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11/2/94 gp Initial Entries are made on p. 56.

4/26/93 gp

XRD analysis of uraninite-bearing sample from Nopal I

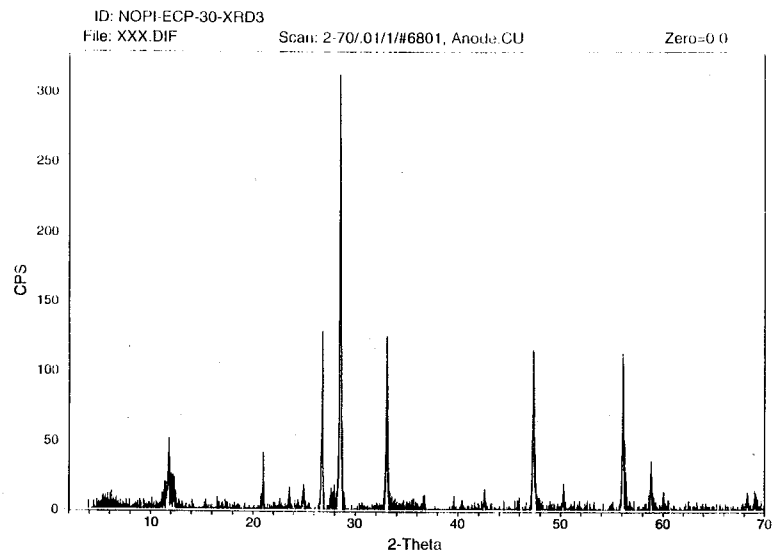
Sample = NOPI-ECP-30

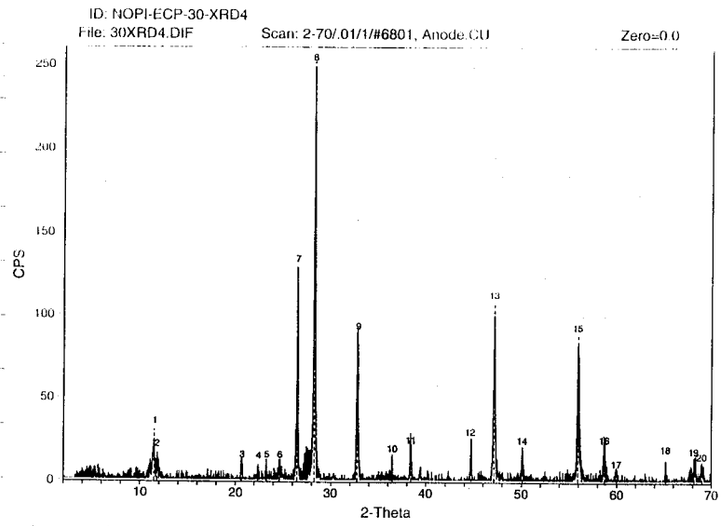
Portion of sample was processed for XRD analysis

Subsample = NOPI-ECP-30-XRD3

4/27/93 gp

Subsample = NOPI-ECP-30-XRD4
This sample contains Al powder for calibration.





Jade: Peak Listing — Fri May 07 1993 02:31pm

File: 30XRD4.DIF> NOPI-ECP-30-XRD4

Scan Parameters:		Search Parameters:
Radiation = CU_1.540598		Filter length(pts) = 9
Scan Range = 2 - 70		Noise level(sigmas) = 5.0
Step Size = .01		Intensity cutoff(%) = .5-100
Count Time = 1 sec.		2-Theta Zero (degs) = 0

	Peak-Position		Centroid-Position		Peak & Area are without Bkgrd					
#	2Theta	d	2Theta	d	Bkgrd	Peak	I%	Area	I%	FWHM*
1:	11.480	7.7019	11.479	7.7025	5	27	10.7	118	2.7	0.039
2:	11.801	7.4929	11.803	7.4919	1	18	7.1	92	2.1	0.046
3:	20.620	4.3039	20.627	4.3025	1	12	4.7	167	3.8	0.125
4:	22.379	3.9696	22.377	3.9698	1	11	4.3	41	0.9	0.034
5:	23.252	3.8225	23.252	3.8225	2	11	4.3	30	0.7	0.025
6:	24.610	3.6144	24.617	3.6134	1	12	4.7	223	5.0	0.167
7:	26.449	3.3672	26.458	3.3660	1	130	51.4	1589	35.7	0.110
8:	28.245	3.1571	28.245	3.1570	1	253	100.0	4446	100.0	0.158
9:	32.736	2.7335	32.743	2.7329	1	89	35.2	1541	34.7	0.156
10:	36.440	2.4637	36.430	2.4643	1	15	5.9	86	1.9	0.052
11:	38.429	2.3406	38.420	2.3411	1	20	7.9	380	8.5	0.171
12:	44.702	2.0256	44.700	2.0257	2	24	9.5	243	5.5	0.091
13:	47.128	1.9268	47.128	1.9268	1	108	42.7	1989	44.7	0.166
14:	50.061	1.8206	50.061	1.8206	1	20	7.9	204	4.6	0.092
15:	55.919	1.6430	55.925	1.6428	1	88	34.8	1526	34.3	0.156
16:	58.700	1.5716	58.702	1.5715	3	19	7.5	189	4.3	0.090
17:	59.939	1.5420	59.941	1.5420	2	6	2.4	79	1.8	0.118
18:	65.157	1.4306	65.152	1.4307	1	15	5.9	74	1.7	0.044
19:	68.173	1.3745	68.176	1.3744	4	11	4.3	53	1.2	0.043
20:	68.980	1.3603	68.977	1.3604	2	10	4.0	150	3.4	0.135

* Intensity values are based on counts per second.

4/27/93 JP

XRD patterns and for NOPI-ECP-30-XRD3 and XRD4 are shown above along with peak listing for NOPI-ECP-30-XRD4. Additional XRD data (for uraninite (bearing samples) is kept in a 3 ring binder entitled "XRD Analysis and Cell Refinment of Nopal I Uraninite".

4/28/93 JP

Microscopy at the Center for High Resolution Electron Microscopy at Arizona State University

Day was spent doing microdiffraction and EELS analysis of granular maninite from Nopal I. A Phillips FEG 400ST was used to do the analyses and Dr. Marija Grabowiec assisted and operated the equipment.

Summary:

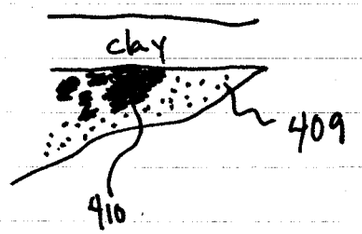
Sample U1 - location 1

All photos and spectra are kept in a 3-ring binder entitled "High Resolution Electron Microscopy of Nopal I uraninite - Arizona State University CHREM".

Exposure 405 (9000x)
Photo of granular uraninite - intergrown Uraninite & kaolinite

Exposures 406 (23000x), 407(100,000x) and 408 (340,000x) are progressive closeups.

Micro diffraction of area -
consists of dark grains or particles of varying shape and size in a gray background. Gray area appears to consist of much smaller dark particle and is crystalline.
Dark particle are UO_{2+x} while gray may be more oxidized or some other uranyl phase or thinner area.
Manya suggested that gray material could be clay with very small dark particle as inclusions based on diffraction pattern.



20Å beam size
Diffraction pattern - 450 mm camera length
Exposure 409 - gray area

Traverse of diffraction patterns from middle of dark particle to edge.
Exposure 410 - middle of dark particle
Exposure 411 - near middle of dark particle - some orientation
Exposure 412 - belt of small particle before gray region. At edge of dark particle
Exposure 413 - At tail of dark particle, small particle like 412.

Same area but looking along zone axis.
Exposure 414 - near center of particle along zone axis
Exposure 415 - at edge of dark particle, many small particle - additional spots on pattern.

Exposure 416 - gray area adjacent to black particles, along zone axis

Same area as before but using smaller convergence angle:

Exposure 417 - Dark particle (2 sec exposure)

Exposure 418 - Dark particle (10 sec exposure)

Exposure 419 - gray region adjacent to dark particle (2 sec exposure)

Exposure 420 - gray region adjacent to dark particle (10 sec exposure).

Exposure 421 - selected area diffraction pattern of whole area

*Note - locations of diffraction patterns are marked on print #407 in 3 ring binder entitled "High .... CTREM".

Sample U1 - location 2

Exposure 422 - 9000 x
Exposure 423 - 23000x
Exposure 424 - 100,000x

Above exposures are progressively higher magnification photos of granular inclusion.

Microdiffraction

Dark, well defined crystal, have ordered diffraction pattern.
Gray are less defined and have disordered diffraction pattern evidenced by spots suggesting many orientations of crystals.
Diffraction pattern have additional spots.

Exposure 425 - Darkest part of particle, zone axis rectangular

Exposure 426 - middle of gray region, additional spots. Gray part of particle. Shows larger spacing rings. Similar to [illegible] pattern

EELS spectra were collected from sample U1 at location 2 ~~1~~. Spectra were saved to disk for later analysis. 4/22/93

Location 1

(EELS - energy loss spectrometry records energy loss due to breaking of chemical bonds) (100 msec analysis time)

SWRI01.eel - gray region, polycrystalline material

SWRI02.eel - dark particle

SWRI03.eel - kaolinite, amorphous material.

At this point parameters of equipment were changed in order to collect better oxygen spectra. (400V offset, 1.0 eV/channel, slit #2, and 137 mm camera length)

SWRI04.eel - gray region

SWRI05.eel - dark particle

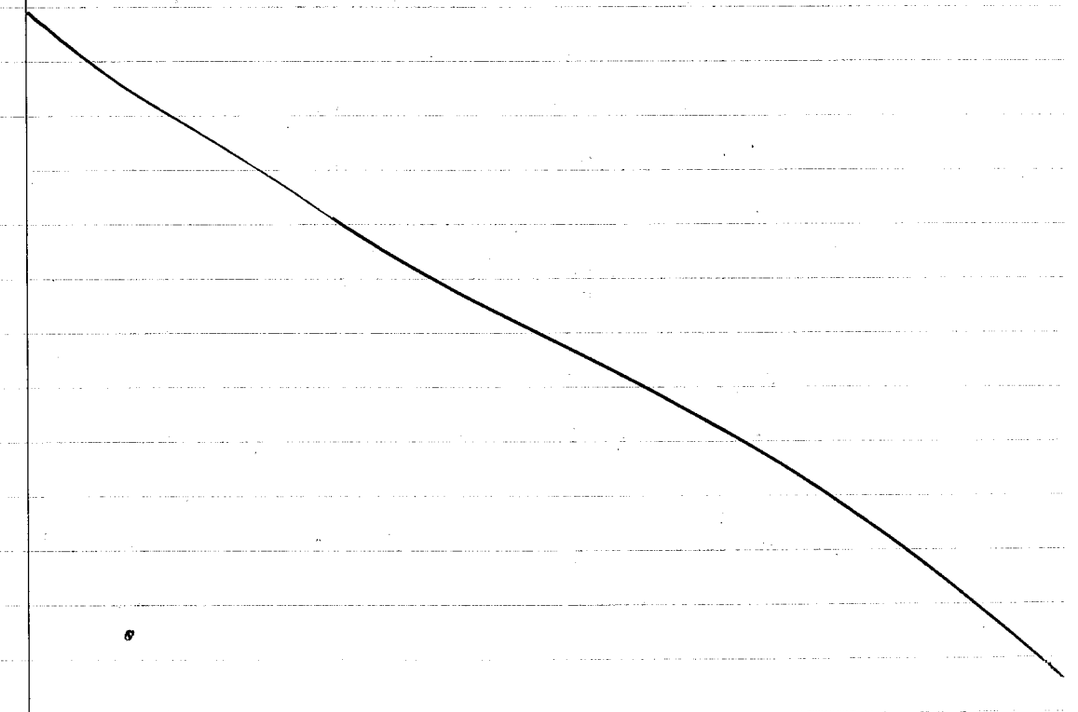
SWRI06.eel - amorphous region, clay

Moved to a thinner region

SWRI07.eel - dark particle

SWRI08.eel - gray polycrystalline region.

Exposure 427 - (23000X) image of area where spectrum SWRI07.eel & SWRI08.eel were taken.



4/29/93 JP EDS microanalysis at ASU using the VG HB 501 microscope. Alan Higgs assisted and operated the equipment.
Parameters - 100 sec acquisition, 5-10 A spot analyses.
Spectra were printed and also saved to disk for latter analysis.

Summary:

Sample U1 - location 1

Filename	Hardcopy spectrum / Photo location	Description
SWRI01.XRA	5 / 5	kaolinite - clay
SWRI02.XRA	2 / 2,6	Dark particle (UO_2).
SWRI03.XRA	1 / 1,7	dark particle (UO_2)
SWRI04.XRA	6,3,4 / 3,4,8	gray region
SWRI05.XRA	7 / 9	Darker gray area
SWRI06.XRA	8 / 10	light gray area, white gray
SWRI07.XRA	9 / 11	~~Dark~~ gray area. JP 4/29/93
SWRI08.XRA	10 / 12	light gray or white gray area

* locations of spot analyses are marked on photos kept in binder "High... CHREM".

Sample U1 - location 2

File name	Hardcopy spectrum / Photo Location	Description
SWRI09.XRA	11 / 13	dark particle
SWRI10.XRA	12 / 14	~~very light~~ gray area JP 4/29/93
SWRI11.XRA	13 / 15	clay - kaolinite
SWRI12.XRA	14 / 16	dark gray region
SWRI13.XRA	15 / 17	dark particle in gray regi
SWRI14.XRA	16 / 18	dark gray area

Sample U1 - location 3
corresponds to location of EELS analyses SWRI07.rel + SWRI08.eel

Filename	Hardcopy spectrum / Photo location	Description
SWRI15.XRA	17 / 19	clay
SWRI16.XRA	18 / 20	dark particle.

Sample U2 -
Yi Ming Pan collected the following spectrum from this sample.

Sample location	Filename	Hardcopy Spectrum / Photo Location	Description
location 1 ↓	SWRI17.xRA	17 / 1	dark particle
	SWRI18.xRA	18 / 2	gray area
	SWRI19.xRA	19 / 3	light area
	SWRI20.xRA	20 / 4	very light area
	SWRI21.xRA	21 / 5	dark large particle
location 2 ↓	SWRI22.xRA	22 / 6	dark area in large particle
	SWRI23.xRA	23 / 7	gray area
	SWRI24.xRA	24 / 8	white area
location 3 ↓	SWRI25.xRA	25 / 9	gray area
	SWRI26.xRA	26 / 10	Dark area
	SWRI27.xRA	27 / 11	Light area
location 4 ↓	SWRI28.xRA	28 / 12	dark area in UO2
	SWRI30.xRA	30 / 13	gray area

4/30/93 JP

Sample U2 -
JP collected additional spectra (EDS) from this sample.
Granular area.

Filename	Hardcopy Spectra / Photo location	Remarks
SWRI31.xRA	31 / 31	dark gray region
SWRI32.xRA	32 / 32	black particle
SWRI33.xRA	33 / 33	light area
SWRI34.xRA	34 / 34	dark gray region
SWRI35.xRA	35 / 35	dark particle
SWRI36.xRA	36 / 36	light gray region

See photo with spot analyses labeled.

Collected an element map of the above analyzed area.
Image was 128x128 pixels and images for Oxygen, aluminum, silicon, and uranium were collected and saved.

Filename	Element
SWRI05.PIC	Oxygen
SWRI06.PIC	aluminum
SWRI07.PIC	Silicon
SWRI08.PIC	Uranium

EDS and EELS files were converted to ascii text files and written to a floppy disk labelled "ASU hrem data". This disk is MAC formatted.

PIC files will be converted to ~~binary~~ ascii text on 4/30/93 and ftped to San Antonio at a later date.

4/30/93 Lattice imaging at ASU using the JEM-4000EX microscope. Molly McCartney and Dave Smith assisted in the analysis and equipment operation.

Sample U1
location 1

Plate No	Mag	Remarks
A62303	5000x / 2 sec	intergrown textures of UO_2 - clay.
A62304	5000x / 4 sec	negatives to be cleaned
A62305	5000x / 5.6 sec	
A62306	5000x / 2.8 sec	No obj. aperture
A62307	50,000x / 2.8 sec	closeup of previous
A62308	500,000x	No obj. aperture
A62309	500,000x	

A62310	500,000x	
A62311	500,000x	
A62312	500,000x	

Sample U1 - location 2

A62313	500,000x	see photo A62317 for location
A62314	500,000x	"
A62315	500,000x	"
A62316	500,000x	"
A62317	250,000x	Photo showing location of 500,000x lattice images.
A62318	250,000x	"
A62319	500,000x	see photo A62317 for location
A62320	500,000x	"

A62321	500,000x	"
A62322	50,000x	

Sample U3 - location 1

A62323	500,000x	white area
A62324	30,000x	location photo.
A62325	500,000x	interface between black + gray.
A62326	500,000x	black region
A62327	500,000x	black/gray interface
A62328	500,000x	"

New location

A62329	500,000x	Contact between amorphous + gray region
A62330	500,000x	"

A62331	500,000x	Dark material
A62332	500,000x	
New location		
A62333	500,000x	Granular material
A62334	500,000x	"
A62335	500,000x	"
A62336	500,000x	"
A62337	500,000x	"
A62338	500,000x	"
A62339	250,000x	"

Sample U2
location 1

A62 340	500,000x	Granular material
A62 341	500,000x	"
A62 342	500,000x	"
A62 343	200,000x	"
New location		
A62344	500,000x	"
A62345	500,000x	"
A62346	500,000x	"
A62347	200,000x	"
A62348	200,000x	"
A62349	20,000x	"

New location

A62350	500,000x	"
A62351	500,000x	"
A62352	500,000x	"
A62353	200,000x	"
A62354	500,000x	"
A62355	20,000x	

Dr YiMing Pan did some lattice constant calculations while at ASU using the negatives of the lattice images. A video camera was used to display parts of the negative on a TV display. A software program was then used to determine distance between lattice planes.

Dr. YiMing Pan's notes on the lattice calculations are kept in the 3 ring binder entitled "High... CHREM."

5/6/93 JP All data files (XRAY, EELS, and PIC) collected at ASU CHREM were transferred by computer to San Antonio using FTP.

XRAY and EELS files were converted to ascii text files before transfer, PIC files were converted to binary before Transfer.

After transfer files were copied to 3.5" floppy disks for archival. Disk labels and contents are explained on the following page.

Disk label	Contents
ASU CHREM 1	EELS files (SWRI01.eel - SWRI08.eel) PIC files (SWRI05.pic - SWRI08.pic)
ASU CHREM 2	XRAY files (SWRI01.xra - SWRI26.XRA) + some calibrated XRAY files (SWRI01c.XRA - SWRI16c.XRA)
ASU CHREM 1	XRAY files (SWRI21.XRA - SWRI36.XRA)

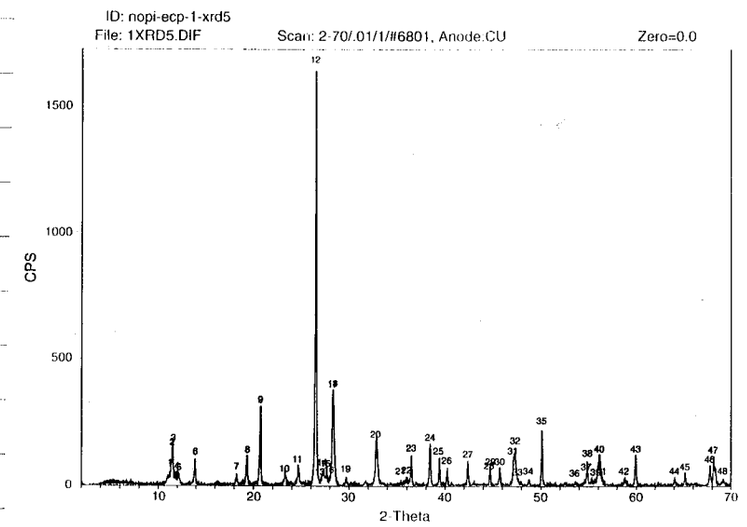
11/2/94 gp

Disks are kept in Fireproof file cabinet in the geochemistry lab.

