

Alpha Spectrometry of Nopal I Samples

U and Th Isotope Activities

FILENAME= NP209U.CHN
NP209TH.CHN

Sample # NOPI-209
Analyst JDP

Sep. date 1/23/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4403	1.0036	454.83	1/22/93	731	446.15055

Th-228/U-232= 0.5817043

Counting time for Th= 957.16 (mins.)
Days btwn. sep. and count.= 2 (days)
CF for Th-228= 0.997689

Counting time for U= 1156.61 (mins.)
Days btwn. sep. and count.= 2 (days)
CF for U-232= 1.0023162

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
181	22423	10973	1514

U-238 counts	U-234 counts	U-232 counts
447	581	560

Bkgd	Bkgd	Bkgd	bkgd
4	8	17	26

bkgd	bkgd	bkgd
6	6	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
177	22415	10725.332	1488

U-238* counts	U-234* counts	U-232sp counts
441	575	548.72903

U-238(dpm/g)= 817.28598 ± 51.83714
U-234(dpm/g)= 1065.6223 ± 63.10504

Th-232(dpm/g)= 9.7624367 ± 0.731596
Th-230(dpm/g)= 1236.2995 ± 14.40328

U-234/U-238= 1.3038549 ± 0.082032
Th-230/U-234= 1.1601667 ± 0.048751
Th-230/Th-232= 126.63842 ± 9.450874
U234/Th-232= 109.15536 ± 10.42584

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

U(ppb)= 1095533.9

Th(ppb)= 39953.972

Spike 25A Spike 25B Spike 25C
10/9/92
Th-228 (dpm/g)= 2598.0684 259.52771 25.939115

NP209U-CHN

MCB # 1 ACQ 01-25-95 AT 13:11:41 RT : 69396.5 LT : 69389.5
No detector description was entered
NOPI-209 U 1/26/95

ROI # 8-1 RANGE : 66 = 405.93keV to 152 = 425.56keV
AREA : Gross = 447 Net = 447 +/- 21
CENTROID : 124.01 = 419.17keV
SHAPE : Fwhm = 2.75keV Fwtm = 6.10keV

ID : No close library match

ROI # 8-2 RANGE : 324 = 464.82keV to 401 = 482.39keV
AREA : Gross = 582 Net = 581 +/- 24
CENTROID : 377.99 = 477.14keV
SHAPE : Fwhm = 4.92keV Fwtm = 13.16keV

ID : No close library match

ROI # 8-3 RANGE : 549 = 516.17keV to 639 = 536.71keV
AREA : Gross = 560 Net = 560 +/- 24
CENTROID : 617.77 = 531.86keV
SHAPE : Fwhm = 3.56keV Fwtm = 4.76keV

ID : No close library match

NP209Th.c#W

MCB # 1 ACQ 01-25-95 AT 16:49:43 RT : 57429.9 LT : 57423.4
No detector description was entered
NOPI-209 Th 1/26/95

ROI # 1-1 RANGE : 32 = 3.87keV to 86 = 4.00keV
AREA : Gross = 234 Net = 181 +/- 25
CENTROID : 75.83 = 3.98keV
SHAPE : Fwhm = 0.00keV Fwtm = 0.07keV

ID : No close library match

ROI # 1-2 RANGE : 225 = 4.35keV to 358 = 4.68keV
AREA : Gross = 22423 Net = 22423 +/- 150
CENTROID : 335.55 = 4.62keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-3 RANGE : 508 = 5.05keV to 654 = 5.41keV
AREA : Gross = 11010 Net = 10973 +/- 109
CENTROID : 631.37 = 5.36keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.14keV

ID : No close library match

ROI # 1-4 RANGE : 672 = 5.46keV to 781 = 5.73keV
AREA : Gross = 1541 Net = 1514 +/- 45
CENTROID : 734.79 = 5.61keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.11keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP294U3.CHN
NP294TH3.CHNSample # NOPI-294
Analyst JDP

Sep. date 1/4/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3414	0.2925	454.83	1/22/93	712	446.37404

Th-228/U-232= 0.5727428

Counting time for Th= 1339.1 (mins.)
Days btwn. sep. and count.= 13 (days)
CF for Th-228= 0.9867313

