ALPHA SPECTROMETRY ANALYSES

NOPAL I, LEVEL 0

Supplemental Material, Technical Activity
Supporting Documentation: Experimental Data
for Alpha Spectrometry Analyses, Nopal I,
Level 0 (09/09/1996 through 09/18/1996)

U and Th Isotope Activities

FILENAME= NP309U.CHN
NP309TH.CHN

Sample # **NOPI-309**
Analyst **JDP**

Sep. date **9/9/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4605	0.5027	454.83	1/22/93	1326	439.2083

Th-228/U-232= 0.7988936

Counting time for Th= 1422.6 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916093

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for U-232= 1.0079701

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
15	1661	480	165
Bkgd	Bkgd	Bkgd	bkgd
1	3	10	5

U-238 counts	U-234 counts	U-232 counts
6727	7184	3633
bkgd	bkgd	bkgd
3	5	5

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
14	1658	451.42526	160

U-238* counts	U-234* counts	U-232sp counts
6724	7179	3599.3131

U-238(dpm/g)=	895.6903	±	18.44142
U-234(dpm/g)=	956.2999	±	19.46846
Th-232(dpm/g)=	11.87903	±	3.114708
Th-230(dpm/g)=	1406.816	±	72.90215
U-234/U-238=	1.067668	±	0.018114
Th-230/U-234=	1.471104	±	0.040052
Th-230/Th-232=	118.4286	±	30.71589
U234/Th-232=	80.50321	±	21.17165

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 1200631

Th(ppb)= 48616.38

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3512.581	350.88071	35.069609

NP309U.CHN

MCB # 1 ACQ 09-17-96 AT 07:36:31 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-309-GAM2 U 9/18/96

ROI # 7-1 RANGE : 74 = 4.07MeV to 153 = 4.25MeV
AREA : Gross = 7057 Net = 6727 +/- 104
CENTROID : 128.15 = 4.19MeV
SHAPE : Fwhm = 0.07MeV Fwtm = 0.12MeV

ID : No close library match

ROI # 7-2 RANGE : 324 = 4.65MeV to 402 = 4.83MeV
AREA : Gross = 7552 Net = 7184 +/- 108
CENTROID : 375.71 = 4.77MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.13MeV

ID : No close library match

ROI # 7-3 RANGE : 563 = 5.21MeV to 638 = 5.38MeV
AREA : Gross = 3880 Net = 3633 +/- 81
CENTROID : 615.06 = 5.33MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.12MeV

ID : No close library match

NP309 Th.CHW

CB # 1 ACQ 09-17-96 AT 07:36:30 RT : 85360.5 LT : 85356.4
No detector description was entered
NOPI-309-GAM2 Th 9/18/96

OI # 2-1 RANGE : 57 = 3.94MeV to 94 = 4.04MeV
AREA : Gross = 16 Net = 15 +/- 5
Could not properly fit the peak.

OI # 2-2 RANGE : 291 = 4.54MeV to 362 = 4.72MeV
AREA : Gross = 1697 Net = 1661 +/- 45
CENTROID : 345.30 = 4.68MeV
SHAPE : Fwhm = 0.04MeV Fwtm = 0.12MeV

ID : No close library match

OI # 2-3 RANGE : 601 = 5.33MeV to 656 = 5.47MeV
AREA : Gross = 631 Net = 480 +/- 42
CENTROID : 642.36 = 5.44MeV
SHAPE : Fwhm = 0.03MeV Fwtm = 0.06MeV

ID : No close library match

OI # 2-4 RANGE : 710 = 5.61MeV to 759 = 5.73MeV
AREA : Gross = 240 Net = 165 +/- 27
CENTROID : 740.14 = 5.69MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.06MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOFI-309-GAM2

Date 8/13/96

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 = .4605 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 # 25B Reference Date 1-22-93

   Reference Activity 204.88 pCi/g Spike wt. .5027 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7ml
HCl	3ml
HNO_3	1ml

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 # 54656A)

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 9/9/96

***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 9/17/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP313U.CHN
NP313TH.CHN

Sample # **NOPI-313**
Analyst **JDP**

Sep. date **9/9/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4743	0.4995	454.83	1/22/93	1326	439.2083

Th-228/U-232= 0.7988936

Counting time for Th= 1423 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916091