5/6/93 gp XRD analysis of uranite-bearing sample from Nopal I

Sample = NOPI-ECP-1

XRD Subsample = NOPI-ECP-1-XRD5 contains Al powder for calibration



Jade: Peak Listing — Fri May 07 1993 01:29pm

File: 1XRD5.DIF> nopi-ecp-1-xrd5

Scan Parameters:
Radiation = CU_1.540598
Scan Range = 2 - 70
Step Size = .01
Count Time = 1 sec.

Search Parameters:
Filter length(pts) = 9
Noise level(sigmas) = 4.0
Intensity cutoff(%) = .5-100
2-Theta Zero (degs) = 0

#	Peak-Position 2Theta	d	Centroid-Position 2Theta	d	Bkgrd	Peak & Area are without Bkgrd Peak	I%	Area	I%	FWHM*
1:	11.198	7.8949	11.198	7.8950	2	58	3.5	1962	7.5	0.304
2:	11.420	7.7422	11.426	7.7381	2	144	8.7	5708	21.8	0.357
3:	11.489	7.6956	11.486	7.6976	2	161	9.7	4148	15.8	0.232
4:	11.861	7.4556	11.861	7.4556	2	51	3.1	2868	10.9	0.506
5:	12.142	7.2836	12.141	7.2841	2	46	2.8	892	3.4	0.175
6:	13.889	6.3712	13.887	6.3719	1	105	6.4	1556	5.9	0.133
7:	18.237	4.8605	18.241	4.8596	1	48	2.9	613	2.3	0.115
8:	19.332	4.5877	19.327	4.5889	2	115	7.0	1782	6.8	0.139
9:	20.710	4.2855	20.710	4.2856	1	316	19.1	4390	16.8	0.125
10:	23.271	3.8193	23.276	3.8186	1	41	2.5	881	3.4	0.193
11:	24.676	3.6050	24.675	3.6050	1	79	4.8	1414	5.4	0.161
12:	26.523	3.3579	26.522	3.3581	4	1653	100.0	26201	100.0	0.143
13:	26.937	3.3073	26.936	3.3074	2	25	1.5	152	0.6	0.055
14:	27.228	3.2726	27.228	3.2726	2	61	3.7	1529	5.8	0.226
15:	27.618	3.2272	27.616	3.2274	2	57	3.4	3426	13.1	0.541
16:	27.952	3.1895	27.957	3.1889	4	34	2.1	202	0.8	0.053
17:	28.282	3.1530	28.287	3.1524	3	374	22.6	10295	39.3	0.248
18:	28.331	3.1476	28.330	3.1478	3	376	22.7	10445	39.9	0.250
19:	29.717	3.0039	29.714	3.0042	1	39	2.4	404	1.5	0.093
20:	32.821	2.7266	32.821	2.7265	1	176	10.6	5275	20.1	0.270
21:	35.365	2.5360	35.370	2.5357	1	22	1.3	201	0.8	0.082
22:	36.034	2.4904	36.034	2.4904	1	35	2.1	966	3.7	0.248
23:	36.465	2.4620	36.461	2.4623	1	126	7.6	1600	6.1	0.114
24:	38.443	2.3397	38.438	2.3401	1	167	10.1	2539	9.7	0.137
25:	39.399	2.2852	39.401	2.2851	1	109	6.6	1311	5.0	0.108
26:	40.241	2.2393	40.231	2.2398	2	71	4.3	702	2.7	0.089
27:	42.372	2.1315	42.374	2.1313	1	95	5.7	1299	5.0	0.123
28:	44.652	2.0278	44.665	2.0272	2	53	3.2	752	2.9	0.128
29:	44.749	2.0236	44.736	2.0241	2	68	4.1	608	2.3	0.080
30:	45.706	1.9834	45.713	1.9831	2	69	4.2	1014	3.9	0.132
31:	47.142	1.9263	47.151	1.9260	1	111	6.7	4248	16.2	0.344
32:	47.352	1.9183	47.349	1.9183	1	151	9.1	5089	19.4	0.303
33:	47.661	1.9065	47.657	1.9067	1	28	1.7	501	1.9	0.161
34:	48.759	1.8661	48.758	1.8662	1	23	1.4	244	0.9	0.095
35:	50.085	1.8198	50.091	1.8196	1	230	13.9	2759	10.5	0.108
36:	53.609	1.7082	53.613	1.7081	1	15	0.9	141	0.5	0.085
37:	54.796	1.6740	54.795	1.6740	1	49	3.0	971	3.7	0.178
38:	54.855	1.6723	54.856	1.6723	1	100	6.0	1336	5.1	0.120
39:	55.739	1.6479	55.739	1.6478	1	29	1.8	709	2.7	0.220
40:	56.140	1.6370	56.138	1.6371	1	118	7.1	4480	17.1	0.342
41:	56.422	1.6295	56.421	1.6295	1	33	2.0	563	2.1	0.154
42:	58.810	1.5689	58.806	1.5690	2	31	1.9	410	1.6	0.119
43:	59.927	1.5423	59.931	1.5422	1	120	7.3	1909	7.3	0.143
44:	64.025	1.4531	64.032	1.4530	1	33	2.0	311	1.2	0.085
45:	65.139	1.4309	65.145	1.4308	1	50	3.0	625	2.4	0.112
46:	67.750	1.3820	67.752	1.3820	1	79	4.8	1340	5.1	0.153
47:	68.180	1.3743	68.177	1.3744	1	113	6.8	2760	10.5	0.220
48:	69.175	1.3570	69.176	1.3569	1	31	1.9	427	1.6	0.124

* Intensity values are based on counts per second.

5/19/93 JP

Lattice constants for Nopal I uraninite were calculated from XRD patterns containing uraninite using the computer program LCLSQ version 8.5. This program was loaded and resides on the IBM PC in the geochemistry lab. The manual explains how xrd. data is input into the program and output from the program is kept in a 3 ring binder entitled "XRD analysis and cell refinement of Nopal I uraninite".

Lattice constants were calculated for uraninite using xrd data from sample NOPI-ECP-1-XRD5 and NOPI-ECP-30-XRD4. Results are kept in binder Entitled "XRD analysis & cell refinement of Nopal I uraninite".

Entries on pages 27-29 made by Sabine Thomas
JP 7/21/94 ECP 7/21/94

7/7,93 TRANSCRIPT OF ACTIVITIES between June 10 and June 24, 1993:

6/10, 6/14, 6/15 checked H/F (= heating / freezing) stage, wires and tube connections,
checked scales / magnification of the various lenses on both H/F stage and other NIKON microscopes: the one with the

on NIKON optiphot-Pol with eye piece CFW 10x:

		10 units on scale bar	1 unit on bar
lense	M Plan 5	200 µm	20 µm
	M Plan 10	100 µm	10 µm
	M Plan 20	50 µm	5 µm
	M Plan 40	25 µm	2.5 µm

on NIKON Optiphot-2 with eye piece CFWN 15x:

		10 units on scale bar	1 unit on bar
lense	~~F~~EF 4	250 µm	25 µm
	EF 10	100 µm	10 µm
	Plan 40	25 µm	2.5 µm

centered sub stage condense lense,
replaced one broken window of the H/F stage,
cleaned all other windows,
studied the manuals of the H/F stage.

6/18 through 6/21

CRUSHED SAMPLE 393-7.5/33.8 (calcite)
checked for f.i. (= fluid inclusions)

measured two of the fragments sorted out
results on one of the worksheets designed by
Jim Prikryl (loose, in ring binder)
observed $T_H \rightarrow L$ between +45°C and +50°C

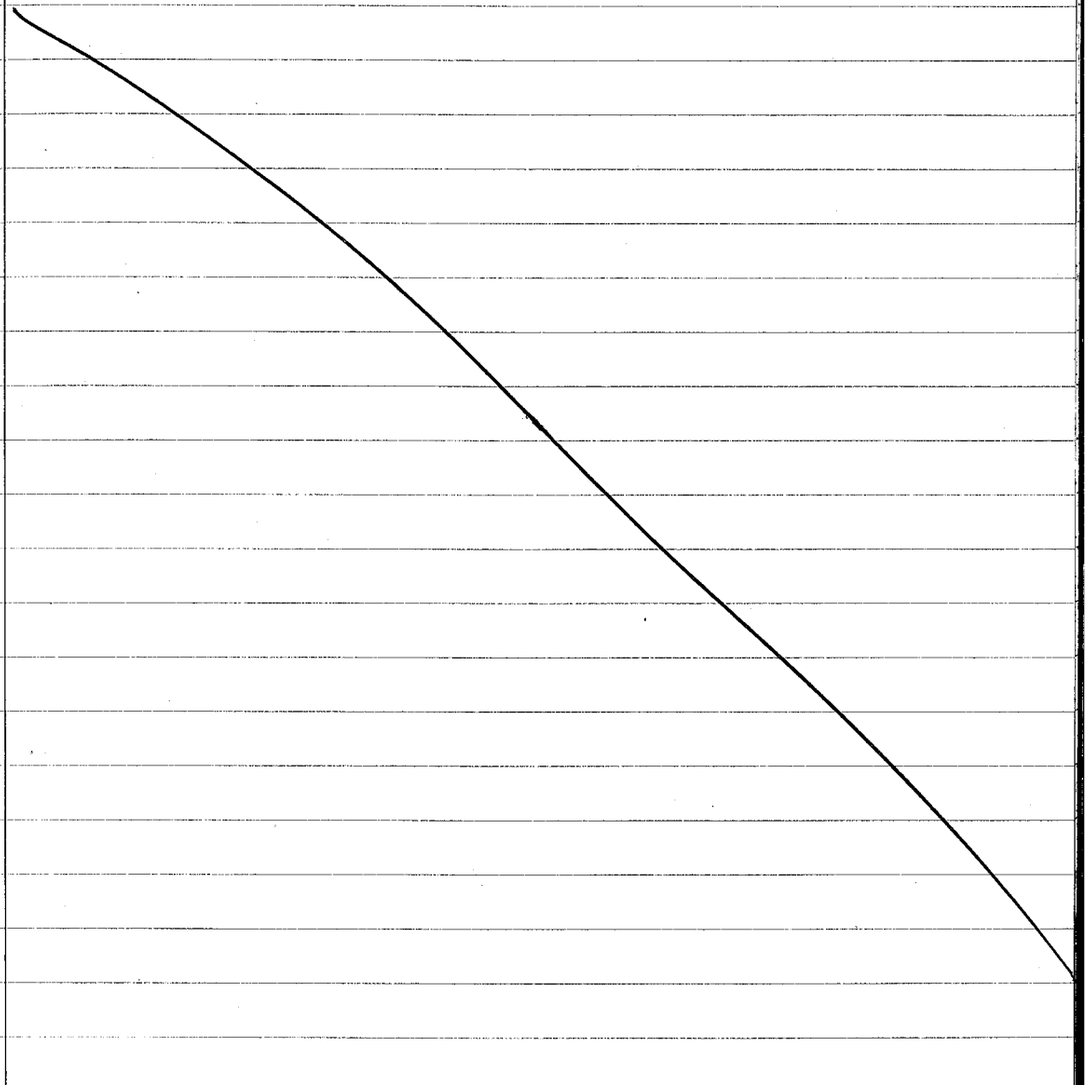
none of the bubbles returned upon cooling to
room temperature; not even after 3 days.
some bubbles returned during the second freezing
run,
final melting T indicate NO SALT.
No clear indication for CO_2-H_2O (or other?)
clathrates, but a suspicious bubble deformation
at T between -27°C and -5°C was observed.

Stretching in calcite yields a second false
T_H in one f.i. of +213°C, $\rightarrow L$ after first
T_h of +46°C.

all f.i. on fractures, presumably secondary
no solid inclusions (daughters etc) observed.

Typically, at T > +250°C, many cracks appeared
at f.i. points and shape changes occurred.
Occasional implosions.

Decided to order thick sections of Sample
393-7.5/33.8, a supposed by "early calcite"
to see if more false high T_H appear
↝ that could explain high T_H reported in
Ariel's thesis.



7/13/93

TEM sample preparation.

Thin sections NOPI-ECP-32-TS1, NOPI-ECP-32-TS3, and NOPI-ECP-30-TS1 were removed from glass slides by soaking in methylene chloride.

Areas on the thin sections that contain appreciable UO_2 especially the colloform variety were selected by viewing the sections under the Nikon petrographic microscope. These areas were marked on 8.5" x 11" photographs of the thin sections that were taken earlier.

	Neg. No.
NOPI-ECP-32-TS1	E90184
NOPI-ECP-32-TS3	E90182
NOPI-ECP-30-TS1	E89585

Two areas on each of the above thin section were selected for TEM analysis

Samples taken from these areas will be labelled as follows.

Sample	Thin section
30A	NOPI-ECP-30-TS1
30B	"
32A	NOPI-ECP-32-TS1
32B	"
32C	NOPI-ECP-32-TS3
32D	

Thin sections removed from the glass slides were placed in plastic boxes and placed in the dehumidifier in the petrography lab.

7/14/93 gp

Samples for TEM analysis removed from the thin sections by Yi-Ming Pan of Div 06.
Dr. Pan will prepare the samples (ion mill and mount) for TEM analysis in Div 06.

TRANSCRIPT, done July 14, 1993

Entries on pages 33 to 40 made by Sabine Thomas. ECP 7/21/94

6/22 and /23

CALIBRATION of H/F stage

checked filters between NIKON optiphot-2 and light source:
with H/F stage in place, top insulation post screwed on, diaphragm open to the max, condenser lens centered
Plan 40 lens in working distance:
Light intensity set to "8"

	Reading of DORIC 410 A
- without any filter	+ 30.0°C
- IR filter (in broken frame, single)*	+ 26.8°C
- with IR and NCB II* filter	+ 24.9°C
- with NCB II filter only	+ 26.2°C

(further temp drop when 2. polas is in light path)

*(NCB II filter used for color photography)
* filter is light blue; labeled frame with "IR"

For calibration of HF I followed description in FLUID INC INSTRUCTION MANUAL, Dec 1991, starting with page 30 (and p. 24, resp.):

(A) - Immersed tip of thermocouple into a bath of ice cubes (probably frozen normal tap water) and liquid nanopure water. Waited several minutes. Without stirring temps fluctuate between −0.2°C and +0.5°C. Temperatures stay

at 0°C (with - sign flashing occasionally) when beaker is swerved around.

→ No adjustment necessary for trendicator.

(B) Measurement with Standard #1 of the TEMPERATURE CALIBRATION STANDARDS by SYNFLINC with $X_{H_2O} = .75$ and $X_{CO_2} = 0.25$ and a Calibration temp of -56.6°C

Did 2 runs with "cycling" (tried to stop or reverse temp course several times ~~before~~ below final melting temp T_f to keep the standard's environment cool and to make it easier to reverse temp course without significant lag. Came to stops at temp several tenths of a degree below -56.6°C, e.g. at -56.8°C and later at -56.7°C; briefly ~~touched~~ touched (split second) -56.6°C without yielding T_f yet.
Then hit -56.6 (with -56.5°C flashing up) and melting occurred.

Results first run: -56.6°C
second run: -56.6°C

→ No correction of "negative span" of trendicator necessary.

(A) Just letting the stage warming up by itself without "cycling" yielded a temperature increase of about 11°C/minute.
T_f in this case -56.4°C.

(Homogenization temp of Standard #1 between +29.5°C and +29.6°C in f.i. in field of view, all into the liquid phase.)

(C) STANDARD #4, pure H_2O
calibration temp: T_f 0.01°C
T_H +374.1°C
with 10 SCFH
at around -5°C: temp increase of 1.3°C/min
between -1°C and 0°C: temp increase of 0.2°C/min
Trafo setting at 7 or 8 ~~Watts~~ Volts
Final melting temp: -0.2°C / -0.2°C / -0.3° to -0.2°C
-0.2°C
set brass screw to 0.0°C while melting was observed during the last two runs, took about 1/6 of a full turn in clockwise direction
→ melting now at at 0.0°C with "-" sign flashing
Then realized that f.i. in standard was not close to the tip of the thermocouple.

Moved same inclusions closer to the tip of the thermocouple
→ melting temp now ±0.2°C

Adjusted brass screw back to an intermediate position between original and newly set.
→ (final) melting now at 0.0°C.

⇒ there is a temp gradient across the thick section with respect to the distance of the f.i. from the tip of the thermocouple (see also paragraph 3, p.25 in Instruction Manual):
a f.i. further away from the thermocouple yield too low a melting temp., i.e. an erroneously higher salt content would be concluded from that (probably also in connection with measurements of clathrate dissociation: T_{diss} too low → too much salinity assumed)

For temps to increase below -5°C setting at trafo between 13 and 15 Watts worked well.

(D) Same STANDARD #4 (pure H_2O) used for calibration in the high temp range
T_{crit} : +374.1°C
left chip in same position;
temp almost constant with trafo setting at 64/65 Watts in temprange of ~370°C

observed critical homogenization temperatures:
+ 373.1 °C
+ 373.1 °C
+ 373.1 °C

⇒ Deviation of -1°C, NO CORRECTION NECESSARY!

Replaced the second window from the bottom of the bottom part (broken)

6/24 cut various samples to find suitable ones to send out for thick sections:
393 - (-) 75.40 14.16
393 - (-) 75.40 14.25
393 - (-) 75.40 14.32
} did not deem suitable

Send out 2 calcite samples with 2 pieces each:
393 - 5.5 / 32.7
393 - 7.5 / 33.8

7/15 Sample 22.25 / 17.15 - TS 3 ;
measured or rather heated fragments up to check if observed inclusions are solid or fluid.
Made two heating runs with same chip, first to +255°C, second to +302°C.
Observed increasing darkening (getting browner and browner) of ? epoxy, surface of rock fragment

also appears wizened, views and observations very difficult.
Immersed another fragment in ~~at~~ acetone (from 1:50 pm until 11 am, July 16.)
added the fragment that had already been heated at 3:40 pm, took it also out at 11 am, July 16.)

"Cycled" a second fragment of sample 22.25/17.15 TS-3 heated up to +212 and after, cooling down to +110°C, heated up to +250° C
↘ all observed inclusions deemed to be solid.

Thick sections of calcite samples (2 × 2 pieces) 393-5.5/32.7 and 393-7.5/33.8 arrived.
Selected one of the 393-5.5/32.7 pieces and immersed in acetone.

July 16 Detached sample 393-5.5/32.7 from glass slide and left pieces in ~~at~~ acetone between 11 am and 3 pm.

Heated fragment-immersed in acetone the previous day-of sample 22.25/17.15-TS3 to see if bathing acetone had any effect.
Sample starts to smell burned at +180°C, but faintly.

Darkening and crumpeling of surface starts at +250°C, coming out of a crack
The rim of the sample is still appearing as epoxy and is brown, too.

Put the burned piece and a "fresh" piece of sample 22.25/17.15-TS3 in a bath with methylene chloride.

With sample 22.5/10.4-TS3 a similar observations
it started to smell strongly at +250°C and got brown, at +350°C it started to creep/run/disperse all over the slip.