Counting time for U= 500.03 (mins.)
Days btwn. sep. and count.= 12 (days)
CF for U-232= 1.0116811

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
61	5934	3586	2812

U-238 counts	U-234 counts	U-232 counts
2260	2521	3130

Bkgd	Bkgd	Bkgd	bkgd
3	20	35	8

bkgd	bkgd	bkgd
2	2	3

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
58	5914	3390.1402	2804

U-238* counts	U-234* counts	U-232sp counts
2258	2519	3090.895

U-238(dpm/g)= 279.38364 ± 7.712035
U-234(dpm/g)= 311.67732 ± 8.34083

Th-232(dpm/g)= 3.7474104 ± 0.48387
Th-230(dpm/g)= 382.10664 ± 8.082099

U-234/U-238= 1.115589 ± 0.032316
Th-230/U-234= 1.2259687 ± 0.029146
Th-230/Th-232= 101.96552 ± 13.12228
U234/Th-232= 83.171388 ± 10.96742

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

U(ppb)= 374500.81

Th(ppb)= 15336.737

Spike 25A Spike 25B Spike 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP294U3.CHN

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-294 U 1/17/95

ROI # 12-1 RANGE : 51 = 399.29keV to 164 = 425.45keV
AREA : Gross = 2261 Net = 2260 +/- 48
CENTROID : 131.72 = 417.98keV
SHAPE : Fwhm = 7.49keV Fwtm = 13.10keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.075 +/- 0.00

ROI # 12-2 RANGE : 299 = 456.71keV to 414 = 483.33keV
AREA : Gross = 2550 Net = 2521 +/- 55
CENTROID : 387.66 = 477.24keV
SHAPE : Fwhm = 7.87keV Fwtm = 14.56keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.084 +/- 0.00

ROI # 12-3 RANGE : 550 = 514.82keV to 646 = 537.05keV
AREA : Gross = 3179 Net = 3130 +/- 62
CENTROID : 620.64 = 531.18keV
SHAPE : Fwhm = 7.53keV Fwtm = 12.38keV

ID : No close library match

NP294 Th 3. CHN

MCB # 1 ACQ 01-16-95 AT 22:19:55 RT : 80345.9 LT : 80332.7
No detector description was entered
NOPI-294 Th 1/18/95

ROI # 7-1 RANGE : 13 to 64
AREA : Gross = 157 Net = 61 +/- 28
Could not properly fit the peak.

ROI # 7-2 RANGE : 211 to 370
AREA : Gross = 5974 Net = 5934 +/- 83
CENTROID : 330.98
SHAPE : Fwhm = 47.02 Channels Fwtm = 78.75 Channels

ROI # 7-3 RANGE : 539 to 681
AREA : Gross = 3821 Net = 3586 +/- 95
CENTROID : 653.57
SHAPE : Fwhm = 26.59 Channels Fwtm = 76.00 Channels

ROI # 7-4 RANGE : 692 to 802
AREA : Gross = 3068 Net = 2812 +/- 85
CENTROID : 766.93
SHAPE : Fwhm = 28.52 Channels Fwtm = 54.46 Channels

U and Th Isotope Activities

FILENAME= NP301U3.CHN
NP301TH3.CHN

Sample # NOPI-301
Analyst JDP

Sep. date 1/4/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3205	0.2975	454.83	1/22/93	712	446.37404

Th-228/U-232= 0.5727428

Counting time for Th= 416.75 (mins.)
Days btwn. sep. and count.= 13 (days)
CF for Th-228= 0.9870448

Counting time for U= 500.03 (mins.)
Days btwn. sep. and count.= 12 (days)
CF for U-232= 1.0116811

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
77	11158	1798	1417

U-238 counts	U-234 counts	U-232 counts
3456	3999	1244

Bkgd	Bkgd	Bkgd	bkgd
12	35	30	12

bkgd	bkgd	bkgd
4	5	7

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
65	11123	1650.763	1405

U-238* counts	U-234* counts	U-232sp counts
3452	3994	1222.7173

U-238(dpm/g)= 1169.7757 ± 38.67715
U-234(dpm/g)= 1353.4426 ± 43.93837

Th-232(dpm/g)= 9.3442864 ± 1.087443
Th-230(dpm/g)= 1599.023 ± 40.63518

U-234/U-238= 1.1570104 ± 0.026872
Th-230/U-234= 1.1814487 ± 0.021775
Th-230/Th-232= 171.12308 ± 19.56846
U234/Th-232= 144.84173 ± 17.49956