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for U-232= 1.0079701

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
11	1339	573	158
Bkgd	Bkgd	Bkgd	bkgd
1	14	8	9

U-238 counts	U-234 counts	U-232 counts
6421	6450	3167
bkgd	bkgd	bkgd
2	3	4

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
10	1325	551.81713	149

U-238* counts	U-234* counts	U-232sp counts
6419	6447	3137.9899

U-238(dpm/g)=	946.1691	±	20.54506
U-234(dpm/g)=	950.2963	±	20.61935
Th-232(dpm/g)=	6.696482	±	2.038353
Th-230(dpm/g)=	887.2838	±	44.29339
U-234/U-238=	1.004362	±	0.017706
Th-230/U-234=	0.933692	±	0.02804
Th-230/Th-232=	132.5	±	40.11401
U234/Th-232=	141.9098	±	43.30576

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 1268296

Th(ppb)= 27406.17

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3512.581	350.88071	35.069609

NP313N.CHN

MCB # 1 ACQ 09-17-96 AT 07:36:31 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-313-GAM2 U 9/18/96

ROI # 8-1 RANGE : 68 = 4.05MeV to 154 = 4.25MeV
AREA : Gross = 6856 Net = 6421 +/- 111
CENTROID : 123.56 = 4.18MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.14MeV

ID : No close library match

ROI # 8-2 RANGE : 317 = 4.63MeV to 407 = 4.84MeV
AREA : Gross = 7087 Net = 6450 +/- 124
CENTROID : 373.83 = 4.76MeV
SHAPE : Fwhm = 0.09MeV Fwtm = 0.14MeV

ID : No close library match

ROI # 8-3 RANGE : 562 = 5.20MeV to 640 = 5.38MeV
AREA : Gross = 3586 Net = 3167 +/- 91
CENTROID : 612.95 = 5.32MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.13MeV

ID : No close library match

NP313Th.CHN

MCB # 1 ACQ 09-17-96 AT 07:36:30 RT : 85388.6 LT : 85384.5
No detector description was entered
NOPI-313-GAM2 Th 9/18/96

ROI # 4-1 RANGE : 91 = 3.93MeV to 123 = 4.02MeV
AREA : Gross = 12 Net = 11 +/- 4
Could not properly fit the peak.

ROI # 4-2 RANGE : 310 = 4.53MeV to 381 = 4.72MeV
AREA : Gross = 1426 Net = 1339 +/- 48
CENTROID : 364.01 = 4.68MeV
SHAPE : Fwhm = 0.04MeV Fwtm = 0.13MeV

ID : No close library match

ROI # 4-3 RANGE : 593 = 5.30MeV to 656 = 5.47MeV
AREA : Gross = 669 Net = 573 +/- 39
CENTROID : 644.87 = 5.44MeV
SHAPE : Fwhm = 0.02MeV Fwtm = 0.07MeV

ID : No close library match

ROI # 4-4 RANGE : 706 = 5.61MeV to 753 = 5.74MeV
AREA : Gross = 216 Net = 158 +/- 24
CENTROID : 739.62 = 5.70MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.05MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-313-GAMZ

Date 8/13/96

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 = .4743 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 # 25B Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .4995 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/9/96

***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 9/17/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP317U.CHN
NP317TH.CHN

Sample # **NOPI-317**
Analyst **JDP**

Sep. date **9/9/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.471	0.5008	454.83	1/22/93	1326	439.2083

Th-228/U-232= 0.7988936

Counting time for Th= 1423.5 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.991609

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for U-232= 1.0079701

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
20	1101	516	184
Bkgd	Bkgd	Bkgd	bkgd
1	2	2	1

U-238 counts	U-234 counts	U-232 counts
6083	6434	4767
bkgd	bkgd	bkgd
2	3	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
19	1099	489.56842	183

U-238* counts	U-234* counts	U-232sp counts
6081	6431	4719.386

U-238(dpm/g)=	601.7325	±	11.63957
U-234(dpm/g)=	636.366	±	12.16108
Th-232(dpm/g)=	14.47915	±	3.299785
Th-230(dpm/g)=	837.5046	±	44.68108
U-234/U-238=	1.057556	±	0.018913
Th-230/U-234=	1.316074	±	0.042923
Th-230/Th-232=	57.84211	±	13.05083
U234/Th-232=	43.9505	±	10.05143