July 21 Removed and checked fragments of sample 22.25/17.15-TS3 from methylene chloride
↘ glue rims unchanged, still there, without any sign of corrosion

put piece of calcite sample 393-5.5/32.7 in methylene chloride.

~~Heat~~ Performed several freezing runs at other fragment of 393-5.5/32.7, see loose f.-i. sheet.

July 22 Performed another measurement with Standard * 4 (pure H_2O) to check the "zero" setting of the DORIC 410 A.

1. run: T_m at +0.3°C
2. " : T_m +0.4°C

— correction of zero setting, it's now actually very close to its very original position: N-S.

3. T_m after correction: 0°C
4. T_m 0°C

The observed fluid inclusions were not close to the tip of the thermocouple. They were at some distance, in a position that is more likely to occur and is more comparable to real measurements.

So it might be that temp. of melting ice in fluid inclusions close to the tip of the thermocouple might be too high.

I corrected the T_m values measured at calcite sample 393-5.5/32.7 by rising them by +0.3°C (e.g. T_m: -0.4 now -0.1°C)

8/12/93 JP

Additional samples for TEM were prepared. Samples were removed from previously removed thin sections that have been removed from their glass slides.

These thin sections are NOPI-ECP-32-TS1, NOPI-ECP-32-TS3, and NOPI-ECP-30-TS1.

Areas that were removed were marked on 8.5" x 11" photographs of the thin sections.

Samples taken were labelled as follows.

Sample	Thin Section
30C	NOPI-ECP-30-TS1
30D	"
32E	NOPI-ECP-32-TS1
32F	"
32G	"
32H	NOPI-ECP-32-TS3.
32I	
32J	

Samples were given to Dr. Ti Ming Pan in Div 06 for TEM milly and monty for TEM analysis.

11/01/93

Entries on pages 43 to 46 made by Sabine Thomas. ECP 7/21/94

Fluid-inclusion investigation during October
- on calcite

Decided to discontinue further research on sample 393-5.5/32.7 because:
f.i. tend to collapse after or during freezing runs, before room temp was attained
lots of bubbles appear after the freezing runs, suggesting that the volume of bubbles in L-V-f.i. might also have changed.
In addition still lots of glue attached to the wafer even after bathing them in methylene chloride.

Measured several chips of 393-7.5/33.8 (also calcite)
volume of gas phase in many cases < 10%
so bubbles often disappears during freezing runs and did not return, rendering T_f (ice) useless, and inhibiting measurements of T_h

Some bubbles (?) did not change at all, neither during freezing nor heating runs.

Calcites are pretty unreliable candidates!

Investigations on Quartz

Sample NOPI-ECP-39-FI
wafers had different thicknesses, the thicker ones (.5 mm?) are preferable.

Same problem with most f.i. because of their low gas content: they blink out during freezing and the bubbles ~~do~~ often do not return in time to measure T_f (ice).
Many bubbles appear after heating (no decripitation)
Often bubble collapse, and no return at all.
during freezing runs

Experiments with the UV lamp:

checked for fluorescence to distinguish SiO_2 lode groundwater precipitate such as Opal from higher-T "vapor-phase quartzes" originating from magmatic fluids.

The only ones that fluoresced were the opals from Sample NOPI-ECP-19.0/5.0

and a tiny yellowish amygdule filling (?) from Sample NOPI-ECP-15.2/10.1

Samples that I checked but that did not fluoresce:

MESA-ECP-1	brown vitrophyr
MESA-ECP-2	reddish devitrified vitrophyr
NOP-ECP-2	Obsidian remnants
NOPI-ECP-5	reddish Tertiary with bleached veinlets
NOPI-ECP-8	Nopal fm with bleached veinlets
-10	silicified vein
-38	vesicle fillings
-39	qtz-xx from vesicles
-40	" " "
-45	vitrophyr
-46	vapor-phase quartz
NOPI-ECP-15.9/9.95	Hydrophane-limonite ... red-yellow veinrock
15.7/10.0	Cc? around bleached vein rock
15.2/10.1	reddish rock in vein (appear leached)

various samples from a prominent E-W trending fracture on the +10 level of Nopal I"
NOPI-ECP-0.0/13.4 through -14.6/13.43
most of the bags were empty, could not find
NOPI-ECP-3.78/13.7,
-8.45/13.82,
-9.40/13.61,
-12.22/13.35.

Inspected also Samples
NOPI - 10-16 /3.5, pieces of cc/qtz concretions
NOPI - 20-28 /1, ~~about~~ euhedral vapor phase qtzes from vug
thick sections and pieces from
NOPI-ECP-39-FI.
None of them fluoresced.

JP 11/29/93
About 8g of UO_2 powder purchased from AESAR was sent to Mark Arendt at UT Austin (Center for Materials Chemistry, Dept. of Chemistry) for XPS analysis to determine U^{+4}/U^{+6} ratio

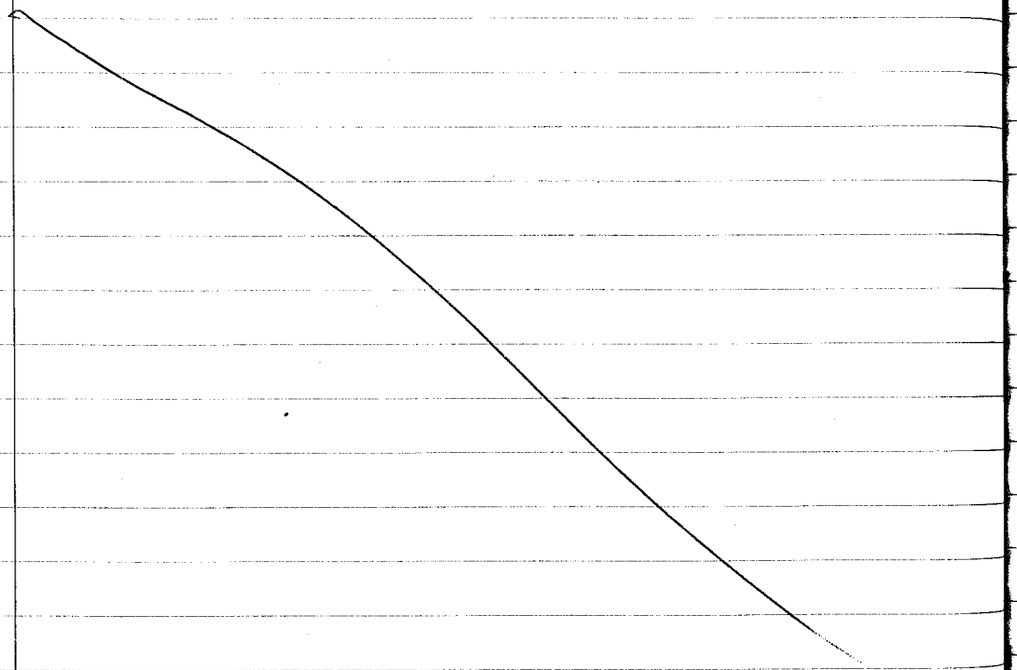
JP 12/17/93
Results and comments on the XPS analysis of the UO_2 powders done at UT Austin were recieved by fax. and are shown on the following pages.

U/O ratio of unheated sample = .30

Analyses have error of ± 5%.

literature reference = National Institute Standards Database.

UO_2 is difficult to characterize by XPS. Thin is variability in reported values (eV) in the literature (UO_2, U_4O_9, U_3O_8). How sample is treated and prepared for analysis may effect analysis.



COLLEGE OF NATURAL SCIENCES
THE UNIVERSITY OF TEXAS AT AUSTIN

Center for Materials Chemistry · Welch Hall 3.310
Department of Chemistry · Austin, Texas 78712-1167 · (512) 471-3704

December 16, 1993

James D. Prikryl
Center for Nuclear Waste Regulatory Analyses
6620 Culebra Rd.
P. O. Drawer 28510
San Antonio, Texas 78228-0510

Dear Mr. Prikryl,

The preliminary XPS analysis you requested on 11/29/93 of the UO_x powder is as follows:

1. The as received powder was pressed into a one cm diameter copper analysis stub. The pressed powder was then loaded into UHV and pumped down to $2x10^{-10}$ torr. Photo-ionization was accomplished with an Aluminum Kα x-ray source (hυ = 1486.6 eV).

2. A survey analysis from 0 to 1000 eV binding energy revealed only U, O, and aliphatic carbon.

3. High resolution XPS of the as is powder (see figures 1-3, "before" spectra) showed multiple oxidation states for the U but due to differential charging individual oxidation states are unresolvable. It appears that clusters of oxides/organics lose electrons to the vacuum space through the photo-ionization process but are electrically isolated from ground. Thus the binding energies of elements in the clusters are shifted higher due to the localized accumulation of charge. If this is the case then each core-orbital should give rise to two peaks; a charged and uncharged peak with intensities proportional to the charged and uncharged sampling volumes. The C 1s, U 4f7/2, and O 1s spectra (figures 1-3 labeled "before") all reveal a twin peak or structure at ~ 3.3 eV higher binding energy. And each charge shifted structure is roughly the same percent intensity of its lower binding energy twin. Finally, the corresponding lowest binding energy peaks in figures 1-3 agree well with literature reference values.

4. In order to resolve the charging problem the sample was heated to 673 K at $1x10^{-9}$ torr for one hour. Baking the sample at high temperature ought to drive off any

residual water, hydroxides, and organic contributions to the substrate which give rise to the insulating clusters.

5. Figures 1-4 reveal the affects of baking on the UO_X powder. The C 1s peak area is decreased by 3.73 times compared to the pre-bake spectra and contains only one aliphatic component. The O 1s peak area remains constant and the shape is consistent with a predominate single metal oxidation state with a small higher oxidation state shoulder. The U 4f spectra are concomitant with the O 1s observations. Figure 4 was generated by first assuming a single oxidation state (U^{4+}) with a peak center acquired from literature. The single oxidation state fit consistently failed to give the same shape envelope or area as the original data envelope. Using literature acquired positions for UO_2 and UO_3 the fit envelope in figure 4 agrees with the data envelope to within 0.4%. Also, from the fit data the stoichiometric ratio of U/O = 0.47 which agrees well with the value (U/O = 0.44) generated by dividing the spectrometer corrected uranium peak area by the spectrometer corrected oxygen peak area.

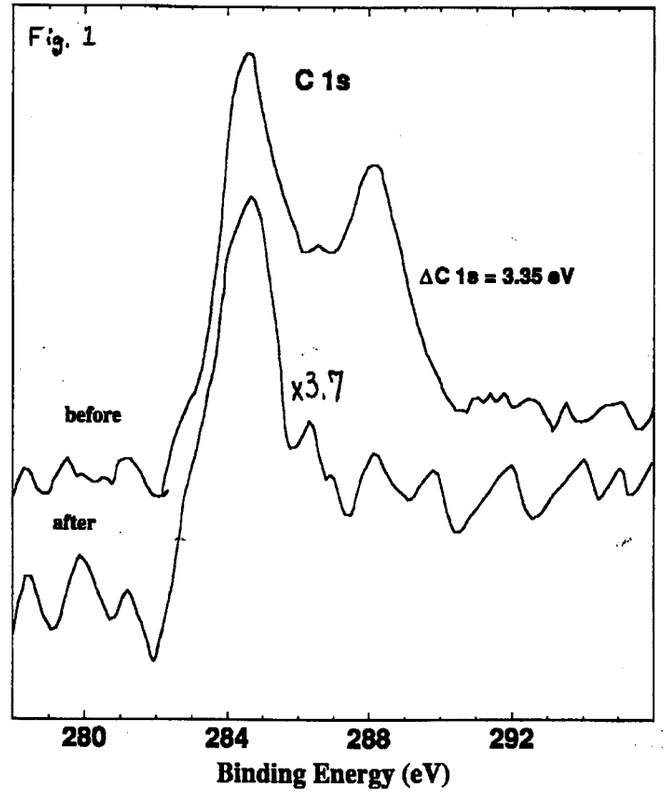
Please let me know if you need further assistance or you have more experiments in mind.

Sincerely,

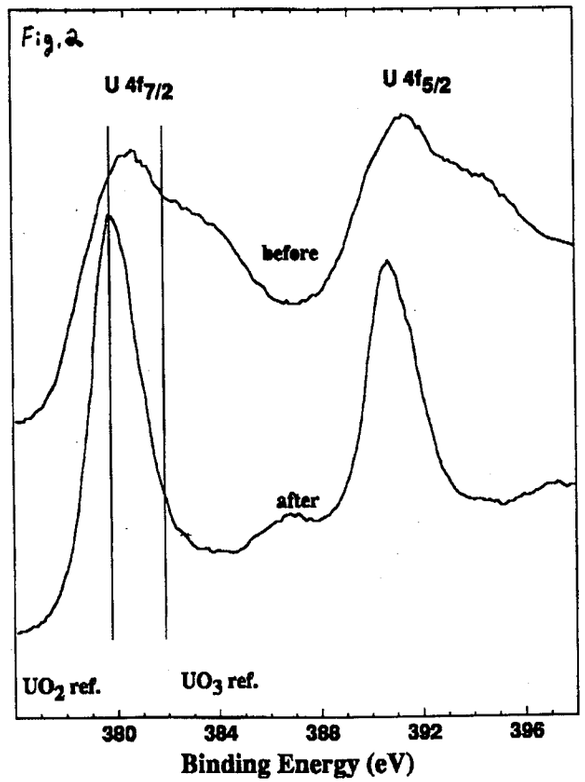
M. Arendt

Mark F. Arendt
Res. Sci. Assc.

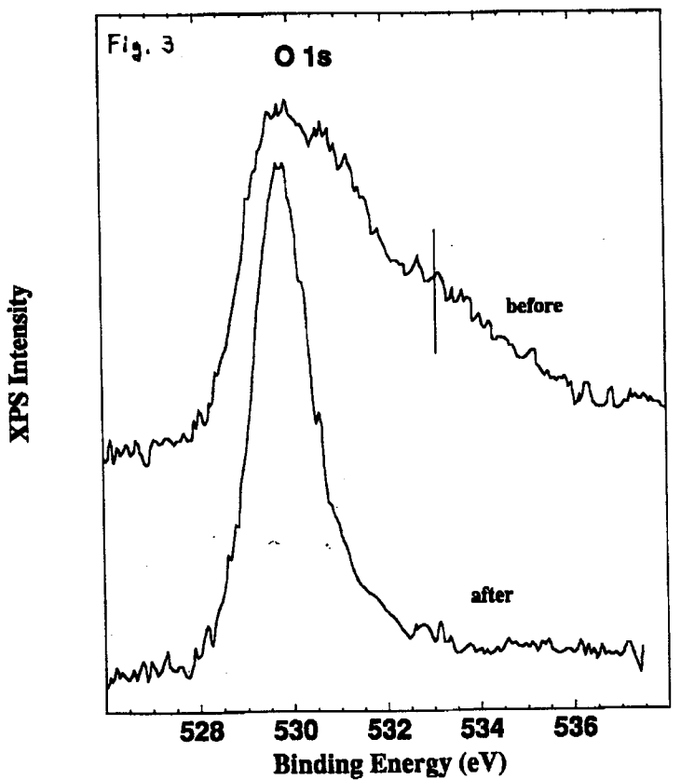
XPS of UO_x
Before and after heating to 673 K



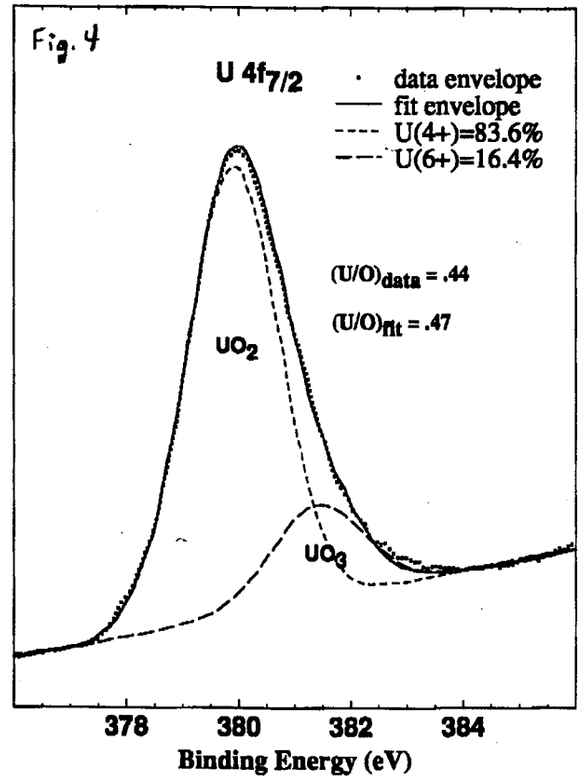
XPS of UO_x
Before and after heating to 673 K



XPS of UO_x
Before and after heating to 673 K



Curve Fit of U $4f_{7/2}$ XPS data
After heating to 673 K for 1 hr.



11/2/94 JP Geochemical Analog of Contaminant Transport in Unsaturated Rock Research Project.

Initial Entry 4/26/93 by James O. Fuhs JP.

This notebook chronicles the laboratory investigations of the Analogs Research Project.

Pages 1 through 56 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Folck
SwRI - QA
11/28/94

1/9/95 JP

Alpha-spectrometry procedure.

The following pages contain a new alpha-spectrometry procedure for Nopal I rock samples that uses microwave digestion and source preparation for U and Th on filters.

ALPHA SPECTROMETRY ANALYSIS OF NOPAL I ROCK SAMPLES USING SAMPLE DIGESTION

Written by: J. D. Prikryl
Date written: 12/29/94

Objective: Determine the distribution and concentration of U and Th series isotopes in powdered rock specimens from Nopal I

Equipment:
- EG&G Alpha Spectrometry System
  - -576A dual spectrometers or 676A single spectrometers with ion-implanted-silicon particle detectors
  - -Model 920-16 multichannel buffer
  - -ALPHAMAT analysis software for acquistion control
  - -MAESTRO II multichannel analyz emulation software for analysis of spectral data
- CEM Model MDS-2000 microwave digestion system
- Teflon PFA vessels
- Analytical balance
- Hot plates
- Fisher Marathon 21K centrifuge
- Ultrasonic bath

Supplies:
- Powdered rock samples from Nopal I
- Glassware as needed (beakers, volumetric flasks, funnels, etc)
- BIO-RAD anion exchange resin AG 1-X8 100-200 mesh chloride form
- BIO-RAD glass columns 1.5 cm diameter
- BIO-RAD glass columns 0.7 cm diameter
- $^{232}U/^{228}Th$ spike solution prepared previously
- Fe carrier solution prepared previously
- Weighing paper and boats
- PP bottles
- 50 ml PP and polycarbonate centrifuge tubes
- 25 mm membrane filters
- 50 ml polysulfone filter funnel
- 1 inch stainless steel planchets

Reagents:

Conc HCl	$HClO_4$	NH_4OH
9 M HCl	0.1 M HCl	1 M HCl
Conc HNO_3	Conc HF	8 M HNO_3
0.1 N HNO_3	0.05 M EDTA solution	

- Cerous nitrate (0.5 mg/ml Ce)
- 25% hydrazine dihydrochloride
- 10 M NaOH
- 80% ethanol
- Ceric hydroxide (10 µg Ce/ml) substrate
- 10% sodium hydrogen sulfate
- 20% titanium trichloride
- Cerous fluoride (~10 µg Ce/ml) substrate

Procedure: The digestion, separation, and source preparation procedure shown below is used to process samples.