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

U(ppb)= 1568030

Th(ppb)= 38242.64

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP301U3.CHN

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-301 U 1/17/95

ROI # 13-1 RANGE : 27 = 396.69keV to 154 = 426.03keV
AREA : Gross = 3582 Net = 3456 +/- 78
CENTROID : 116.96 = 417.48keV
SHAPE : Fwhm = 7.44keV Fwtm = 15.86keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.115 +/- 0.00

ROI # 13-2 RANGE : 267 = 452.14keV to 405 = 484.03keV
AREA : Gross = 4087 Net = 3999 +/- 77
CENTROID : 364.76 = 474.73keV
SHAPE : Fwhm = 10.50keV Fwtm = 15.65keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.133 +/- 0.00

ROI # 13-3 RANGE : 522 = 511.06keV to 634 = 536.94keV
AREA : Gross = 1272 Net = 1244 +/- 42
CENTROID : 605.30 = 530.31keV
SHAPE : Fwhm = 9.68keV Fwtm = 17.37keV

ID : No close library match

NP301Th3.CHW

MCB # 1 ACQ 01-16-95 AT 22:19:55 RT : 25004.9 LT : 25000.0
No detector description was entered
NOPI-301 Th 1/18/95

ROI # 6-1 RANGE : 25 = 3.90keV to 89 = 4.07keV
AREA : Gross = 77 Net = 77 +/- 9
CENTROID : 53.11 = 3.97keV
SHAPE : Fwhm = 0.00keV Fwtm = 0.01keV

ID : No close library match

ROI # 6-2 RANGE : 213 = 4.39keV to 344 = 4.74keV
AREA : Gross = 11290 Net = 11158 +/- 118
CENTROID : 313.06 = 4.66keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.18keV

ID : No close library match

ROI # 6-3 RANGE : 516 = 5.19keV to 624 = 5.48keV
AREA : Gross = 1800 Net = 1798 +/- 43
CENTROID : 595.27 = 5.40keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.16keV

ID : No close library match

ROI # 6-4 RANGE : 640 = 5.52keV to 722 = 5.74keV
AREA : Gross = 1581 Net = 1417 +/- 59
CENTROID : 696.89 = 5.67keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.14keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP417U3.CHN
NP417TH3.CHN

Sample # **NOPI-417**
Analyst **JDP**

Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3068	0.2972	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th= **416.75** (mins.)
Days btwn. sep. and count.= **13** (days)
CF for Th-228= **0.9870448**

Counting time for U= **500.03** (mins.)
Days btwn. sep. and count.= **12** (days)
CF for U-232= **1.0116811**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
39	38876	2320	2568

U-238 counts	U-234 counts	U-232 counts
3817	4907	620

Bkgd	Bkgd	Bkgd	bkgd
3	35	12	10

bkgd	bkgd	bkgd
2	2	6

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
36	38841	2164.9395	2558

U-238* counts	U-234* counts	U-232sp counts
3815	4905	606.91062

U-238(dpm/g)= **2718.0797** ± **117.6928**
U-234(dpm/g)= **3494.6739** ± **148.9524**

Th-232(dpm/g)= **4.1182125** ± **0.664961**
Th-230(dpm/g)= **4443.2081** ± **94.95971**

U-234/U-238= **1.2857143** ± **0.027748**
Th-230/U-234= **1.2714228** ± **0.019262**
Th-230/Th-232= **1078.9167** ± **172.8516**
U234/Th-232= **848.58998** ± **141.7139**

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

U(ppb)= **3643459.7**

Th(ppb)= **16854.291**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP41743.CHN

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-417 U 1/17/95

ROI # 9-1 RANGE : 58 = 401.57keV to 158 = 424.68keV
AREA : Gross = 3998 Net = 3817 +/- 82
CENTROID : 129.99 = 418.20keV
SHAPE : Fwhm = 7.32keV Fwtm = 16.33keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.127 +/- 0.00