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 806594.5

Th(ppb)= 59257.71

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3512.581	350.88071	35.069609

NP317K.CHN

```
CB # 1      ACQ 09-17-96  AT 07:36:31   RT : 50004.0    LT : 50000.0
            No detector description was entered
            NOPI-317-GAM2 U 9/18/96

OI #  9-1   RANGE :   86 =    4.08MeV  to  160 =    4.25MeV
            AREA : Gross = 6445      Net = 6083       +/- 101
            CENTROID : 132.90  =    4.19MeV
            SHAPE : Fwhm =   0.07MeV    Fwtm =   0.12MeV

            ID : No close library match


OI #  9-2   RANGE :  330 =    4.65MeV  to  409 =    4.83MeV
            AREA : Gross = 6754      Net = 6434       +/- 102
            CENTROID : 384.10  =    4.77MeV
            SHAPE : Fwhm =   0.07MeV    Fwtm =   0.13MeV

            ID : No close library match


OI #  9-3   RANGE :  570 =    5.20MeV  to  645 =    5.38MeV
            AREA : Gross = 5007      Net = 4767       +/- 87
            CENTROID : 621.82  =    5.33MeV
            SHAPE : Fwhm =   0.06MeV    Fwtm =   0.13MeV

            ID : No close library match
```

NP317Th.CHW

ICB # 1 ACQ 09-17-96 AT 07:36:30 RT : 85413.2 LT : 85409.1
No detector description was entered
NOPI-317-GAM2 Th 9/18/96

ROI # 5-1 RANGE : 61 = 3.91MeV to 113 = 4.04MeV
AREA : Gross = 20 Net = 20 +/- 4
Could not properly fit the peak.

ROI # 5-2 RANGE : 310 = 4.54MeV to 384 = 4.72MeV
AREA : Gross = 1161 Net = 1101 +/- 42
CENTROID : 367.70 = 4.68MeV
SHAPE : Fwhm = 0.03MeV Fwtm = 0.13MeV

ID : No close library match

ROI # 5-3 RANGE : 624 = 5.33MeV to 685 = 5.49MeV
AREA : Gross = 650 Net = 516 +/- 42
CENTROID : 668.27 = 5.44MeV
SHAPE : Fwhm = 0.03MeV Fwtm = 0.07MeV

ID : No close library match

ROI # 5-4 RANGE : 734 = 5.61MeV to 793 = 5.76MeV
AREA : Gross = 231 Net = 184 +/- 25
CENTROID : 777.71 = 5.72MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.05MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-317-GAM2

Date 8/13/96

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 = .4710 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 # 25B Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .5008 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/9/96

***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 9/17/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP320U.CHN
NP320TH.CHN

Sample # **NOPI-320**
Analyst **JDP**

Sep. date **9/9/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5039	0.5006	454.83	1/22/93	1326	439.2083

Th-228/U-232= 0.7988936

Counting time for Th= 1424 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916088

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for U-232= 1.0079701

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
9	207	480	139
Bkgd	Bkgd	Bkgd	bkgd
4	12	2	1

U-238 counts	U-234 counts	U-232 counts
5407	5098	7837
bkgd	bkgd	bkgd
2	2	11

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
5	195	469.66905	138

U-238* counts	U-234* counts	U-232sp counts
5405	5096	7764.1191

U-238(dpm/g)=	303.753	±	5.370034
U-234(dpm/g)=	286.3876	±	5.15303
Th-232(dpm/g)=	3.710941	±	1.248523
Th-230(dpm/g)=	144.7267	±	12.03431
U-234/U-238=	0.942831	±	0.018406
Th-230/U-234=	0.505352	±	0.03583
Th-230/Th-232=	39	±	13.2796
U234/Th-232=	77.17386	±	26.00177

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 407166.8

Th(ppb)= 15187.48

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3512.581	350.88071	35.069609

NP320U.CHN

MCB # 1 ACQ 09-17-96 AT 07:36:31 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-320-GAM2 U 9/18/96