U/Th Digestion (Nopal rock samples)

Sample I. D. # _____

Date _____

Acid Digestion by Microwave: This method is useful for total decomposition of many different types of materials including rocks.

1. Consult CEM Microwave Sample Preparation Applications Manual for the microwave sample preparation note for the type of sample (e.g., Fe-oxides, tuff, etc...) to be dissolved. The application note discusses the amount of sample and reagents to be used, and program parameters to be entered for the microwave digestion.

   Application Note = _____.

2. Record dry weight of sample = _____ g and place in a teflon PFA vessel.

3. Using a weighing boat, quantitatively add a known amount of $^{232}U/^{228}Th$ spike to the PFA vessel.

   $^{232}U/^{228}Th$ spike # _____ Reference Date _____

   Reference Activity _____ pCi/g Spike wt. _____ g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
_____	_____
_____	_____
_____	_____
_____	_____

4. Seal vessel, place on turntable in microwave, enter and run digestion program. Cool and vent vessel. If sample is not completely dissolved repeat step 4 .

5. Quantitatively transfer sample to a clean teflon beaker, washing the PFA vessel several times with ultrapure water.

6. Split sample into two parts: half is saved in a PP bottle for later processing if necessary and the other half is analyzed for U and Th isotopes.

Extraction:

7. Add 1 ml of perchloric acid ($HCLO_4$) to the sample solution in the teflon beaker. Evaporate to fumes of $HCLO_4$. Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).

8. Transfer solution to a 50 ml PP centrifuge tube. Add ~10 mg Fe carrier and coprecipitate actinides by addition of NH_4OH to pH = 7.
   **Note: if sample already contains significant Fe, then Fe carrier does not need to be added.

9. Separate the Fe scavange by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.

10. Dissovle the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Column Separation:

Resin: BIO-RAD Anion Exchange Resin AG 1-X8 100-200 mesh chloride form.
(Lot # _____)

Main Column (Biorad 1.5 cm diameter column with 10 cm resin; prewash with 4-5 column vols 9M HCl)

Load sample in 9M HCl and allow to drain	___			
Wash 3 volumes (~35 ml) 9M HCl --> Th	___	___	___	
Elute 4 volumes (~50 ml) 0.1M HCl --> U and Fe	___	___	___	___

Separation Date _____

***Note: May work on U and Th fractions simultaneously from this point on.

Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Heat Th fraction to evaporate HCl	___			
Add ~5 ml conc HNO_3 to dissolve residue	___			
Add equal vol (~5 ml) DI water so soln 8M HNO_3	___			
Load onto column in 8M HNO_3	___			
Wash 3-4 column vols 8M HNO_3	___	___	___	
Elute 4-5 column vols 9M HCl --> Th	___	___	___	___

Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Evaoprate U fraction to near dryness	___			
Pick up in about 2 ml conc HNO_3	___			
Dilute with about 2 ml DI water to approx 8M HNO_3	___			
Load onto column in 8M HNO_3	___			
Wash 2 vols (8-10 ml) 8M HNO_3 --> Fe	___	___		
Elute 4-5 vols 0.1M HCl --> U	___	___	___	___

Source Preparation:

Thorium - OH^- precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO_3 and evaporate to dryness.
2. Add 6 ml 0.05 M EDTA soln to dissolve residue, transfer soln to a 50 ml PP centrifuge tube and place tube in boiling water for a few minutes.
3. Add 100 µl purified cerous nitrate (0.5 mg/ml Ce) to precipitate hydroxides. Mix, add 2 drops 25% hydrazine dihydrochloride, and 2 ml of 10M NaOH.
4. Place tube in boiling water bath for 10 minutes, remove, and place tube in cold water for 10 minutes to ensure complete precipitation
5. Wet a 25 mm membrane filter with 80% ethanol and place in a 50 ml polysulfone filter funnel.
6. Shake bottle of substrate suspension (ceric hydroxide containing 10 µg Ce/ml) vigorously and draw 2 consecutive 5 ml portions through the filter with full suction. Allow each portion to suck dry for 10-15 sec.
7. Without interrupting suction, pour the sample into the filter chimney and allow to suck dry.
8. While the sample is still filtering, add 0.5 ml 10M NaOH to the sample tube and about 5 ml ultrapure water down the sides of the tube. After the sample has sucked dry, swirl the wash solution around the sides and add it to the filter chimney.
9. Wash tube, filter chimney, precipitate, and filter with three consecutive 5 ml portions of 80% ethanol.
10. Suck filter dry for about 15 sec and remove the chimney and filter carefully without interrupting the suction. Transfer filter to a plastic container and at low temperature (~60°C).
11. Glue to tape the dry filter onto a 1 inch stainless steel planchet and count.

Th Counting Date_____

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 100 µl puriified cerous nitrate (0.5 mg/ml Ce, 50 µg of Ce carrier) and evaporate the solution until completely dry and no more fumes are given off.
2. Add 2 ml of 1M HCl to the beaker containing the purified U fraction and heat gently to dissolve the sodium hydrogen sulfate cake and any possible insoluble double salts with the Ce carrier.
3. Transfer solution to a 50 ml polycarbonate centrifuge tube with two more 2 ml portions of 1M HCl.
4. Add 2 drops of 20% titanium trichloride which should produce a strong violet color. If not, iron is probably present and a few more drops of titanium trichloride must be added to produce a permanent violet color or reduction and precipitation of U will be incomplete.
5. Add 0.5 ml (10 drops) of 48% HF. The violet color dissappears and any slight turbidity should clear up.
6. Mix thoroughly and allow solution to stand for 30 min in a cold water bath to obtain complete precipitation of cerous and uranous fluorides.
7. Place the tube in an ultrasonic bath for 1 min to disperse the precipitate.
8. Mount the precipitate on a 25 mm membrane filter previously treated with two 5 ml portions of cerous fluoride substrate as described above.
9. After sucking the precipitate dry, wash with 5 ml of water containing 2 drops of 48% HF and then with 80% ethanol.
10. Dry and analyze as described above.

U Counting Date_____

1/3/95 JP

The following samples were analyzed for U + Th isotopes by α-spectrometry using procedure on p 59-62.

NOPI-294	powder from	NOPI-294-XRD2
NOPI-301	powder from	NOPI-301-XRD2
NOPI-417	portion of	NOPI-417-WR1
NOPI-418	" "	NOPI-418-WR1
NOPI-419	" "	NOPI-419-WR1
NOPI-420	" "	NOPI-420-WR1
NOPI-421	" "	NOPI-421-WR1
NOPI-422	" "	NOPI-422-WR1
NOPI-423	" "	NOPI-423-WR1
NOPI-425	" "	NOPI-425-WR1

Results are kept in a 3-ring binder entitled "Alpha-Spectrometry of Nopal I samples".

1/9/95 gp

The following samples were analyzed for U + Th isotopes by α-spec using procedure on p 59-62.

NOPI-113	Portion of	NOPI-113-GAM1
NOPI-114	" "	NOPI-114-GAM1
NOPI-115	" "	NOPI-115-GAM1
NOPI-116	" "	NOPI-116-GAM1
NOPI-117	" "	NOPI-117-GAM1
NOPI-118	" "	NOPI-118-GAM1
NOPI-119	" "	NOPI-119-GAM1
NOPI-120	" "	NOPI-120-GAM1
NOPI-121	" "	NOPI-121-GAM1
NOPI-122	" "	NOPI-122-GAM1
NOPI-123	" "	NOPI-123-GAM1
NOPI-424	" "	NOPI-424-WR1

Results are kept in 3-ring binder entitled "Alpha Spectrometry of Nopal I samples".

1/20/95 gp

The following samples were analyzed for U + Th isotopes by α-spec using procedure on p 59-62.

NOPI-209	Portion of	NOPI-209-GAM1
NOPI-307	Portion of	NOPI-307-WR1
NOPI-397	Portion of	NOPI-397-WR1
NOPI-398	" "	NOPI-398-WR1
NOPI-399	" "	NOPI-399-GAM1
NOPI-400	" "	NOPI-400-GAM1
NOPI-401	" "	NOPI-401-WR1

Results are kept in 3-ring binder entitled "Alpha Spectrometry of Nopal I samples".

1/26/95 gp

The Following samples were analyzed for U + Th isotopes by α-spec using procedure on p 59-62

NOPI-402	portion of NOPI-402-WR1
NOPI-403	" " NOPI-403-WR1
NOPI-404	" " NOPI-404-WR1
NOPI-405	" " NOPI-405-WR1
NOPI-406	" " NOPI-406-WR1
NOPI-407	" " NOPI-407-WR1
NOPI-408	" " NOPI-408-WR1
NOPI-409	" " NOPI-409-WR1
NOPI-410	" " NOPI-410-WR1
NOPI-411	" " NOPI-411-WR1
NOPI-372	" " NOPI-372-WR1
NOPI-373	" " NOPI-373-WR1

Results are kept in 3-ring binder entitled "Alpha Spectrometry of Nopal I Samples".

2/1/95 gp

The Following samples were analyzed for U + Th isotopes by α-spec using procedure on p 59-62.

NOPI-212	portion of NOPI-212-GAM1
NOPI-214	" " NOPI-214-GAM1
NOPI-215	" " NOPI-215-GAM1
NOPI-216	" " NOPI-216-GAM1
NOPI-217	" " NOPI-217-GAM1
NOPI-218	" " NOPI-218-GAM1
NOPI-219	" " NOPI-219-GAM1
NOPI-221	" " NOPI-221-GAM1
NOPI-374	" " NOPI-374-WR1
NOPI-375	" " NOPI-375-WR1
NOPI-376	" " NOPI-376-WR1
NOPI-377	" " NOPI-377-WR1

Results are kept in 3-ring binder entitled "Alpha Spectrometry of Nopal I Samples".

2/13/95 JP

The following samples were analyzed for U+Th isotopes by α-spec using procedure on p 55-62.

NOPI-378	portion of	NOPI-378-WR1
NOPI-379	" "	NOPI-379-WR1
NOPI-380	" "	NOPI-380-WR1
NOPI-381	" "	NOPI-381-WR1
NOPI-382	" "	NOPI-382-WR1
NOPI-383	" "	NOPI-383-WR1
NOPI-384	" "	NOPI-384-WR1
NOPI-385	" "	NOPI-385-WR1
NOPI-386	" "	NOPI-386-WR1
NOPI-387	" "	NOPI-387-WR1
NOPI-388	" "	NOPI-388-WR1
NOPI-389	" "	NOPI-389-WR1

Results are kept in a 3 ring binder entitled "Alpha Spectrometry of Nopal I Samples.

2/22/95 JP

The following samples were analyzed for U+Th isotopes by α-spec using procedure on p 59-62

NOPI-390	portion of	NOPI-390-WR1
NOPI-391	"	NOPI-391-WR1
NOPI-399	"	NOPI-399-WR1
NOPI-400	"	NOPI-400-WR1
NOPI-209	"	NOPI-209-GAM1
NOPI-205	"	NOPI-205-GAM1
NOPI-139	"	NOPI-139-GAM1
NOPI-137	"	NOPI-137-GAM1
NOPI-298	"	NOPI-298-GAM1
NOPI-302	"	NOPI-302-GAM1
NOPI-144	"	NOPI-144-GAM1
NOPI-142	"	NOPI-142-GAM1

Results are kept in a 3-ring binder entitled "Alpha Spectrometry of Nopal I Samples."

* Metler AE240 Balance was used to weigh all reagents.

gf 4/7/95 Preparation of reagents for alpha-spec analysis of Wopal I samples

0.05 M EDTA - dissolve 9.31 g of disodium ethylenediaminetetraacetic acid dehydrate (lot #p5518) in 400 ml of H_2O + 4.5 ml of 10 M sodium hydroxide. Dilute solution to 500 ml and add additional 10 M sodium hydroxide dropwise to bring pH to 10.6. Transfer to 500 ml PP bottle + label "0.05 M EDTA".

Cevic hydroxide substrate - Add 1 ml of cerous nitrate containing 5 mg/ml of cerium in 0.1 M nitric acid, 50 ml H_2O, (lot 725181) 1 ml of 30% hydrogen peroxide, and 5 ml of 10 M sodium hydroxide to a 150 ml beaker. Cover beaker with cover glass + boil for 5 min. Pour boiling solution into 450 ml of water + dilute to 500 ml. Transfer to 500 ml PP bottle + label "ceric hydroxide substrate" gf 4/7/95

10 M NaOH - add 40 g NaOH (lot NO 941745A) to 100 ml H_2O + dissolve. Transfer to 125 ml PP bottle labeled "10 M NaOH".

Cerous Floride Substrate - mix 1 ml of 5 mg/ml cerous nitrate with 500 ml of 1 M HCl and add 40 ml of 48% hydrofloric acid (lot 932383). Transfer to 500 ml PP bottle + label "Cerous Floride Substrate".

1 M HCl - Dilute 8.2 ml conc. HCl (lot 945500) to 100 ml in a glass beaker. Transfer to a 250 ml PP bottle + label "1 M HCl".

0.1 M HNO_3 - Dilute 3.15 ml conc. HNO_3 (lot 945563) to 500 ml in a volumetric beaker. Transfer solution to a 500 ml PP bottle + label "0.1 M HNO_3"

Ceron nitrate 5.0 mg/ml Ce
in 0.1 m HNO_3 - add ~~.2020~~ 1613 g gp 4/7/95
ceric nitrate (lot 10294-41-4)
4/7/95 gp to ~~200~~ 100 ml of 0.1 m HNO_3
and dissolve. Transfer to 125 ml PP
bottle & label "Ceron nitrate
5.0 mg/ml Ce in 0.1 m HNO_3"

Ceron nitrate 0.5 mg/ml Ce
in 0.1 m HNO_3 - dilute 10 ml
of 5.0 mg/ml solution of Ce
in 0.1 m HNO_3 with to 100 ml gp 4/7/95
of 0.1 m HNO_3. Transfer solution
to a 125 ml PP bottle &
label "Ceron nitrate 0.5 mg/ml
Ce in 0.1 m HNO_3".

25% hydrazine dehydrochloride -
dissolve 5.25 g of hydrazine
dehydrochloride (Lot No 5341-61-7)
in 50 ml H_2O. Transfer to
125 ml PP bottle and label
"25% hydrazine dehydrochloride".

10% Sodium hydrogen sulfate -
gp 4/7/95 ~~10.0~~ 10.0 g of sodium bisulfate (lot 936825)
was dissolved in 90 g H_2O.
Solution was transferred to a
125 ml PP bottle & labeled
"10% sodium hydrogen sulfate".

05-2-95
OCL

THE FOLLOWING IS A LIST OF REAGENTS ALONG WITH PREPARATION SCHEMES. FOR ALL SOLUTIONS MADE, GLASSWARE USED HAS BEEN CLEANED WITH ALCONOX SOAP, ACID WASHED (NITRIC), AND RINSED WITH ULTRAPURE WATER. ALL REAGENTS WILL BE LABELED IN THE FOLLOWING MANNER:

REAGENT (LOT #)
DATE MADE
PREPARER'S INITIALS
NOTEBOOK # / PAGE #

- 2.0 L OF A 0.1 M HCl WILL BE PREPARED BY MEASURING 16.5 mL OF CONCENTRATED 12.1 M HCl AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 1983.5 mL OF ULTRAPURE H_2O WILL BE ADDED, AND SOLUTION IS MIXED AND LABELED.

- 2.0 L OF A 1 M HCl WILL BE PREPARED BY MEASURING 165.3 mL OF 12.1 M HCl AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 1834.7 mL OF ULTRAPURE H_2O WILL BE ADDED, AND SOLUTION IS MIXED AND LABELED.

- 2.0 L OF A 8 M HCl WILL BE PREPARED BY MEASURING 1322.3 mL OF CONC. 12.1 M HCl AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 677.69 mL OF ULTRAPURE H_2O WILL BE ADDED, AND SOLUTION IS MIXED AND LABELED.

- 2.0 L OF A 9 M HCl WILL BE PREPARED BY MEASURING 1487.6 mL OF CONC. 12.1 M HCl AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 512.4 mL OF ULTRAPURE H_2O WILL BE ADDED, AND SOLUTION IS MIXED AND LABELED.

- 2.0 L OF A 0.1 M HNO_3 WILL BE PREPARED BY MEASURING 12.66 mL OF CONC. 15.8 N HNO_3 AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE, AND SOLUTION IS MIXED AND LABELED. AFTER ADDITION OF 1987.34 mL OF ULTRAPURE H_2O.

- 2.0 L OF A 7 M HNO_3 WILL BE PREPARED BY MEASURING 886.1 mL OF CONC. 15.8 N HNO_3. AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 1113.9 mL OF ULTRAPURE H_2O IS ADDED FOLLOWED BY MIXING AND LABELING.

HNO_3

- 2.0 L OF A 8.0 M ~~HCl~~ SOLUTION
  OCL 05/12/95

WILL BE PREPARED BY ADDING 1012.7 mL OF CONC. 15.8 N HNO_3, AND QUANTITATIVELY TRANSFERRING INTO A CLEANED 2 L ACID BOTTLE. 987.34 mL OF ULTRAPURE H_2O IS ADDED, AND SOLUTION IS MIXED AND LABELED.

* ALL THE ABOVE ACID SOLUTIONS WILL BE USED IN 250/500 mL NALGENE SQUIRT BOTTLES. THE BOTTLES WILL BE LABELED IDENTICAL TO STOCK ACID SOLUTIONS.

- 0.05 M NH_4OH WILL BE PREPARED BY ADDING 3.38 mL OF CONC. NH_4OH (14.8 N) INTO A CLEANED 1 L VOLUMETRIC FLASK. ULTRAPURE H_2O IS ADDED TO 1 L MARK, AND SOLUTION IS MIXED AND LABELED. SOLUTION WILL BE TRANSFERRED INTO A 500/1000 mL SQUIRT BOTTLE.

- 1.0 L OF A 1.0 M $MgCl_2$ WILL BE PREPARED BY ADDING 105.2 g OF SOLID $MgCl_2$ INTO A CLEANED 1 L VOLUMETRIC FLASK. ULTRA PURE H_2O IS ADDED TO 1 L MARK, AND SOLUTION IS MIXED AND LABELED.

HNO_3 LOT # 945565

HCl LOT # 945500

NH_4OH LOT # FL-04-0390

$MgCl_2$ LOT # 940527

05/4/95
OCL

• THE FOLLOWING REAGENTS WILL BE PREPARED. ALL GLASSWARE USED HAS BEEN CLEANED WITH ALCONOX SOAP, ACID WASHED, AND RINSED WITH ULTRAPURE H_2O. LABELING SCHEME IS SAME AS SAMPLES ON PP. 74-76 OF THIS NOTEBOOK.

• TAMM'S REAGENT: APPROXIMATELY 28.4 g OF AMMONIUM OXALATE IS DISSOLVED IN DISTILLED H_2O IN A CLEANED 1L VOLUMETRIC FLASK. 5.25 g OF CITRIC ACID IS ADDED AND DISTILLED H_2O IS ADDED UNTIL SOLUTION VOLUME IS 1L. SOLUTION IS MIXED AND LABELED.

• MORGAN'S REAGENT: 82.04 g OF SODIUM ACETATE IS DISSOLVED IN DISTILLED H_2O IN A 1 L VOLUMETRIC FLASK. pH OF SOLUTION IS ADJUSTED TO 5.0 BY ADDITION OF CONC. ACETIC ACID. SOLUTION IS MIXED AND LABELED.

• 0.3 M SODIUM CITRATE: 88.23 g OF SODIUM CITRATE IS ADDED AND DISSOLVED IN A CLEANED 1 L VOLUMETRIC FLASK. SOLUTION IS MIXED AND LABELED.