ROI # 9-2 RANGE : 278 = 452.41keV to 408 = 482.46keV
AREA : Gross = 5083 Net = 4907 +/- 93
CENTROID : 376.40 = 475.16keV
SHAPE : Fwhm = 9.80keV Fwtm = 17.82keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.164 +/- 0.00

ROI # 9-3 RANGE : 543 = 513.66keV to 645 = 537.24keV
AREA : Gross = 622 Net = 620 +/- 26
CENTROID : 598.55 = 526.50keV
SHAPE : Fwhm = 0.60keV Fwtm = 15.82keV

ID : No close library match

NP417Th3.CHW

MCB # 1 ACQ 01-16-95 AT 22:19:54 RT : 25004.9 LT : 25000.0
No detector description was entered
NOPI-417 Th 1/18/95

ROI # 1-1 RANGE : 10 = 3.81keV to 71 = 3.97keV
AREA : Gross = 70 Net = 39 +/- 18
CENTROID : 64.86 = 3.95keV
SHAPE : Fwhm = 0.00keV Fwtm = 0.01keV

ID : No close library match

ROI # 1-2 RANGE : 195 = 4.27keV to 364 = 4.69keV
AREA : Gross = 39019 Net = 38876 +/- 207
CENTROID : 331.32 = 4.61keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

ROI # 1-3 RANGE : 548 = 5.15keV to 647 = 5.40keV
AREA : Gross = 2470 Net = 2320 +/- 68
CENTROID : 627.62 = 5.35keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.16keV

ID : No close library match

ROI # 1-4 RANGE : 658 = 5.42keV to 785 = 5.74keV
AREA : Gross = 2824 Net = 2568 +/- 88
CENTROID : 731.79 = 5.61keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.15keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP418U3.CHN
NP418TH3.CHN

Sample # **NOPI-418**
Analyst **JDP**

Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4288	0.493	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th= **416.75** (mins.)
Days btwn. sep. and count.= **13** (days)
CF for Th-228= **0.9870448**

Counting time for U= **500.03** (mins.)
Days btwn. sep. and count.= **12** (days)
CF for U-232= **1.0116811**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
44	38200	2826	2429

U-238 counts	U-234 counts	U-232 counts
3899	5007	774

Bkgd	Bkgd	Bkgd	bkgd
1	10	40	20

bkgd	bkgd	bkgd
0	0	5

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
43	38190	2650.214	2409

U-238* counts	U-234* counts	U-232sp counts
3899	5007	760.12095

U-238(dpm/g)= **2632.4589** ± **103.5887**
U-234(dpm/g)= **3380.539** ± **130.5654**

Th-232(dpm/g)= **4.7691197** ± **0.724547**
Th-230(dpm/g)= **4235.6437** ± **82.5717**

U-234/U-238= **1.2841754** ± **0.027428**
Th-230/U-234= **1.2529492** ± **0.018832**
Th-230/Th-232= **888.13953** ± **133.9692**
U234/Th-232= **708.83921** ± **111.1157**

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

U(ppb)= **3528689**

Th(ppb)= **19518.208**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP41843.CHN

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-418 U 1/17/95

ROI # 10-1 RANGE : 30 = 401.27keV to 146 = 428.13keV
AREA : Gross = 4056 Net = 3899 +/- 82
CENTROID : 113.36 = 420.57keV
SHAPE : Fwhm = 8.88keV Fwtm = 17.41keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.130 +/- 0.00

ROI # 10-2 RANGE : 263 = 455.23keV to 396 = 486.03keV
AREA : Gross = 5074 Net = 5007 +/- 80
CENTROID : 362.65 = 478.30keV
SHAPE : Fwhm = 9.62keV Fwtm = 18.90keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.167 +/- 0.00

ROI # 10-3 RANGE : 536 = 518.45keV to 631 = 540.45keV
AREA : Gross = 798 Net = 774 +/- 34
CENTROID : 603.72 = 534.13keV
SHAPE : Fwhm = 5.56keV Fwtm = 15.24keV

ID : No close library match

NP418Th3.CHW

MCB # 1 ACQ 01-16-95 AT 22:19:55 RT : 25004.9 LT : 25000.0
No detector description was entered
NOPI-418 Th 1/18/95

ROI # 5-1 RANGE : 17 = 3.79keV to 67 = 3.92keV
AREA : Gross = 46 Net = 44 +/- 8
Could not properly fit the peak.