ROI # 10-1 RANGE : 67 = 4.07MeV to 148 = 4.26MeV
AREA : Gross = 5666 Net = 5407 +/- 93
CENTROID : 119.17 = 4.19MeV
SHAPE : Fwhm = 0.07MeV Fwtm = 0.13MeV

ID : No close library match

ROI # 10-2 RANGE : 321 = 4.67MeV to 395 = 4.84MeV
AREA : Gross = 5610 Net = 5098 +/- 105
CENTROID : 367.84 = 4.78MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.12MeV

ID : No close library match

ROI 10-3 RANGE : 551 = 5.21MeV to 632 = 5.40MeV
AREA : Gross = 8426 Net = 7837 +/- 124
CENTROID : 603.74 = 5.33MeV
SHAPE : Fwhm = 0.10MeV Fwtm = 0.15MeV

ID : No close library match

NP320Th.CHW

ICB # 1 ACQ 09-17-96 AT 07:36:31 RT : 85439.2 LT : 85435.1
No detector description was entered
NOPI-320-GAM2 Th 9/18/96

ROI # 6-1 RANGE : 68 = 3.93MeV to 101 = 4.02MeV
AREA : Gross = 9 Net = 9 +/- 3
Could not properly fit the peak.

ROI # 6-2 RANGE : 294 = 4.55MeV to 349 = 4.70MeV
AREA : Gross = 208 Net = 207 +/- 15
CENTROID : 331.09 = 4.65MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.10MeV

ID : No close library match

ROI # 6-3 RANGE : 546 = 5.25MeV to 614 = 5.43MeV
AREA : Gross = 514 Net = 480 +/- 29
CENTROID : 598.23 = 5.39MeV
SHAPE : Fwhm = 0.04MeV Fwtm = 0.13MeV

ID : No close library match

ROI # 6-4 RANGE : 657 = 5.55MeV to 710 = 5.70MeV
AREA : Gross = 152 Net = 139 +/- 16
CENTROID : 682.09 = 5.62MeV
SHAPE : Fwhm = 0.00MeV Fwtm = 0.06MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-320-GAM2

Date 8/13/96

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 = .5039 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 # 25B Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .5006 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/9/96

***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 9/17/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP323U.CHN
NP323TH.CHN

Sample # **NOPI-323**
Analyst **JDP**

Sep. date **9/10/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4807	0.5016	454.83	1/22/93	1327	439.1967

Th-228/U-232= 0.7991733

Counting time for Th= 1405 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916153

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 7 (days)
CF for U-232= 1.0070123

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
246	24305	28003	9290
Bkgd	Bkgd	Bkgd	bkgd
1	3	12	5

U-238 counts	U-234 counts	U-232 counts
4949	4743	4755
bkgd	bkgd	bkgd
4	4	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
245	24302	27486.415	9285

U-238* counts	U-234* counts	U-232sp counts
4945	4739	4709.9724

U-238(dpm/g)=	481.161	±	9.770839
U-234(dpm/g)=	461.1167	±	9.462919
Th-232(dpm/g)=	3.264611	±	0.209056
Th-230(dpm/g)=	323.8228	±	2.838843
U-234/U-238=	0.958342	±	0.019473
Th-230/U-234=	0.702258	±	0.011148
Th-230/Th-232=	99.19184	±	6.356165
U234/Th-232=	141.2471	±	9.498162

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 644974

Th(ppb)= 13360.82

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3513.718	350.99429	35.080961

NP3234.CHN

ICB # 1 ACQ 09-17-96 AT 07:36:31 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-323-GAM2 U 9/18/96

ROI # 11-1 RANGE : 91 = 4.09MeV to 163 = 4.25MeV
AREA : Gross = 5217 Net = 4949 +/- 89
CENTROID : 137.39 = 4.20MeV
SHAPE : Fwhm = 0.06MeV Fwtm = 0.12MeV

ID : No close library match

ROI # 11-2 RANGE : 339 = 4.66MeV to 415 = 4.83MeV
AREA : Gross = 5229 Net = 4743 +/- 102
CENTROID : 389.06 = 4.77MeV
SHAPE : Fwhm = 0.07MeV Fwtm = 0.12MeV

ID : No close library match

ROI # 11-3 RANGE : 576 = 5.20MeV to 650 = 5.38MeV
AREA : Gross = 5191 Net = 4755 +/- 99
CENTROID : 625.96 = 5.32MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.12MeV