* ALL SOLUTIONS WILL BE STORED IN 1L NALGENE BOTTLES.

AMMONIUM OXALATE	LOT #	922654
CITRIC ACID	LOT #	951297
SODIUM ACETATE	LOT #	937077
ACETIC ACID	LOT #	956018
SODIUM CITRATE	LOT #	940621

OCL 10 M NaOH WAS PREPARED BY MEASURING OUT 40.0 g OF NaOH (LOT# 941745A) IN 100 mL VOLUMETRIC FLASK. ULTRAPURE H_2O WAS ADDED TO MARK, AND SOLUTION MIXED AND LABELED. SOLUTION WAS TRANSFERRED INTO A 125 mL NALGENE BOTTLE.

05/8/95
OCL

THE FOLLOWING SAMPLES HAVE BEEN OBTAINED FROM FRACTURE FILL MATERIAL IN WHOLE ROCK SAMPLES. SAMPLES WERE POWDERED IN AGATE MORTAR AND PESTLE.

417 OCL 5/8/95

NOPI - ~~420~~ - WR1
NOPI - 418 - WR1
NOPI - 419 - WR1
NOPI - 420 - WR1
NOPI - 421 - WR1
NOPI - 422 - WR1
NOPI - 423 - WR1
NOPI - 424 - WR1
NOPI - 425 - WR1

A 0.02 M EDTA SOLUTION HAS BEEN PREPARED BY MEASURING OUT 3.72 g OF EDTA IN A 500 mL VOLUMETRIC FLASK. ULTRAPURE H_2O IS ADDED TO MARK. SOLUTION IS MIXED AND TRANSFERRED INTO A LABELED 500 mL NALGENE BOTTLE.

EDTA LOT # 905518

OCL

KINETIC TESTS WILL BE PERFORMED ON SAMPLE NOPI-418-WR1, DUE TO ITS LARGE SAMPLE SIZE. THESE TESTS WILL CONSIST OF PERFORMING SEQUENTIAL PHASE EXTRACTIONS ON THREE MINERAL PHASES IN SAMPLE NOPI-418-WR1; ION EXCHANGEABLE (IE), ADSORBED (AD), AND AMORPHOUS (AM). THE FOLLOWING IS A STEP BY STEP OUTLINE OF THE SEQUENTIAL EXTRACTIONS.

After weighing powdered sample, transfer sample into a 50 mL polypropylene (PP) tube.

Sequential Phase Extractions

Sample powders will undergo a series of phase extractions separating main mineral phases of interest.

I. Ion exchangeable phase:
   a. Add 50 mL of 1M magnesium chloride to powder in centrifuge tube. Shake mixture for 1 hour on wrist shaker.
   b. Centrifuge mixture for 10 minutes at 10000 rpm.
   c. Take out a 3-4 mL aliquot of supernate and transfer into a liquid scintillation vial.
   d. Return original mixture to wrist shaker and continue mixing.

once all kinetic experiments are completed with IE phase, residue is washed with ultrapure water.

II. Adsorbed phase:
   a. To washed residue, 30 mL of Morgan's reagent and 0.3 mL of 0.02 M EDTA are added to residue in 50 mL PP tube.
   b. Shake mixture for six hours on wrist shaker.
   c. Centrifuge for 10 minutes at 10000 rpm and take out a 3-4 mL aliquot of supernate. This solution is placed in a liquid scintillation vial.
   d. Return original mixture to wrist shaker and continue mixing.

once all kinetic measurements are completed with AD phase, residue is washed with ultrapure water.

III. Amorphous phase:
a. To washed residue, add 50 mL of Tamm's reagent to 50 mL PP tube.
b. Mix solution in dark for four hours. To ensure no light hits sample mixture, PP tube is also covered with aluminum foil. Check by opening cap to release any pressure.
c. Centrifuge mixture at 10000 rpm for 10 minutes.
d. Take out a 3-4 mL aliquot of supernate and place in a liquid scintillation vial.
e. Return original mixture to wrist shaker and continue mixing.

THE ABOVE STEPS WILL BE MODIFIED FOR KINETIC STEP MEASUREMENTS. FOR THE ION EXCHANGEABLE PHASE EXTRACTION, RESIDUE WILL BE MIXED AND ALIQUOTS OF SUPERNATE WILL BE TAKEN OUT AT 1 HOUR, 2 HOURS, 4 HOURS, 6 HOURS, AND 8 HOURS FROM STARTING MIXING TIME. EACH SOLUTION WILL THEN FOLLOW OUTLINE Ib.
FOR THE ADSORBED PHASE EXTRACTION, RESIDUE WILL BE MIXED AND ALIQUOTS OF SUPERNATE WILL BE TAKEN OUT @ 6, 8, AND 11 HOURS FROM STARTING MIXING TIME. SOLUTION CAN THEN START

FROM OUTLINE IIc
FOR AMORPHOUS PHASE EXTRACTION, RESIDUE WILL MIX AND ALIQUOTS WILL BE TAKEN OUT @ 4, 6, 8, AND 10 HOURS FROM STARTING MIXING TIME. EACH SOLUTION WILL THEN FOLLOW FROM OUTLINE IIIc. ~~* STEP e WILL BE OMITTED~~ * OCh 5/9/95

ALL SOLUTIONS WILL THEN BE READY FOR LIQUID SCINTILLATION MEASUREMENTS.

05/11/95
~~05/10/95~~
~~05/9/95~~ OCL 05/10/95
OCL

KINETIC EXPERIMENTS HAVE BEGUN ON SAMPLE NOPI-418-WR1. SAMPLE WAS STORED IN A 50 mL pp tube AND 50 mL OF 1 M $MgCl_2$ WAS ADDED. MOST OF THE SOLUTION REACHED NEAR TOP OF TUBE. THIS MAY CAUSE PROBLEMS SINCE MIXTURE MAY NOT MIX AS WELL IF LESS SOLUTION REMAINED IN pp TUBE. INCLINATION OF WRISTSHAKER WAS SET TO 10 MAX TO ENSURE MIXTURE AGITATION. SINCE TWO OTHER EXTRACTION STEPS REQUIRE 40 mL AND 50 mL OF SOLUTION, IT MAY BE NECESSARY TO PURCHASE LARGER TUBES (MUST ALSO FIT IN CENTRIFUGE).

SAMPLE MASS:
1.0171 g

- 1 HOUR MIXING 4.0 mL ALIQUOT IN LABELED 20 mL LIQUID SCINTILLATION VIAL (NOPI-418, KINETICS, I 1hr.)

- 2 HOUR MIXING 4.0 mL ALIQUOT IN LABELED 20 mL LIQUID SCINTILLATION VIAL (NOPI-418, KINETICS, I 2hrs)

OCL 5/11/95

- 4 HOUR MIXING 4.0 mL ALIQUOT IN LABELED 20 mL LIQUID SCINTILLATION VIAL (NOPI-418, KINETICS, I 4hrs) OCL 5/11/95

- 6 HOUR MIXING 4.0 mL ALIQUOT IN LABELED 20 mL LIQUID SCINTILLATION VIAL (NOPI-418-KINETICS, I 6 HOURS)

05/12/95
OCL

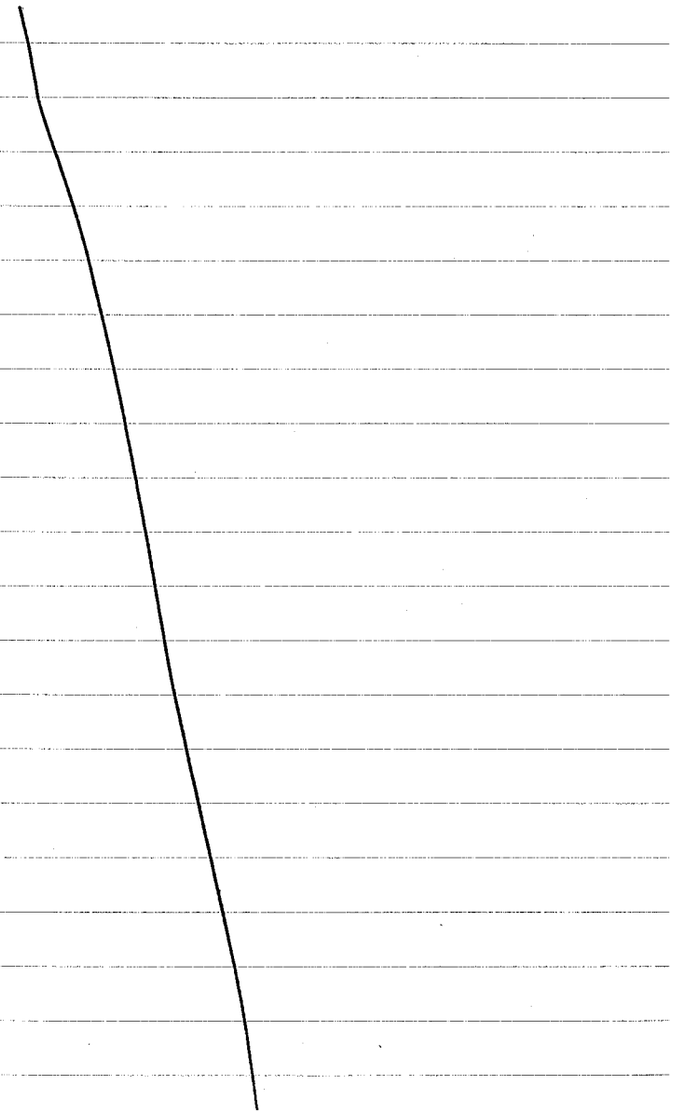
KINETIC EXPERIMENTS HAVE CONTINUED. RESIDUE FROM 6 HOUR MIXING WAS STORED IN PP TUBE OVERNIGHT. REMAINING SUPERNATE SOLUTION WAS ADDED TO RESIDUE AND SOLUTION MIXTURE MIXED FOR ADDITIONAL 2 HOURS.

- 8 HOUR MIXING 4.0 mL ALIQUOT IN LABELED 20 mL LIQUID SCINTILLATION VIAL (NOPI-418, KINETICS I - 8hrs)

RESIDUE IS WASHED WITH ULTRAPURE H_2O. SOLUTION IS WASHED AGAIN AND CENTRIFUGED. SUPERNATE IS SAVED IN 100 mL NALGENE BOTTLE. REMAINING WASHES ARE ALSO TRANSFERRED INTO 100 mL NALGENE BOTTLE. RESIDUE, AFTER WASHING, IS STORED IN ORIGINAL 50 mL PP TUBE.

05/15/95
OCL

RESIDUE WILL NOW UNDERGO EXTRACTION OF ADSORBED PHASE. TO RESIDUE, 30 mL OF MORGAN'S SOLUTION AND 0.3 mL OF 0.02 M EDTA ARE ADDED. SHAKINGS WILL CONSIST OF 6, 8, AND 11 HOUR INTERVALS. STARTING MIXING TIME WAS AT 12:54 PM.



05/16/95
OCL

OCL 5/16/95

CONTINUATION OF [redacted] KINETIC EXPERIMENTS ON PHASE II EXTRACTION OF ADSORBED PHASE CONCLUDES TODAY. ALIQUOTS @ 6, 8, AND 11 HOURS FROM STARTING MIXING TIME WILL BE PLACED IN 20 mL LIQUID SCINTILLATION VIALS AND LABELED IN SIMILAR FASHION AS ON PAGES 81-86 OF THIS NOTEBOOK

ONCE AD PHASE EXTRACTIONS ARE COMPLETED, PREPARATION FOR FOLLOWING EXTRACTION WILL BEGIN. THE AMORPHOUS PHASE EXTRACTION REQUIRES MIXING IN THE ABSENCE OF LIGHT. FOR THIS STEP, WRISTSHAKER WILL BE MOVED TO ANOTHER ROOM WHICH ENSURES VERY LITTLE LIGHT. IN ADDITION, PP TUBE WILL BE COVERED WITH ALUMINUM FOIL.

SAVED SUPERNATE + WASHINGS FROM AD PHASE EXTRACTIONS TRANSFERRED INTO LABELED NALGENE BOTTLE AND STORED ON SHELVES IN LABORATORY.

05/18/95
OCL

PHASE EXTRACTION OF ~~CRYSTALLINE Fe-OXIDE~~ OCL 5/19 AMORPHOUS PHASE WILL BE CONDUCTED IN LAB ROOM L006 IN BLDG. 57. THE LAB DOOR WINDOWS ARE COVERED ENSURING A COMPLETELY DARK ROOM. ALUMINUM FOIL IS USED TO COVER PP TUBE WHILE MIXING @ 4, 6, 8 AND 10 HOUR INTERVALS.

AMORPHOUS 4 HOUR MIXING WAS CENTRIFUGED AND 4.0 mL TRANSFERRED OF SUPERNATE WAS EXTRACTED AND ~~STORED~~ OCL 5/18/95 INTO A 20 mL LABELED LIQUID SCINTILLATION VIAL. (NOPI-418-KINETICS-III 4 hrs)

OCL

FOLLOWING SAMPLES ARE DRIED IN THERMOLYNE OVEN @ 110°C FOR 24 HOURS. THEY ARE COOLED IN DESSICATOR AND STORED UNTIL SEQUENTIAL EXTRACTIONS BEGIN.

NOPI - 419	NOPI - 421
NOPI - 424	NOPI - 422
NOPI - 423	NOPI - 425

05/19/95
OCL

6 AND 8 HOUR ALIQUOTS OF PHASE III EXTRACTION KINETIC EXPERIMENTS ARE COMPLETE. SUPERNATES ~~ARE~~ OCL 5/19 WERE TRANSFERRED INTO 20 mL LIQUID SCINTILLATION VIALS FOR MEASUREMENTS.

12 HOUR MIXING ALIQUOT OF PHASE III HAS BEEN COMPLETED. A TOTAL OF 12, 4.0 mL SUPERNATE SOLUTIONS IN 20 mL LIQUID SCINTILLATION VIALS ARE READY FOR MEASUREMENTS.

05/22/95

KINETIC EXPERIMENT PHASE EXTRACTIONS ONCE COMPLETE WERE MEASURED ON LIQUID SCINTILLATOR ON 05/19/95. ALL 4 mL ALIQUOTS WERE MIXED WITH 15.0 mL OF A COCKTAIL SOLUTION (ULTIMA GOLD) AND MIXED THOROUGHLY. THEY WERE THEN PLACED IN DEVICE AND MEASURED OVER WEEKEND. (TRAY)

05/23/95
OCL

1 M SODIUM BICARBONATE SOLUTION WAS PREPARED BY ADDING 22.00 g OF SODIUM BICARBONATE TO A CLEANED 250 mL VOLUMETRIC FLASK. ULTRAPURE H_2O IS ADDED TO MARK AND SOLUTION MIXED. SOLUTION THEN TRANSFERRED INTO 250/500 mL NALGENE BOTTLE.

05/25/95
OCL

SAVED SUPERNATE FROM KINETICS EXPERIMENT PHASE I EXTRACTION WILL BE PREPARED FOR U/Th COLUMN SEPARATION. A QUANTITATIVE AMOUNT OF U SPIKE WILL BE ADDED TO SOLUTION AND ALLOWED TO EQUILIBRATE:

SPIKE # 25B REF. DATE 1/22/93
REF. ACTIVITY 204.88 pCi/g SPIKE WEIGHT 0.7162

HALF OF SOLUTION IS STORED IN PP BOTTLE AS A SPLIT, AND OTHER HALF TRANSFERRED INTO TEFLON BEAKER. SOLUTION IS GENTLY HEATED TO DRYNESS. 2 mL OF CONC. HNO_3 AND 2 mL $HClO_4$ ARE ADDED, AND SOLUTION IS HEATED UNTIL PERCHLORIC FUMES.

* SPLIT (spiked) IS STORED IN SAME PP bottle
* SUPERNATE WAS FILTER PASSED THROUGH BEFORE SPIKE WAS ADDED.

05/26/95
OCL

NOPI-428-WR1 PHASE I EXTRACTIONS OCD IS PREPARED FOR U/Th COLUMN SEPARATION. U AND Th SEPARATES ARE DRIED OVER HOT PLATE FOR PLATING AND α COUNTING.

05/29/95

SUPERNATE NOPI-418 - KINETICS II WILL BE PREPARED FOR U/Th COLUMN SEPARATION IN A SIMILAR FASHION TO SAMPLE PROCEDURE ON pp. 94-95. THE FOLLOWING IS A SUMMARY OF THE AMOUNT AND SPIKE # USED.

SPIKE # 25B REF. DATE 1/22/93

REF. ACTIVITY 204.88 pCi/g SPIKE ~~DATE~~ WT. OCS 5/29/95 0.7353 g

05/30/95

SUPERNATE NOPI-418 - KINETICS II IS READY FOR U/Th PLATING. PHASE III SUPERNATE WILL ALSO BE PREPARED FOR U/Th PLATING AND ∝ COUNTING. FOLLOWING IS A SUMMARY OF SPIKE # AND AMOUNT USED (g).

SPIKE # 25B REF. DATE 1/22/93

REF. ACTIVITY 204.88 pCi/g SPIKE WT 1.3809

SOLUTION IS DRIED OVER HOT PLATE FOLLOWED BY ADDITIONS OF 2 mL EACH OF PERCHLORIC AND NITRIC ACIDS. RESIDUE IS DISSOLVED IN ACIDS AND SOLUTION HEATED UNTIL PERCHLORIC FUMES CEASE.

05/31/95
OCL

RESIDUE REMAINING FROM "AM" PHASE WILL UNDERGO AN EXTRACTION TO RETRIEVE CRYSTALLINE IRON OXIDES. TO RESIDUE, 40 mL OF 0.3 M SODIUM CITRATE AND 5 mL OF 1.0 M SODIUM BICARBONATE ARE ADDED. SOLUTION IS MIXED AND PLACED IN HOT H_2O BATH. ~1.0 g OF SODIUM DITHIONITE ($H_2S_2O_4$) IS ADDED AND MIXED FOR 5 MINUTES. TWO ADDITIONAL 1.0 g PORTIONS OF $H_2S_2O_4$ ARE ADDED WITH MIXING. THIS STEP IS FOLLOWED by ADDITIONS OF 10 mL EACH OF ACETONE (GC RESOLVE) AND A SATURATED NaCl SOLUTION. SOLUTION IS MIXED, CENTRIFUGED, AND SUPERNATE SAVED. RESIDUE REMAINING SHOULD BE GREY / OFF WHITE. IF NOT, REPEAT EXTRACTION.

* 4 TIMES RESIDUE WAS EXTRACTED FOR SAMPLE NOPI-418-WR1

SUPERNATE IS STORED IN LABELED 500 mL pp BOTTLE. SOLUTION IS SPIKED AND PREPARED FOR U/Th COLUMN SEPARATION.

ON FOLLOWING PAGE IS A SUMMARY OF SPIKE # AND AMOUNT USED ON "CR" PHASE OF NOPI-418-WR1.

SPIKE # 25A
REFERENCE DATE 1/22/93
REF. ACTIVITY 2.051 nCi/g
SPIKE WT 3.0098 g

06/5/95
OCL

NOPI-423-WR1

0.4459 g

1.0 M $MgCl_2$ SOLUTION IS PREPARED by ADDING 203.31 g OF $MgCl_2$ (LOT # 940527) INTO A CLEANED 1.0 L VOLUMETRIC FLASK. SOLUTION IS MIXED AND TRANSFERRED INTO A LABELED 6/15 OCL 1.0L NALGENE BOTTLE.

500 mL OF CEROUS FLOURIDE SUBSTRATE WAS PREPARED IN SAME WAY OUTLINED ON p. 71 OF THIS NOTEBOOK.

