11	26.3
RPD	75397	0.4%	0.3%	0.4%
20U2	75398	559	3.92	28.1
20U3	75399	440	3.25	2.65
20U4	75400	447	3.32	2.68
20U5	75401	440	3.21	0.249
20U6	75402	461	3.36	0.254
20U7	75403	499	88.7	810
20U8	75404	497	88.3	796
20U9	75405	470	12.7	68.8
20U10	75406	490	13.4	70.2
20U11	75407	475	7.39	13.3
20U12	75408	487	7.72	12.9
20U13	75409	552	98.5	796
20U12	75410	600	108	755
20U14	75411	479	14.1	67.6
20U15	75412	454	13.6	64.4
20U16	75413	459	8.66	11.7
20U17	75414	458	8.61	11.8
20U19	75415	392	2.78	27.0
20U20	75416	498	2.96	2.80
20U21	75417	435	3.19	1.12
20U22	75418	467	2.99	16.8
20U23	75419	438	2.68	11.2
20U24	75420	451	3.36	5.37
Duplicate	75420	457	3.63	5.39
RPD	75420	1.3%	1.9%	0.4%
20U25	76145	505	89.8	781
Duplicate	76145	507	90.3	796
RPD	76145	0.4%	0.6%	1.9%
20U26	76146	504	90.3	788
20U27	76147	450	14.4	64.7
20U28	76148	446	14.3	63.4
20U29	76149	445	9.59	11.9
20U30	76150	449	9.72	11.8
20U31	76151	502	89.8	772
20U32	76152	496	89.4	760
20U33	76153	446	14.9	63.4
20U34	76154	450	15.3	65.1
20U35	76155	448	10.2	11.9
20U36	76156	447	10.3	12.6
20U37	76157	377	2.79	26.6
20U38	76158	485	2.99	2.6
20U39	76159	432	3.21	1.05
20U40	76160	460	3.01	16.2
20U41	76161	407	2.43	10.9
20U42	76162	438	3.50	5.80

Detection Limit: 0.1 ug/mL 0.05 ug/mL 0.001 ug/mL

8/29/96 gp

1040 hrs Preparation of new standard set
for ICP analysis of Ca , SiO_2 , + U
in sample solubility experiments.

5 ml of standards prepared
previously (p 208-210) were placed in
15 ml bottles + labeled as
below. The pH was measured
and adjusted to ~2 if needed
with 6M HCl

	meas pH	Adjusted pH	Vol of 6M HCl added
USE S1-3	2.15	-	-
USE S2-3	3.23	1.99	10ul
USE S3-3	3.85	2.03	10ul
USE S4-3	2.38	1.87	10ul
USE S5-3	2.54	1.92	10ul
USE S6-3	2.90	1.93	10ul

8/30/96 gp

0945 hrs gp

Preparation of sample for ICP analysis
(see procedure ~ p 211-212).

3 ml of the following samples + standards
were transferred to 15 ml PP bottles,
labeled as below, + sent to Div 01 for
ICP analysis of Ca , SiO_2 , + U .

Sample	label for ICP analysis
USE 1-5	20u43
USE 2-5	20u44
USE 3-5	20u45
USE 4-5	20u46
USE 5-5	20u47
USE 6-5	20u48
USE 6-1	20u49
USE 6-2	20u50
USE 6-3	20u51
USE 6-4	20u52
USE 6-5	20u53
USE 6-6	20u54
USE S1-3	20u55
USE S2-3	20u56
USE S3-3	20u57
USE S4-3	20u58
USE S5-3	20u59
USE S6-3	20u60

9/16/96 gp
0900hrs Results of ICP analysis for samples listed
on previous page.

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Liquid

Project No.: 20-5708-573

Client: Division 20

Date Received: 08/30/96

Date Analyzed: 07/18/96

Sample ID	Lab System ID	Calcium Result (ug/mL)	Silicon Result (ug/mL)	Uranium Result (ug/mL)
20U43	77520	491	88.6	740
Duplicate	77520	473	84.8	714
RPD	77520	3.7%	4.4%	3.6%
20U44	77521	496	90.7	755
Spike	77521	882	170	1550
Spike Added	77521	444	88.9	889
Recovery	77521	86.9%	89.2%	89.4%
20U45	77522	440	15.3	61.9
20U46	77523	444	15.4	62.7
20U47	77524	438	10.4	11.8
20U48	77525	443	10.5	11.7
20U49	77526	485	87.4	732
20U50	77527	482	88.2	737
20U51	77528	430	15.3	60.3
20U52	77529	439	15.4	61.5
20U53	77530	466	11.2	12.2
20U54	77531	432	10.5	11.2
20U55	77532	364	2.42	25.8
20U56	77533	472	2.60	2.53
20U57	77534	412	2.73	1.08
20U58	77535	442	2.53	15.5
20U59	77536	390	2.03	10.3
20U60	77537	414	2.91	5.03

Detection Limit:

0.1 ug/mL

0.1 ug/mL

0.1 ug/mL

I have reviewed this notebook
and find it in general
compliance with QAP-001.
There is adequate information
for another qualified
person to repeat the
activity.

E.C. Perry
1/9/87

Final Entry:

This notebook appears to comply
with QAP-001.

E.C. Perry
2/4/2000