ID : No close library match

NP323Th.CHN

ICB # 1 ACQ 09-18-96 AT 07:35:12 RT : 84357.2 LT : 84352.0
No detector description was entered
NOPI-323-GAM2 Th 9/19/96

OI # 2-1 RANGE : 47 = 3.92MeV to 93 = 4.04MeV
AREA : Gross = 310 Net = 246 +/- 26
CENTROID : 75.46 = 3.99MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.07MeV

ID : No close library match

OI # 2-2 RANGE : 261 = 4.46MeV to 363 = 4.72MeV
AREA : Gross = 25678 Net = 24305 +/- 216
CENTROID : 342.46 = 4.67MeV
SHAPE : Fwhm = 0.06MeV Fwtm = 0.15MeV

ID : No close library match

OI # 2-3 RANGE : 543 = 5.18MeV to 661 = 5.48MeV
AREA : Gross = 29650 Net = 28003 +/- 243
CENTROID : 638.12 = 5.43MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.17MeV

ID : No close library match

OI # 2-4 RANGE : 684 = 5.54MeV to 765 = 5.75MeV
AREA : Gross = 11313 Net = 9290 +/- 187
CENTROID : 739.74 = 5.68MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.14MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-323-GAM2

Date 8/13/96

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 = .4807 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 # 2513 Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .5016 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/10/96

***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 9/18/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP325U.CHN
NP325TH.CHN

Sample # **NOPI-325**
Analyst **JDP**

Sep. date **9/10/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4713	0.5019	454.83	1/22/93	1327	439.1967

Th-228/U-232= 0.7991733

Counting time for Th= 1406 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916149

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 7 (days)
CF for U-232= 1.0070123

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
92	2596	21120	6740
Bkgd	Bkgd	Bkgd	bkgd
2	10	20	12

U-238 counts	U-234 counts	U-232 counts
2800	2704	18895
bkgd	bkgd	bkgd
2	4	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
90	2586	20828.822	6728

U-238* counts	U-234* counts	U-232sp counts
2798	2700	18755.481

U-238(dpm/g)=	69.77476	±	1.412946
U-234(dpm/g)=	67.3309	±	1.384377
Th-232(dpm/g)=	1.615093	±	0.168751
Th-230(dpm/g)=	46.40701	±	0.965173
U-234/U-238=	0.964975	±	0.026018
Th-230/U-234=	0.689238	±	0.018939
Th-230/Th-232=	28.73333	±	3.048277
U234/Th-232=	41.68855	±	4.439324

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 93529.83

Th(ppb)= 6609.968

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3513.718	350.99429	35.080961

NP325U.CHW

ICB # 1 ACQ 09-17-96 AT 07:36:31 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-325-GAM2 U 9/18/96

ROI # 12-1 RANGE : 90 = 4.08MeV to 161 = 4.25MeV
AREA : Gross = 3255 Net = 2800 +/- 88
CENTROID : 136.14 = 4.19MeV
SHAPE : Fwhm = 0.06MeV Fwtm = 0.12MeV

ID : No close library match

ROI # 12-2 RANGE : 339 = 4.66MeV to 411 = 4.83MeV
AREA : Gross = 3178 Net = 2704 +/- 89
CENTROID : 383.86 = 4.77MeV
SHAPE : Fwhm = 0.07MeV Fwtm = 0.12MeV

ID : No close library match

ROI 12-3 RANGE : 564 = 5.18MeV to 652 = 5.39MeV
AREA : Gross = 19651 Net = 18895 +/- 171
CENTROID : 622.71 = 5.32MeV
SHAPE : Fwhm = 0.09MeV Fwtm = 0.14MeV

ID : No close library match

WP325Th.CHW

ICB # 1 ACQ 09-18-96 AT 07:35:12 RT : 84390.6 LT : 84385.3
No detector description was entered
NOPI-325-GAM2 Th 9/19/96

ROI # 4-1 RANGE : 85 = 3.92MeV to 125 = 4.03MeV
AREA : Gross = 135 Net = 92 +/- 19
CENTROID : 117.05 = 4.00MeV
SHAPE : Fwhm = 0.00MeV Fwtm = 0.04MeV