500 mL OF CERIC HYDROXIDE SUBSTRATE WAS PREPARED IN SAME WAY OUTLINED ON p. 70 OF THIS NOTEBOOK.

06/6/95
OCL

U AND Th SEPARATES FOR 4 PHASES OF NOPI-418-WR1 HAVE BEEN PREPARED FOR αC SPECTROMETRY.

SAMPLE NOPI-423-WR1 IS READY FOR SEQUENTIAL PHASE EXTRACTIONS. FOLLOWING IS THE DESIGNATED METHOD.

Sequential Phase Extractions of 13m transect samples from NOPAL I Uranium Deposit

Objective: Determine Uranium and Thorium content along with Uranium and Thorium isotopic ratios in different mineral phases of samples collected from the NOPAL I natural analog site.

Method: Sequential phase extraction technique, Uranium/Thorium column separation, deposition on filters, and alpha counting.

Equipment:

- ALPHA-KING Multi-Channel Alpha Spectrometer
  - 576A dual spectrometer or 676 single spectrometer with ion implanted silicon particle detectors
  - Model 920 multichannel buffer
  - APLHAMAT analysis software for acquisition control
  - MAESTRO II multichannel emulation software for analysis of data
- Thermolyne drying oven
- Ohaus analytical balance
- Centrifuge
- SI Vortex Genie hand homogenizer
- Burrell wrist shaker
- Hot plate
- infrared lamp
- perchloric fume hood
- agate mortar and pestle

Materials and Supplies:

- necessary glassware (e.g., petri dishes, beakers, volumetric flasks, etc...)
- necessary plastic ware (e.g., pp bottles, weighing boats, beakers)
- 50 mL polypropylene centrifuge tubes
- Eppendorf pipets and tips
- Teflon dish and lid for teflon bomb
- filters and filter funnels
- BIO-RAD 1.5 cm diameter columns
- stainless steel 1 inch planchettes
- 25mm membrane filters
- 50 mL polysulfone filter funnel

Reagents:

- concentrated HNO_3, $HClO_4$, HCl, HF, acetic acid, and ammonia
- 0.1 M, 1 M, 8 M, 9 M HCl
- 0.1 M, 7 M, 8 M HNO_3
- 0.05 M EDTA*
- 0.05 M ammonia
- nanopure water
- Cerous nitrate (0.5mg/mL) substrate*
- 25% hydrazine dihydrochloride
- 10 M NaOH
- 80% ethanol
- Ceric hydroxide (10 µg Ce/mL) substrate*
- 10% sodium hydrogen sulfate
- 20% titanium trichloride
- cerous fluoride (~10 µg Ce/mL) substrate
- 0.3 M sodium citrate
- 1 M $NaHCO_3$
- saturated NaCl solution
- acetone
- $^{232}U/^{228}Th$ spike
- sodium dithionite ($Na_2S_2O_4$)
- 1 M magnesium chloride
- Tamm's reagent: 28.4 g ammonium oxalate (MW = 142.1) dissolved in distilled water and added with 5.25 g citric acid (MW = 210.14) dissolved in distilled water until total volume of solution is 1 L.
- Morgan's reagent: 82.04 g sodium acetate dissolved in distilled water with pH adjusted to 5.0 using concentrated acetic acid.
- Fe carrier solution: 72.3 g ferric nitrate dissolved in distilled water to a volume of 1 L.
- BIO-RAD anion exchange resin (AG 1x8, 100-200 mesh chloride form)

Procedure:

Sample I.D. # _____
Date _____

Sample Preparation: Sample is obtained from bulk rock sample, crushed in an agate mortar and pestle, dried in oven, and cooled in dessicator.

Sample Mass _____ g

After weighing, powdered sample is transferred into 50 mL polypropylene (PP) tube.

Sequential Phase Extractions: Sample powders will undergo a series of phase extractions, separating the five main mineral phases of interest.

I. *Ion exchangeable phase:*

a. Add 50 mL of 1 M magesium chloride to powder in centrifuge tube. Shake for 1 hour on wristshaker.
b. Centrifuge mixture at 10000 rpm for 10 minutes.
c. Transfer supernate into a clean and labeled PP bottle. Label bottle as "IE."
d. Wash residue in PP tube with 30 mL of ultrapure water. Centrifuge for 10 minutes and decant supernate into labeled PP bottle.
e. Add appropriate amount of spike to saved supernate and record spike weight (~10% total U in sample). Allow solution to equilibrate. Transfer half of solution into a cleaned teflon beaker.

Spike # _____ Reference Date _____
Reference Activity _____ Spike weight _____ g

f. Evaporate solution on hot plate. Add 2 mL each of concetrated nitric and perchloric acids and evaporate solution until perchloric fumes.
g. Add 2 mL of concentrated HCL to dissolve residue and 8 mL of ultrapure water to dilute to 2 M.
h. Transfer solution to a clean 50 mLcentrifuge tube. Add Fe carrier and allow to equilibrate. Add concentrated ammonia to coprecipitate actinides. Centrifuge and decant supernate. Wash precipitate at least twice with 0.05 M ammonia.
i. Dissolve precipitate in 3 mL of concentrated HCl and then add 1 mL of ultrapure water to dilute to 9M.

II. *Adsorbed phase:*

a. Add 30 mL of Morgan's reagent and .03 mL of 0.02 M EDTA to residue in 50 mL PP tube.
b. Shake mixture for six hours on wristshaker.
c. Centrifuge mixture at 10000 rpm for 10 minutes.
d. Label a cleaned PP bottle as "AD" and transfer supernate into this bottle.
e. To remaining residue, wash with 30 mL of ultrapure water and centrifuge for 10 minutes. Transfer supernate to the labeled PP bottle.
f. To supernate solution, add radioactive spike and record spike weight. Follow with addition of 0.5 mL of Fe carrier solution and leave overnight to equilibrate.

Spike # _____ Reference Date _____
Reference Activity _____ Spike weight _____ g

g. Transfer half of this solution to a cleaned teflon beaker. The supernate in the bottle will be saved.
h. Evaporate supernate in teflon beaker to dryness on hotplate.
i. Add 2 mL each of concentrated nitric and perchloric acids. Gently heat solution until color changes to a pale straw or water white color. Cover to reduce evaporation.
j. Evaporate to dryness on hot plate.
k. Add 5 mL each of 8 M HCl and ultrapure water to teflon beaker to dissolve residue. Follow by transferring solution into a cleaned 50 mL PP tube.
l. Add concentrated ammonia to coprecipitate actinides. Centrifuge and decant supernate. Wash precipitate at least twice with 0.05 M ammonia.
m. Dissolve precipitate in 3 mL of concentrated HCl and then add 1 mL of ultrapure water to dilute to 9M.

III. *Amorphous phase*

a. Add 50 mL of Tamm's reagent to residue in 50 mL PP tube.
b. Solution mixture must be shaken in dark for four hours. This can be accomplished by covering PP tube with aluminum foil and shaking solution in a dark room. Check solution hourly by opening cap and releasing any excess pressure.
c. Centrifuge mixture at 10000 rpm for 10 minutes
d. Label a cleaned PP bottle as "AM" and transfer supernate into this bottle.
e. Add 15 mL of oxalate solution to residue and shake for 30 minutes. Centrifuge at 10000 rpm for 10 minutes and add supernate to lableled PP bottle.
f. To remaining residue, wash with 30 mL of ultrapure water and centrifuge for 10 minutes. Transfer supernate to the labeled PP bottle.
g. To supernate solution, add radioactive spike and record spike weight.

Spike # _____ Reference Date _____
Reference Activity _____ Spike weight _____ g

h. Transfer half of this solution to a cleaned teflon beaker. The supernate in the bottle will be saved.
i. Evaporate supernate in teflon beaker to dryness on hotplate.
j. Add 2 mL each of concentrated nitric and perchloric acids. Gently heat solution until color changes to a pale straw or water white color. Cover to reduce evaporation.
k. Evaporate to dryness on hot plate.
l. Add 5 mL each of 8 M HCl and ultrapure water to teflon beaker to dissolve residue. Follow by transferring solution into a cleaned 50 mL PP tube.
m. Add concentrated ammonia to coprecipitate actinides. Centrifuge and decant supernate. Wash precipitate at least twice with 0.05 M ammonia.
n. Dissolve precipitate in 3 mL of concentrated HCl and then add 1 mL of ultrapure water to dilute to 9M.

IV. *Crystalline iron oxides*

a. Add 40 mL of 0.3 M sodium citrate and 5 mL of 1 M sodium bicarbonate to residue in 50 mL centrifuge tube.
b. Place tube into a water bath at 75-80 degrees.
c. Add 1 g of solid sodium dithionite to centrifuge tube and mix for 1 minute. Continue occasional mixing for five minutes.
d. Repeat step c twice.
e. Add 10 mL of saturated sodium chloride and 10 mL of acetone into PP tube. Mix solution, place in warm water bath, and centrifuge for five minutes at 3000 rpm.
d. Decant supernate into a cleaned PP bottle labeled "CR." Observe color of residue. If not grey or off white, residue still contains Fe-oxides and extraction steps must be repeated.
e. Add radioactive spike and record spike weight. Allow solution to equilibrate overnight.

Spike # _____ Reference Date _____
Reference Activity _____ Spike weight _____ g

f. Transer half of solution into a clean teflon beaker. Evaporate solution in teflon beaker to dryness and save the other fraction in PP bottle.
g. Add concentrated nitric until bubbling ceases. Follow by evaporating solution on hotplate.
h. Follow steps k-l from extraction II.

V. Final residue is spiked and dissolved by acid digestion in microwave. Procedure is outlined below.

a. Consult the CEM Microwave Sample Preparation Applications Manual for the microwave sample preparation note for the type of sample being dissolved. This discusses the amount of sample and reagents to be used, and program parameters to be entered for microwave digestion.

Application note _____

b. Record the dry weight of the sample and place in teflon PFA vessel.

Sample dry weight _____ g

c. Quantitatively add a known amount of radioactive spike to the PFA vessel.

Spike # _____ Reference Date _____

Reference Activity _____ Spike weight _____ g

d. Add reagents to vessel and record volumes used.

Reagents	Volume (mL)
_____	_____
_____	_____
_____	_____
_____	_____

e. Seal vessel, place on turntable in microwave, enter and run digestion program. If after digestion, sample is not completely dissolved, repeat this step.
d. Quantitatively transfer half of the sample into a cleaned teflon beaker washing sides of PFA vessel with ultrapure water.
e. Split sample into two parts; half is saved and stored in a labeled PP bottle while the other half is kept in teflon beaker.
f. To sample solution in teflon beaker, add 1 mL of perchloric acid. Evaporate over hot plate until perchloric fumes.
g. Add 2 mL of centrated HCl to dissolve residue and 8 mL of ultrapure water. Transfer solution into a 50 mL PP tube.
h. Add 0.5 mL of Fe carrier and allow to equilibrate. Add concentrated ammonia to solution and precipitate out actinides. Decant out supernate and save precipitate. Wash precipitate twice with .05 M ammonia.
i. Dissolve precipitate in 3 mL of concentrated HCl. Add 1 mL of ultrapure water to dilute to 9M.

Column Separation:

Resin: BIO-RAD Anion Exchange Resin AG 1-X8 100-200 mesh chloride form.
(Lot # _____)

Main Column (Biorad 1.5 cm diameter column with 10 cm resin; prewash with 4-5 column vols 9M HCl)

Load sample in 9M HCl and allow to drain	___
Wash 3 volumes (~35 ml) 9M HCl --> Th	___ ___ ___
Elute 4 volumes (~50 ml) 0.1M HCl --> U and Fe	___ ___ ___ ___

Separation Date _____

***Note: May work on U and Th fractions simultaneously from this point on.

Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Heat Th fraction to evaporate HCl	___
Add ~5 ml conc HNO_3 to dissolve residue	___
Add equal vol (~5 ml) DI water so soln 8M HNO_3	___
Load onto column in 8M HNO_3	___
Wash 3-4 column vols 8M HNO_3	___ ___ ___ ___
Elute 4-5 column vols 9M HCl --> Th	___ ___ ___ ___

Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Evaoprate U fraction to near dryness	___
Pick up in about 2 ml conc HNO_3	___
Dilute with about 2 ml DI water to approx 8M HNO_3	___
Load onto column in 8M HNO_3	___
Wash 2 vols (8-10 ml) 8M HNO_3 --> Fe	___ ___
Elute 4-5 vols 0.1M HCl --> U	___ ___ ___ ___

Source Preparation:

Thorium - OH^- precipitation onto filter

1. Evaporate solution containing Th to near dryness.
2. Add 6 ml 0.05 M EDTA soln to dissolve residue, transfer soln to a 50 ml PP centrifuge tube and place tube in boiling water for a few minutes.
3. Add 100 µl purified cerous nitrate (0.5 mg/ml Ce) to precipitate hydroxides. Mix, add

All results of sequental extrations will be kept in a 3-ring binder entitled "Sequential Extrations Nopal I samples".

2 drops 25% hydrazine dihydrochloride, and 2 ml of 10M NaOH.

4. Place tube in boiling water bath for 10 minutes, remove, and place tube in cold water for 10 minutes to ensure complete precipitation
5. Wet a 25 mm membrane filter with 80% ethanol and place in a 50 ml polysulfone filter funnel.
6. Shake bottle of substrate suspension (ceric hydroxide containing 10 µg Ce/ml) vigorously and draw 2 consecutive 5 ml portions through the filter with full suction. Allow each portion to suck dry for 10-15 sec.
7. Without interrupting suction, pour the sample into the filter chimney and allow to suck dry.
8. While the sample is still filtering, add 0.5 ml 10M NaOH to the sample tube and about 5 ml ultrapure water down the sides of the tube. After the sample has sucked dry, swirl the wash solution around the sides and add it to the filter chimney.
9. Wash tube, filter chimney, precipitate, and filter with three consecutive 5 ml portions of 80% ethanol.
10. Suck filter dry for about 15 sec and remove the chimney and filter carefully without interrupting the suction. Transfer filter to a plastic container and at low temperature (~60°C).
11. Glue to tape the dry filter onto a 1 inch stainless steel planchet and count.

Th Counting Date_____

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 100 µl puriified cerous nitrate (0.5 mg/ml Ce, 50 µg of Ce carrier) and evaporate the solution until completely dry and no more fumes are given off.
2. Add 2 ml of 1M HCl to the beaker containing the purified U fraction and heat gently to dissolve the sodium hydrogen sulfate cake and any possible insoluble double salts with the Ce carrier.
3. Transfer solution to a 50 ml polycarbonate centrifuge tube with two more 2 ml portions of 1M HCl.
4. Add 2 drops of 20% titanium trichloride which should produce a strong violet color. If not, iron is probably present and a few more drops of titanium trichloride must be added to produce a permanent violet color or reduction and precipitation of U will be incomplete.
5. Add 0.5 ml (10 drops) of 48% HF. The violet color dissappears and any slight turbidity should clear up.
6. Mix thoroughly and allow solution to stand for 30 min in a cold water bath to obtain complete precipitation of cerous and uranous fluorides.
7. Place the tube in an ultrasonic bath for 1 min to disperse the precipitate.
8. Mount the precipitate on a 25 mm membrane filter previously treated with two 5 ml portions of cerous fluoride substrate as described above.

9. After sucking the precipitate dry, wash with 5 ml of water containing 2 drops of 48% HF and then with 80% ethanol.
10. Dry and analyze as described above.

U Counting Date_____

07/7/95
OCL

10% SODIUM HYDROGEN SULFATE WAS PREPARED BY DISSOLVING 10.0 g OF SODIUM BISULFATE (LOT # 930825) IN 90.0 g OF ULTRAPURE H_2O. SOLUTION WAS MIXED AND STORED IN 125 mL PP BOTTLE.

2.0L OF A 9M HCl SOLUTION WAS PREPARED IN SIMILAR FASHION AS OUTLINED ON PAGE 75 OF THIS NOTEBOOK

07/18/95
OCL

SPLITS FROM "AD" AND "CR" PHASE WILL BE USED TO PREPARE ADDITIONAL SAMPLES FOR OC SPEC. ANALYSIS.

SAMPLES NOPI-419 AND NOPI-425 WILL BE PREPARED FOR THE SEQUENTIAL PHASE EXTRACTION TECHNIQUE.

12/8/95 gp

Sequential extractions of Nopal I samples.

Fracture-infilling materials from samples collected along the 13.5 m N fracture were separated from bulk specimens.

Samples included:
NOPI-417
NOPI-418
NOPI-420
NOPI-421
NOPI-423
NOPI-425

The collected materials were powdered in a SPEX mixer mill using a tungsten carbide vial. The powdered specimens were placed in plastic vials and labeled with the sample number shown ~~above~~. gp 12/8/95 on next page.

Sample #s of powders.

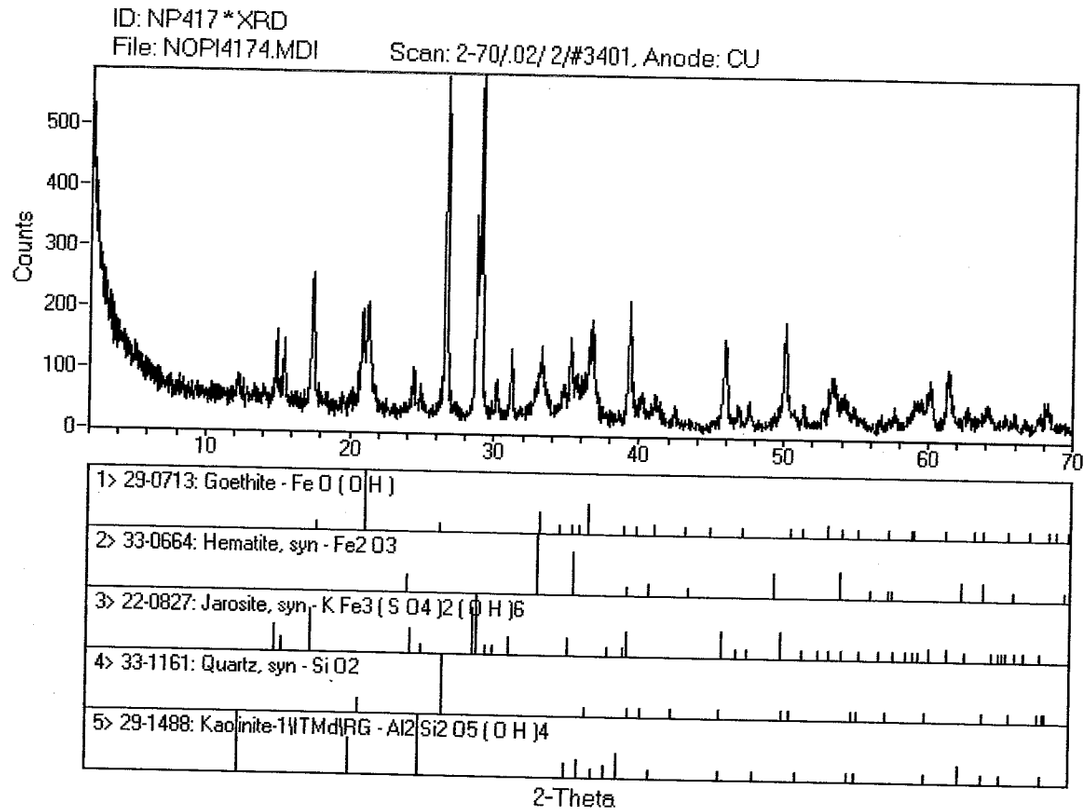
NOPI*417*PWD
NOPI*418*PWD
NOPI*420*PWD
NOPI*421*PWD
NOPI*423*PWD
NOPI*425*PWD

Portions of these powders were mounted in glass holders for XRD analysis in Div 06 using the Siemans D500 X-ray diffractometer with Datalogg. XRD pattern were analyzed using MDI Jade software to determine mineralogy.