ROI # 5-2 RANGE : 162 = 4.18keV to 348 = 4.68keV
AREA : Gross = 38643 Net = 38200 +/- 227
CENTROID : 316.45 = 4.59keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.22keV

ID : No close library match

ROI # 5-3 RANGE : 503 = 5.09keV to 625 = 5.42keV
AREA : Gross = 3256 Net = 2826 +/- 106
CENTROID : 601.52 = 5.36keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.19keV

ID : No close library match

ROI # 5-4 RANGE : 634 = 5.44keV to 736 = 5.72keV
AREA : Gross = 3150 Net = 2429 +/- 119
CENTROID : 697.77 = 5.62keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.19keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP419U3.CHN
NP419TH3.CHN

Sample # NOPI-419
Analyst JDP

Sep. date 1/4/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.2608	0.2984	454.83	1/22/93	712	446.37404

Th-228/U-232= 0.5727428

Counting time for Th= 1339.82 (mins.)
Days btwn. sep. and count.= 13 (days)
CF for Th-228= 0.9867311

Counting time for U= 500.03 (mins.)
Days btwn. sep. and count.= 12 (days)
CF for U-232= 1.0116811

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
199	14824	3554	2873

U-238 counts	U-234 counts	U-232 counts
2731	4461	2414

Bkgd	Bkgd	Bkgd	bkgd
0	6	2	1

bkgd	bkgd	bkgd
3	5	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
199	14818	3246.502	2872

U-238* counts	U-234* counts	U-232sp counts
2728	4456	2374.266

U-238(dpm/g)= 586.82033 ± 16.39337
U-234(dpm/g)= 958.53058 ± 24.21908

Th-232(dpm/g)= 17.930285 ± 1.306144
Th-230(dpm/g)= 1335.1305 ± 24.93627

U-234/U-238= 1.6334311 ± 0.039687
Th-230/U-234= 1.3928929 ± 0.023786
Th-230/Th-232= 74.462312 ± 5.313805
U234/Th-232= 53.458747 ± 4.121844

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

U(ppb)= 786605.43

Th(ppb)= 73381.895

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP419K3.CHW

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-419 U 1/17/95

ROI # 11-1 RANGE : 39 = 396.63keV to 165 = 425.66keV
AREA : Gross = 2859 Net = 2731 +/- 73
CENTROID : 136.65 = 419.13keV
SHAPE : Fwhm = 7.70keV Fwtm = 13.34keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.091 +/- 0.00

ROI # 11-2 RANGE : 265 = 448.70keV to 414 = 483.02keV
AREA : Gross = 4498 Net = 4461 +/- 73
CENTROID : 383.46 = 475.99keV
SHAPE : Fwhm = 7.93keV Fwtm = 20.28keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.149 +/- 0.00

ROI # 11-3 RANGE : 527 = 509.06keV to 650 = 537.39keV
AREA : Gross = 2416 Net = 2414 +/- 50
CENTROID : 620.89 = 530.69keV
SHAPE : Fwhm = 10.24keV Fwtm = 18.68keV

ID : No close library match

NP419Th3.ctw

MCB # 1 ACQ 01-16-95 AT 22:19:55 RT : 80389.0 LT : 80375.8
No detector description was entered
NOPI-419 Th 1/18/95

ROI # 8-1 RANGE : 12 = 393.61keV to 72 = 407.30keV
AREA : Gross = 474 Net = 199 +/- 52
CENTROID : 18.26 = 395.04keV
SHAPE : Fwhm = 0.50keV Fwtm = 1.03keV

ID : No close library match

ROI # 8-2 RANGE : 226 = 442.45keV to 365 = 474.17keV
AREA : Gross = 15133 Net = 14824 +/- 147
CENTROID : 333.39 = 466.96keV
SHAPE : Fwhm = 8.67keV Fwtm = 16.37keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.184 +/- 0.00

ROI # 8-3 RANGE : 542 = 514.57keV to 686 = 547.44keV
AREA : Gross = 3865 Net = 3554 +/- 104
CENTROID : 656.72 = 540.75keV
SHAPE : Fwhm = 5.44keV Fwtm = 16.38keV