ID : No close library match

ROI # 4-2 RANGE : 307 = 4.52MeV to 381 = 4.72MeV
AREA : Gross = 2810 Net = 2596 +/- 71
CENTROID : 362.86 = 4.67MeV
SHAPE : Fwhm = 0.06MeV Fwtm = 0.15MeV

ID : No close library match

ROI 4-3 RANGE : 565 = 5.22MeV to 663 = 5.49MeV
AREA : Gross = 22738 Net = 21120 +/- 215
CENTROID : 639.98 = 5.43MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.17MeV

ID : No close library match

ROI # 4-4 RANGE : 688 = 5.56MeV to 757 = 5.75MeV
AREA : Gross = 8117 Net = 6740 +/- 146
CENTROID : 735.49 = 5.69MeV
SHAPE : Fwhm = 0.08MeV Fwtm = 0.14MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-325-GAMZ

Date 8/13/96

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 = .4713 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 # 25B Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .5019 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/10/96

***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 9/18/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP327U.CHN
NP327TH.CHN

Sample # **NOPI-327**
Analyst **JDP**

Sep. date **9/10/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4942	0.5007	454.83	1/22/93	1327	439.1967

Th-228/U-232= 0.7991733

Counting time for Th= 1407 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916146

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 7 (days)
CF for U-232= 1.0070123

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
208	5125	26866	9219
Bkgd	Bkgd	Bkgd	bkgd
1	3	3	2

U-238 counts	U-234 counts	U-232 counts
3594	2675	15063
bkgd	bkgd	bkgd
10	10	15

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
207	5122	26390.53	9217

U-238* counts	U-234* counts	U-232sp counts
3584	2665	14943.214

U-238(dpm/g)=	106.723	±	1.981226
U-234(dpm/g)=	79.35735	±	1.665029
Th-232(dpm/g)=	2.789312	±	0.194151
Th-230(dpm/g)=	69.01864	±	1.052039
U-234/U-238=	0.743583	±	0.018988
Th-230/U-234=	0.86972	±	0.020745
Th-230/Th-232=	24.74396	±	1.750155
U234/Th-232=	28.45051	±	2.068321

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 143057.2

Th(ppb)= 11415.6

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3513.718	350.99429	35.080961

NP327Th.CHN

```
MCB # 1        ACQ 09-18-96  AT 07:35:12   RT : 84427.7    LT : 84422.5
               No detector description was entered
               NOPI-327-GAM2 Th 9/19/96

ROI #   5-1    RANGE :   60 =    3.91MeV  to  115 =    4.05MeV
               AREA : Gross = 264       Net = 208        +/- 26
               CENTROID : 95.56   =    4.00MeV
               SHAPE : Fwhm =   0.04MeV    Fwtm =   0.10MeV

               ID : No close library match


ROI #   5-2    RANGE :  306 =    4.53MeV  to  385 =    4.73MeV
               AREA : Gross = 5659      Net = 5125       +/- 108
               CENTROID : 365.67  =    4.68MeV
               SHAPE : Fwhm =   0.07MeV    Fwtm =   0.13MeV

               ID : No close library match


ROI #   5-3    RANGE :  590 =    5.25MeV  to  689 =    5.50MeV
               AREA : Gross = 29182     Net = 26866      +/- 251
               CENTROID : 667.98  =    5.44MeV
               SHAPE : Fwhm =   0.05MeV    Fwtm =   0.16MeV

               ID : No close library match


ROI #   5-4    RANGE :  722 =    5.58MeV  to  796 =    5.77MeV
               AREA : Gross = 10925     Net = 9219       +/- 170
               CENTROID : 771.54  =    5.70MeV
               SHAPE : Fwhm =   0.08MeV    Fwtm =   0.13MeV

               ID : No close library match
```

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-327.6AMZ

Date 8/13/96

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 = .4942 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 # 253 Reference Date 1/22/93

   Reference Activity 204.88 pCi/g Spike wt. .5007 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/10/96

***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 9/18/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96

U and Th Isotope Activities

FILENAME= NP329U.CHN
NP329TH.CHN

Sample # **NOPI-329**
Analyst **JDP**

Sep. date **9/10/96**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4892	0.4997	454.83	1/22/93	1327	439.1967