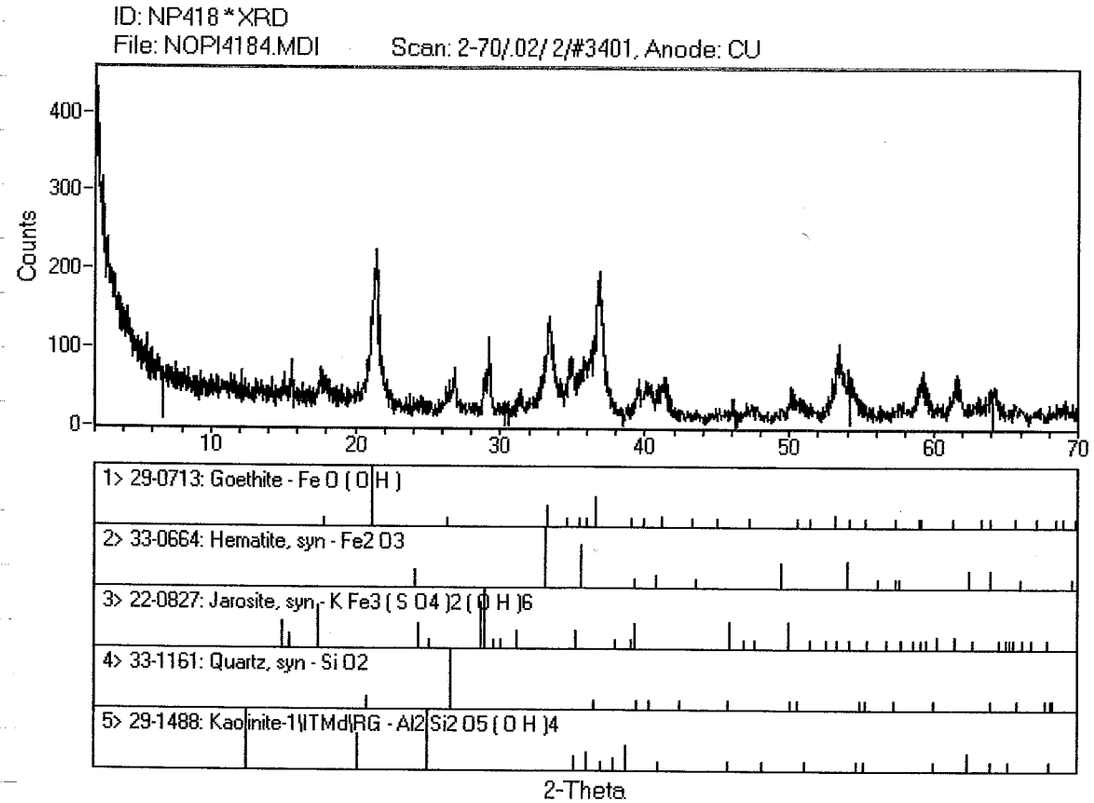
Analyses are shown on following pages.

After analysis powders were returned to their respective plastic vials.

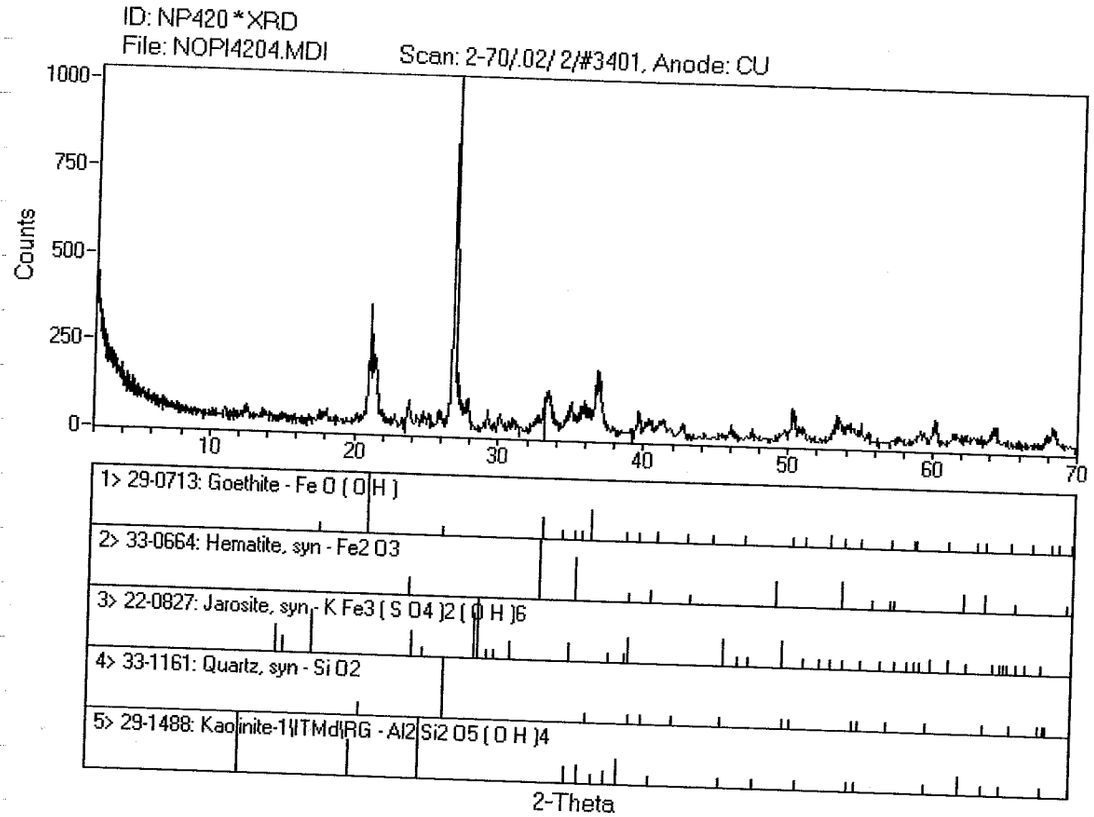
XRD pattern of 13.5 m N samples.



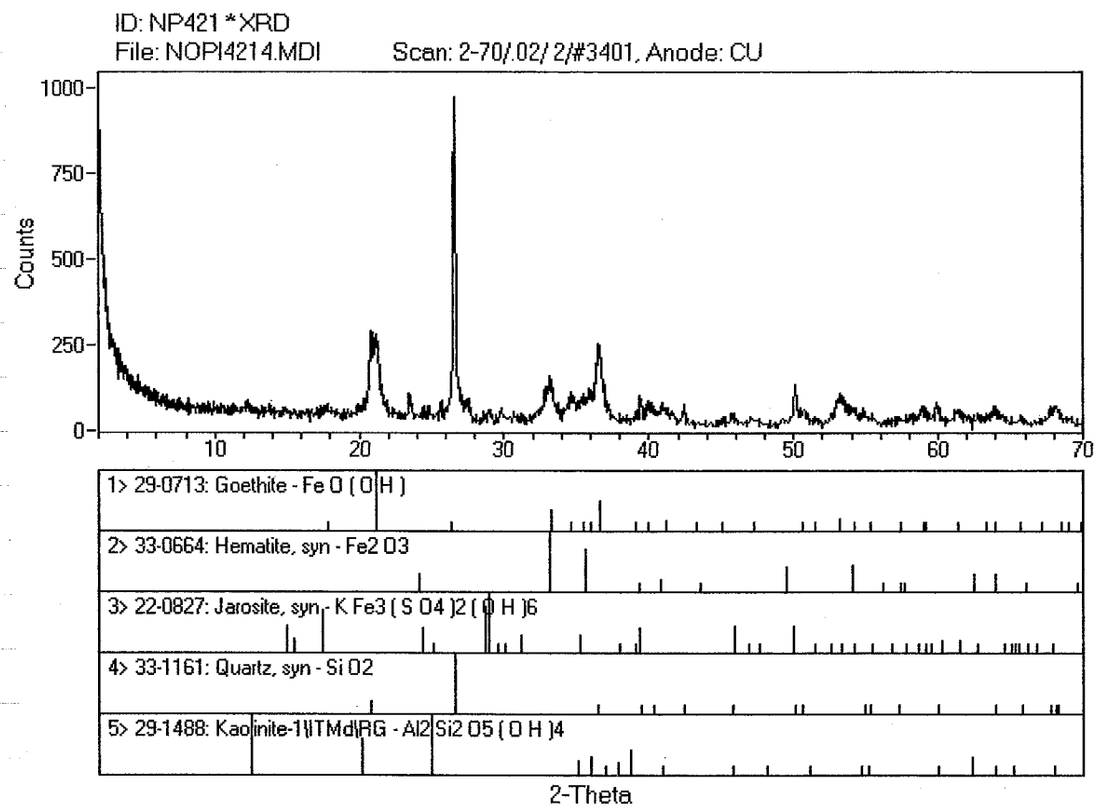
NOPI*417*PWD



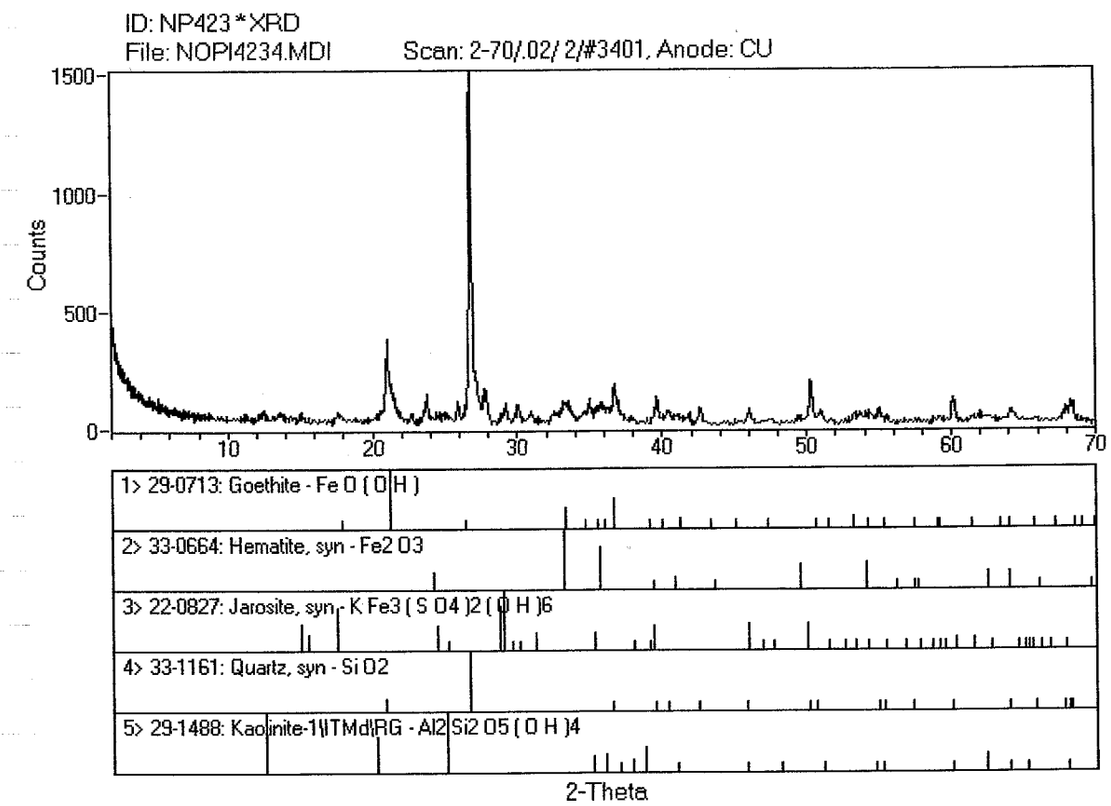
NOPI*418*PWD



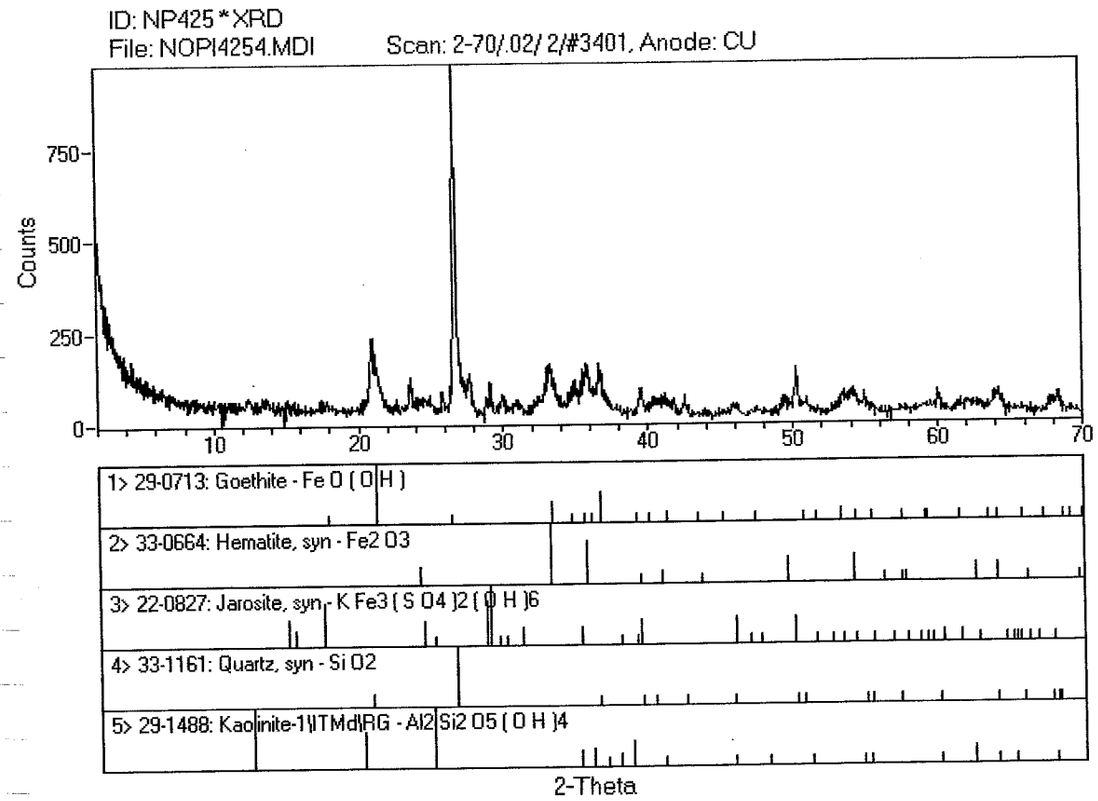
NOPI*420*PWD


ID: NP421 * XRD
File: NOPI4214.MDI
Scan: 2-70/.02/ 2/#3401, Anode: CU
Counts
1000
750
500
250
0
10
20
30
40
50
60
70
1> 29-0713: Goethite - Fe O (O H)
2> 33-0664: Hematite, syn - Fe2 O3
3> 22-0827: Jarosite, syn - K Fe3 (S O4)2 (O H)6
4> 33-1161: Quartz, syn - Si O2
5> 29-1488: Kaolinite-1\TMd\RG - Al2 Si2 O5 (O H)4
2-Theta

NOPI#421 * PWD



NOPI*423*PWD



NOPI*425*PWD

12/11/95 JP

Portions of the following samples were analyzed for U+Th isotopes by α-spec using procedure on p 59-62.

NOPI-417-PWD
NOPI-418-PWD
NOPI-490-PWD
NOPI-451-PWD
NOPI-423-PWD
NOPI-425-PWD

Results are kept in a 3-ring binder entitled "Alpha Spectrometry of NopalI Samples".

12/18/95 JP

Sequential extraction procedure.

The procedure to be used for sequential extraction of U+Th from phases in Nopal rock samples is presented on the following pages.

SEQUENTIAL PHASE EXTRACTIONS

Objective: determine U and Th content and activity ratios in different phases in samples from Nopal I

Method: sequential phase extraction; alpha spectrometry

Equipment: -EG&G Ortec alpha spectrometry system
576A dual or 676 single chamber detectors
Model 920 multichannel buffer
ALPHAMAT analysis software for acquisition control
MAESTRO II software for data analysis
-perchloric fume hood
-drying oven (Blue M)
-analytical balance
-Fisher 21K Marathon centrifuge
-SI Vortex Genie hand homogenizer
-Burrell wrist shaker
-Corning hot plates
-Infrared lamp
-CEM Model MDS-2000 microwave digestion system
-Teflon PFA vessels
-ultrasonic bath

Materials and Supplies:
-necessary glassware (e.g., beakers, flasks, etc...)
-necessary platicware (e.g., PP bottles, beakers, etc...)
-50 ml polypropylene and teflon centrifuge tubes
-Eppendorf pipets and tips
-Teflon beakers
-filters and filter funnels
-Bio-Rad columns 1.5 cm diameter
-Bio-Rad columns 0.7 cm diameter
-Bio-Rad anion exchange resin AG 1-X8 100-200 mesh
-$^{232}U/^{228}Th$ spike solutions prepared previously
-weighing paper and boats
-PP bottles
-1 inch stainless steel planchets
-25 mm membrane filters
-50 ml polysulfone filter funnel

Reagents: -Conc HCl, $HClO_4$, NH_4OH, HNO_3, HF, acetic acid, ammonia
-9M, 0.1M, 1M, 8M HCl
-0.1M, 8M HNO_3
-0.05, 0.02 M EDTA solution
-0.05 ammonia
-Cerous nitrate (0.5 mg/ml Ce)
-25% hydrazine dihydrochloride
-10 M NaOH
-80% ethanol
-Ceric hydroxide (10 µg Ce/ml) substrate
-10% sodium hydrogen sulfate
-20% titanium chloride

-Cerous fluoride (10 µg Ce/ml) substrate
-sodium dithionite ($Na_2S_2O_4$)
-1 M $MgCl_2$ - 203.3 g $MgCl_2$ dissolved in 1L DI water at pH 7
-Tamm's reagent - 28.4 g ammonium oxalate dissolved in DI water, mixed with 25.2 g oxalic acid dissolved in DI water, made up to 1L
-Fe carrier solution (10 mg/ml) - 72.3 g ferric nitrate dissolved in DI water, 50 ml conc HNO_3 added, made up to 1L with DI water
-Morgan's reagent - 82.04 g sodium acetate dissolved in 1L DI water, adjusted to pH 5 with acetic acid
-Coffin's reagent - 51.5 g sodium citrate dissolved in DI water, mixed with 5.25 g citric acid dissolved in DI water, made up to 1L

Procedure: The extraction, separation, and source preparation procedures shown below are used to process samples.

Sample ID # _____
Date _____

Sequential phase extractions

(I) IE *Readily ion-exchangeable*

1. Weigh 1-3 g powdered sample into a 50 ml PP centrifuge tube.

Sample weight = _____ g
Sample weight + tube = _____g

Add 50 ml of 1 M $MgCl_2$ at pH 7 and shake for 1 hour on wrist shaker.

2. Centrifuge at 10,000 rpm for 10 min; transfer supernate to PP bottle and label as 'IE' fraction. Add 30 ml ultrapure water to tube; homogenize; centrifuge at 10,000 rpm for 10 min and add supernate to PP bottle.

Place tube with residue in drying oven overnight.

3. Add $^{232}U/^{228}Th$ spike to supernate and record spike weight.

Spike # _____ Reference Date _____
Reference Activity _____pCi/g Spike weight _____ g

Add 0.5 mls Fe carrier and allow to equilibrate overnight.