Th-228/U-232= 0.7991733

Counting time for Th= 1407.5 (mins.)
Days btwn. sep. and count.= 8 (days)
CF for Th-228= 0.9916144

Counting time for U= 833.33 (mins.)
Days btwn. sep. and count.= 7 (days)
CF for U-232= 1.0070123

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
201	3635	22312	7627
Bkgd	Bkgd	Bkgd	bkgd
5	23	3	1

U-238 counts	U-234 counts	U-232 counts
3641	3290	20136
bkgd	bkgd	bkgd
8	10	20

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
196	3612	21894.06	7626

U-238* counts	U-234* counts	U-232sp counts
3633	3280	19975.923

U-238(dpm/g)=	81.59068	±	1.469339
U-234(dpm/g)=	73.66293	±	1.385203
Th-232(dpm/g)=	3.209613	±	0.227406
Th-230(dpm/g)=	59.14859	±	1.057953
U-234/U-238=	0.902835	±	0.021717
Th-230/U-234=	0.802963	±	0.019322
Th-230/Th-232=	18.42857	±	1.335306
U234/Th-232=	22.95072	±	1.682392

Decay constant (m-1)	U-238	Th-232
	2.948E-16	9.413E-17

U(ppb)= 109368.5

Th(ppb)= 13135.74

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	3513.718	350.99429	35.080961

NP329U.CHW

ICB # 1 ACQ 09-17-96 AT 07:36:32 RT : 50004.0 LT : 50000.0
No detector description was entered
NOPI-329-GAM2 U 9/18/96

ROI # 14-1 RANGE : 81 = 4.07MeV to 151 = 4.24MeV
AREA : Gross = 3985 Net = 3641 +/- 86
CENTROID : 127.83 = 4.18MeV
SHAPE : Fwhm = 0.06MeV Fwtm = 0.11MeV

ID : No close library match

ROI # 14-2 RANGE : 333 = 4.66MeV to 400 = 4.82MeV
AREA : Gross = 3777 Net = 3290 +/- 91
CENTROID : 378.11 = 4.77MeV
SHAPE : Fwhm = 0.04MeV Fwtm = 0.11MeV

ID : No close library match

ROI # 14-3 RANGE : 555 = 5.18MeV to 639 = 5.38MeV
AREA : Gross = 20816 Net = 20136 +/- 171
CENTROID : 614.77 = 5.32MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.13MeV

ID : No close library match

NP 329 Th.CHN

MCB # 1 ACQ 09-18-96 AT 07:35:12 RT : 84457.1 LT : 84451.9
No detector description was entered
NOPI-329-GAM2 Th 9/19/96

ROI # 6-1 RANGE : 63 = 3.92MeV to 106 = 4.03MeV
AREA : Gross = 201 Net = 129 +/- 24
CENTROID : 95.73 = 4.01MeV
SHAPE : Fwhm = 0.01MeV Fwtm = 0.05MeV

ID : No close library match

ROI # 6-2 RANGE : 293 = 4.55MeV to 354 = 4.72MeV
AREA : Gross = 4317 Net = 3635 +/- 100
CENTROID : 335.57 = 4.67MeV
SHAPE : Fwhm = 0.05MeV Fwtm = 0.12MeV

ID : No close library match

ROI 6-3 RANGE : 531 = 5.20MeV to 622 = 5.46MeV
AREA : Gross = 23384 Net = 22312 +/- 194
CENTROID : 602.12 = 5.40MeV
SHAPE : Fwhm = 0.04MeV Fwtm = 0.15MeV

ID : No close library match

ROI # 6-4 RANGE : 647 = 5.52MeV to 718 = 5.72MeV
AREA : Gross = 8875 Net = 7627 +/- 146
CENTROID : 692.18 = 5.65MeV
SHAPE : Fwhm = 0.07MeV Fwtm = 0.13MeV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-329-GAM 2

Date 8/13/96

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 = .4892 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 # 25B Reference Date 1/22/93

   Reference Activity 204-88 pCi/g Spike wt. .4997 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
HF	7
HCl	3
HNO_3	1

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 9/10/96

***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 9/18/96

Uranium - F^- precipitation onto filter

1. To the solution containing U, add 1 ml of 10% sodium hydrogen sulfate and 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 substate 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 9/17/96