4. Transfer half of solution into a 50 ml PP centrifuge tube. Add 2 mls conc HCl and mix; add ammonia solution gradually, stirring after each addition until Fe-hydroxide precipiates (red-brown).

Note: Take care not to add too much ammonia or the Mg will precipitate too, producing a thick white precipitate. If this does happen re-dissolve in HCl and repeat precipitation.

5. Centrifuge and discard supernate. Wash with 0.05 M ammonia, centrifuge and discard washings. Precipitate is now ready for U/Th ion exchange separation.

(II). Ad *Adsorbed*

1. Cool and record weight of dried residue in tube.

   Weight = _____ g

2. Add 30 ml of Morgan's reagent and 0.03 ml 0.02 M EDTA to the residue in the tube. Shake for at least 6 hrs on wrist shaker.

3. Centifuge at 10,000 rpm for 10 min. Transfer supernate to PP bottle and label as 'AD' fraction. Add 30 mls ultrapure water, homogenize, centrifuge at 10,000 rpm for 10 min, and add supernate to PP bottle.

   Place tube with residue in drying oven overnight.

4. Add $^{232}U/^{228}Th$ spike to supernate and record spike weight.

   Spike # _____ Reference Date _____
   Reference Activity _____pCi/g Spike weight _____ g

   Add 0.5 ml Fe carrier to supernate and allow to equilibrate overnight.

5. Transfer half of solution into a large teflon beaker. Evaporate to dryness under an infrared lamp.

6. Add 20 ml of conc HNO_3 and then add 20 mls perchloric acid. Reflux gently on a hotplate until the solution has turned pale straw to water-white in color.

7. Evaporate to dryness under an infrared lamp.

8. Dissolve in 50 mls 8 M HCl and 50 mls DI water, warm on hotplate if necessary. Allow to cool.

9. Co-precipitate the U, Th, and Fe with conc ammonia added dropwise. Transfer solution to 50 ml PP centrifuge tubes, centrifuge and discard supernate. Wash with 0.05 M ammonia, centrifuge and discard washings. Precipitate is now ready for U/Th ion exchange separation.

(III) AM *Amorphous iron oxides*

1. Cool and record weight of dried residue in tube.

   Weight = _____ g

2. Add 50 ml of Tamm's acid oxalate reagent to the residue in the tube. Shake for 4 hrs on wrist shaker in the dark. Check after the 1st hour and carefully release any built up pressure.

3. Centifuge quickly at 10,000 rpm for 10 min; minimize exposure to light. Transfer supernate to PP bottle and label as 'AM' fraction. Add a further 30 mls oxalate solution to the residue and shake for a further half hour.

4. Centrifuge at 10,000 rpm for 10 min, and add supernate to PP bottle. Add 30 mls ultrapure water to residue, homogenize, centrifuge, and add supernate to PP bottle.

   Place tube with residue in drying oven overnight.

5. Add $^{232}U/^{228}Th$ spike to supernate and record spike weight.

   Spike # _____ Reference Date _____
   Reference Activity _____pCi/g Spike weight _____ g

   Allow to equilibrate overnight.

6. Transfer half of solution into a large teflon beaker. Evaporate to dryness under an infrared lamp.

7. Add 20 ml of conc HNO_3 and then add 20 mls perchloric acid. Reflux gently on a hotplate until the solution has turned pale straw to water-white in color.

8. Evaporate to dryness under an infrared lamp.

9. Dissolve in 50 mls 8 M HCl and 50 mls DI water, warm on hotplate if necessary. Allow to cool.

10. Co-precipitate the U, Th, and Fe with conc ammonia added dropwise. Transfer solution to 50 ml PP centrifuge tubes, centrifuge and discard supernate. Wash with 0.05 M ammonia, centrifuge and discard washings. Precipitate is now ready for U/Th ion exchange separation.

(IV) CR *Crystalline iron oxides*

1. Cool and record weight of dried residue in tube.

   Weight = _____ g

2. Add 30 ml of Coffin's reagent to the residue in the tube. Add 1.5 g (5%) of sodium dithionite ($Na_2S_2O_4$) and shake for 1 hr on wrist shaker.

3. Centifuge at10,000 rpm for 10 min. Transfer supernate to PP bottle and label as 'CR' fraction.

4. Re-extract with fresh solution if residue is not completely free of Fe (residue should be white or gray). Centrifuge at 10,000 rpm for 10 min, and add supernate to PP bottle. Add 30 mls ultrapure water to residue, homogenize, centrifuge, and add supernate to PP bottle.

   Place tube with residue in drying oven overnight.

5. Add $^{232}U/^{228}Th$ spike to supernate and record spike weight.

   Spike # _____ Reference Date _____
   Reference Activity _____pCi/g Spike weight _____ g

   Allow to equilibrate overnight.

6. Transfer half of solution into a large teflon beaker. Evaporate to dryness under an infrared lamp. (A crystalline crust will form and it may be necessary to break it up with a stirring rod to assist drying-out.)

7. Add conc nitric acid drop-by-drop until effervescence ceases. (Be careful, reaction can be quite violent.)

8. Evaporate to dryness under an infrared lamp.

9. Add 20 ml of conc HNO_3 and then add 20 mls perchloric acid. Reflux gently on a hotplate until the solution has turned pale straw to water-white in color.

10. Evaporate to dryness under an infrared lamp.

11. Repeat steps 9-10.

12. Dissolve in 50 mls 8 M HCl and 50 mls DI water, warm on hotplate if necessary. Allow to cool.

13. Co-precipitate the U, Th, and Fe with conc ammonia added dropwise. Transfer solution to 50 ml PP centrifuge tubes, centrifuge and discard supernate. Wash with 0.05 M ammonia, centrifuge and discard washings. Precipitate is now ready for U/Th ion exchange separation.

(V) R *Resistate (primary minerals and clays)*

1. Cool and record weight of dried residue in tube.

   Weight = _____ g

2. Transfer residue to a teflon PFA vessel for microwave digestion.

3. Using a weighing boat, quantitatively add a known amount of $^{232}U/^{228}Th$ spike to the PFA vessel.

   $^{232}U/^{228}Th$ spike # _____ Reference Date _____

   Reference Activity _____pCi/g Spike wt. _____g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
_____	_____
_____	_____
_____	_____
_____	_____

4. Seal vessel, place on turntable in microwave, enter and run digestion program. Cool and vent vessel. If sample is not completely dissolved repeat step 4 .

5. Quantitatively transfer sample to a clean teflon beaker, washing the PFA vessel several times with ultrapure water.

6. Split sample into two parts: half is saved in a PP bottle for later processing if necessary and the other half is analyzed for U and Th isotopes.

7. Add 1 ml of perchloric acid ($HCLO_4$) to the sample solution in the teflon beaker. Evaporate to fumes of $HCLO_4$. Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).

8. Transfer solution to a 50 ml PP centrifuge tube. Add 0.5 ml Fe carrier and coprecipitate actinides by addition of NH_4OH to pH = 7.
   **Note: if sample already contains significant Fe, then Fe carrier does not need to be added.

9. Separate the Fe scavange by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing. Precipitate is ready for ion-exchange separation.

Ion Exchange Separation - all precipitates

Dissovle the precipitates in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Main Column (Biorad 1.5 cm diameter column with 10 cm resin; prewash with 4-5 column vols 9M HCl)

Load sample in 9M HCl and allow to drain	___			
Wash 3 volumes (~35 ml) 9M HCl –> Th	___	___	___	
Elute 4 volumes (~50 ml) 0.1M HCl –> U and Fe	___	___	___	___

Separation Date _____

***Note: May work on U and Th fractions simultaneously from this point on.

Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Heat Th fraction to evaporate HCl	___			
Add ~5 ml conc HNO_3 to dissolve residue	___			
Add equal vol (~5 ml) DI water so soln 8M HNO_3	___			
Load onto column in 8M HNO_3	___			
Wash 3-4 column vols 8M HNO_3	___	___	___	
Elute 4-5 column vols 9M HCl –> Th	___	___	___	___

Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO_3)

Evaoprate U fraction to near dryness	___			
Pick up in about 2 ml conc HNO_3	___			
Dilute with about 2 ml DI water to approx 8M HNO_3	___			
Load onto column in 8M HNO_3	___			
Wash 2 vols (8-10 ml) 8M HNO_3 –> Fe	___	___		
Elute 4-5 vols 0.1M HCl –> U	___	___	___	___

Source Preparation:

Thorium - OH^- precipitation onto filter

1. Evaporate solution containing Th to near dryness.
2. Add 6 ml 0.05 M EDTA soln to dissolve residue, transfer soln to a 50 ml PP centrifuge tube and place tube in boiling water for a few minutes.
3. Add 100 µl purified cerous nitrate (0.5 mg/ml Ce) to precipitate hydroxides. Mix, add 2 drops 25% hydrazine dihydrochloride, and 2 ml of 10M NaOH.
4. Place tube in boiling water bath for 10 minutes, remove, and place tube in cold water for 10 minutes to ensure complete precipitation
5. Wet a 25 mm membrane filter with 80% ethanol and place in a 50 ml polysulfone filter funnel.
6. Shake bottle of substrate suspension (ceric hydroxide containing 10 µg Ce/ml) vigorously and draw 2 consecutive 5 ml portions through the filter with full suction. Allow each portion to suck dry for 10-15 sec.
7. Without interrupting suction, pour the sample into the filter chimney and allow to suck dry.
8. While the sample is still filtering, add 0.5 ml 10M NaOH to the sample tube and about 5 ml ultrapure water down the sides of the tube. After the sample has sucked dry, swirl the wash solution around the sides and add it to the filter chimney.
9. Wash tube, filter chimney, precipitate, and filter with three consecutive 5 ml portions of 80% ethanol.
10. Suck filter dry for about 15 sec and remove the chimney and filter carefully without interrupting the suction. Transfer filter to a plastic container and at low temperature (~60C).
11. Glue to tape the dry filter onto a 1 inch stainless steel planchet and count.

Th Counting Date_____

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 100 µl purified cerous nitrate (0.5 mg/ml Ce, 50 µg of Ce carrier) and evaporate the solution until completely dry and no more fumes are given off.
2. Add 2 ml of 1M HCl to the beaker containing the purified U fraction and heat gently to dissolve the sodium hydrogen sulfate cake and any possible insoluble double salts with the Ce carrier.
3. Transfer solution to a 50 ml polycarbonate centrifuge tube with two more 2 ml portions of 1M HCl.
4. Add 2 drops of 20% titanium trichloride which should produce a strong violet color. If not, iron is probably present and a few more drops of titanium trichloride must be added to produce a permanent violet color or reduction and precipitation of U will be incomplete.
5. Add 0.5 ml (10 drops) of 48% HF. The violet color dissappears and any slight turbidity should clear up.
6. Mix thoroughly and allow solution to stand for 30 min in a cold water bath to obtain complete precipitation of cerous and uranous fluorides.
7. Place the tube in an ultrasonic bath for 1 min to disperse the precipitate.
8. Mount the precipitate on a 25 mm membrane filter previously treated with two 5 ml portions of cerous fluoride substrate as described above.
9. After sucking the precipitate dry, wash with 5 ml of water containing 2 drops of 48% HF and then with 80% ethanol.
10. Dry and analyze as described above.

U Counting Date_____

12/18/95 gp

Sequential extraction for U & Th was performed on samples NOPI-418-PWD and NOPI-423-PWD using procedure on p 122-129.

Results will be kept in a 3-ring binder entitled "Sequential Extraction Nopal I Samples".

12/18/95 gp

Reagent Preparation

1M $MgCl_2$ at pH 7 - dissolved 203.3g $MgCl_2$ (lot 940527) in 1L of H_2O in a 1L beaker. Added a few drops of 0.05M NaOH to bring to pH = 7. Transferred solution to a 1L PP bottle + labeled bottle "1M $MgCl_2$ at pH 7".

Morgan's Reagent - dissolved 82.04g sodium acetate (lot 937077) in 1L DI water. Adjusted pH to 5 with acetic acid (lot 945545) Transferred solution to a 1L PP bottle & labeled bottle "Morgan's Reagent".

12/20/95 gp

1M HCl - added 82 ml HCl (lot 945500) to 500 ml H_2O in volumetric flask. Made up to 1L with H_2O. Transferred solution to 1L PP bottle & labeled "1M HCl".

12/21/95 gp

Coffin's Reagent - dissolve 51.5g sodium citrate (lot 940621) in DI water, mix with 5.25g citric acid (lot 951297) and dissolve, made up to 1L. Transfer solution to 1L PP bottle and label "Coffin's Reagent".

Tamm's Reagent - 28.4g ammonium oxalate (lot 922654) dissolved in H_2O, mix with 25.2g oxalic acid (lot 905504), make up to 1L. Transfer solution to 1L PP bottle and label "Tamm's Reagent".

0.02M EDTA - dissolve 3.72g EDTA (lot 905518) in 500 ml H_2O. Transfer to 500ml PP bottle & label "0.02M EDTA".

0.05 m Ammonia - Mix 2.5 ml ammonia hydroxide (lot 893429) with 1L DI H_2O. Transfer to 1L PP bottle + label "0.05 m Ammonia"

8m HCl - add 656 ml conc HCl (lot 945500) to 300 ml H_2O in volumetric flask. Make up to mark with H_2O. Transfer solution to 1L PP bottle + label "8 m HCl".

1/9/96 gp

Sequential extractions for U+Th was performed on samples NOPI-417-PWD and NOPI-421-PWD using procedure on p 122-129.

Results are kept in a 3-ring binder entitled "Sequential Extraction Nopal I Samples".

1/16/96 gp

~~Sequential extraction~~ Sov U+Th was prepared on sample NOPI-420-PWD and NOPI-425-PWD using procedure on p 122-129.

Results are kept in a 3-ring binder entitled "Sequntial Egtraction Nopal I samples".

5/8/96 gp

0.3 5/8/96
~~0.6~~ M EDTA - dissolve 55.84 g EDTA (lot 905518) in ~~500~~ 250 gp 5/8/96 ml H_2O

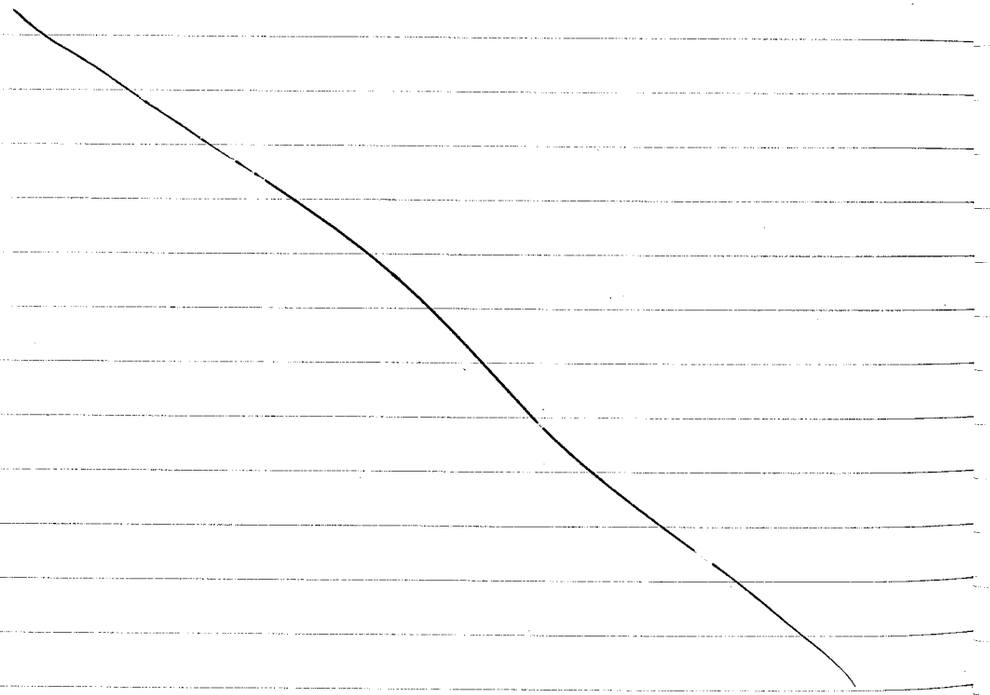
Transfer to 500 ml PP bottle labeled "0.3 gp 5/8/96 ~~0.6~~ M EDTA".

5/9/96

Sequential extractions for U + Th were performed on samples NOPI-418-PWD + NOPI-425-PWD using procedure on p 122-129.

Results are kept in a 3-ring binder entitled "Sequential Extraction Nopal I Samples".

This is a verum to determine reliability of procedure + reproducibility of results.

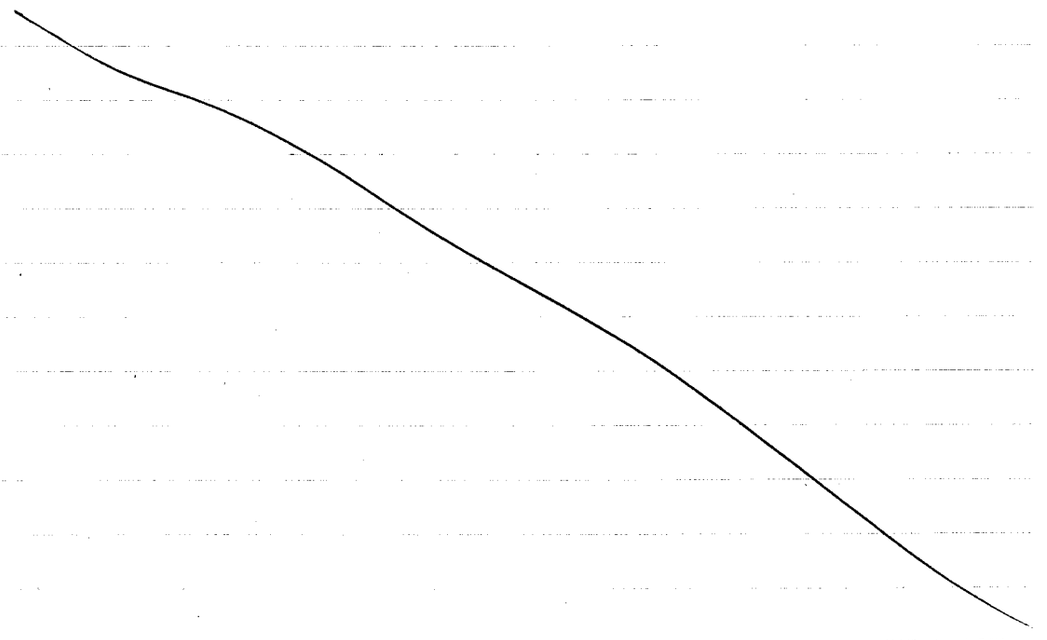


5/9/96

The following standard powders were analyzed for U + Th isotopes by α-spectrometry using procedure on p 59-62.

BL-5
DP2
SP1

Results are kept in a 3-ring binder entitled "Alpha Spectrometry of U/Th Standards" 1



8/13/96

The following powders from Nopal I were analyzed for U-Th isotopic by α-spectrometry using procedure on p 59-62.

NOPI-309-GAM2
NOPI-313-GAM2
NOPI-317-GAM2
NOPI-320-GAM2
NOPI-323-GAM2
NOPI-325-GAM2
NOPI-327-GAM2
NOPI-329-GAM2

Results are kept in a 3 ring binder entitled "Alpha-Spectrometry of Nopal I Samples".

This project was ended due to U.S. government budget cuts.

James O. Pinkerton
12/20/96