ID : No close library match

ROI # 8-4 RANGE : 696 = 549.72keV to 800 = 573.46keV
AREA : Gross = 3085 Net = 2873 +/- 80
CENTROID : 768.19 = 566.20keV
SHAPE : Fwhm = 6.18keV Fwtm = 12.36keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP420U3.CHN
NP420TH3.CHNSample # NOPI-420
Analyst JDP

Sep. date 1/4/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3412	0.2981	454.83	1/22/93	712	446.37404

Th-228/U-232= 0.5727428

Counting time for Th=	416.75	(mins.)
Days btwn. sep. and count.=	13	(days)
CF for Th-228=	0.9870448	

Counting time for U=	500.03	(mins.)
Days btwn. sep. and count.=	12	(days)
CF for U-232=	1.0116811	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
44	10224	1204	838

U-238 counts	U-234 counts	U-232 counts
3329	4276	1094

Bkgd	Bkgd	Bkgd	bkgd
0	0	10	6

bkgd	bkgd	bkgd
13	15	13

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
44	10224	1120.9967	832

U-238* counts	U-234* counts	U-232sp counts
3316	4261	1068.5185

U-238(dpm/g)=	1210.2758	±	42.17715
U-234(dpm/g)=	1555.1825	±	52.69155

Th-232(dpm/g)=	8.7671787	±	1.345636
Th-230(dpm/g)=	2037.1735	±	62.0711

U-234/U-238=	1.2849819	±	0.029701
Th-230/U-234=	1.3099257	±	0.023856
Th-230/Th-232=	232.36364	±	35.10543
U234/Th-232=	177.38688	±	27.8818

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

U(ppb)= 1622318.5

Th(ppb)= 35880.756

	Spike 25A	Spike 25B	Spide 25C
	10/9/92		
Th-228 (dpm/g)=	2559.3248	255.65752	25.5523

NP42003.CHN

MCB # 1 ACQ 01-16-95 AT 01:23:05 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-420 U 1/17/95

ROI # 7-1 RANGE : 49 to 156
AREA : Gross = 3410 Net = 3329 +/- 69
CENTROID : 127.36
SHAPE : Fwhm = 26.65 Channels Fwtm = 59.18 Channels

ROI # 7-2 RANGE : 286 to 405
AREA : Gross = 4306 Net = 4276 +/- 70
CENTROID : 377.27
SHAPE : Fwhm = 28.96 Channels Fwtm = 58.36 Channels

ROI # 7-3 RANGE : 542 to 641
AREA : Gross = 1119 Net = 1094 +/- 39
CENTROID : 616.85
SHAPE : Fwhm = 27.67 Channels Fwtm = 58.11 Channels

NP420Th3.CHW

MCB # 1 ACQ 01-16-95 AT 22:19:56 RT : 25004.9 LT : 25000.0
No detector description was entered
NOPI-420 Th 1/18/95

ROI # 10-1 RANGE : 17 = 398.26keV to 86 = 414.24keV
AREA : Gross = 45 Net = 44 +/- 7
Could not properly fit the peak.

ROI # 10-2 RANGE : 216 = 444.34keV to 356 = 476.76keV
AREA : Gross = 10225 Net = 10224 +/- 101
CENTROID : 328.15 = 470.31keV
SHAPE : Fwhm = 5.00keV Fwtm = 14.90keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.409 +/- 0.00

ROI # 10-3 RANGE : 576 = 527.71keV to 670 = 549.48keV
AREA : Gross = 1252 Net = 1204 +/- 44
CENTROID : 648.95 = 544.61keV
SHAPE : Fwhm = 4.18keV Fwtm = 14.27keV

ID : No close library match

ROI # 10-4 RANGE : 685 = 552.95keV to 788 = 576.81keV
AREA : Gross = 942 Net = 838 +/- 50
CENTROID : 761.07 = 570.57keV
SHAPE : Fwhm = 6.15keV Fwtm = 11.68keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP421U3.CHN
NP421TH3.CHN

Sample # **NOPI-421**
Analyst **JDP**

Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3235	0.2966	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th= **416.75** (mins.)
Days btwn. sep. and count.= **13** (days)
CF for Th-228= **0.9870448**

Counting time for U= **500.03** (mins.)
Days btwn. sep. and count.= **12** (days)
CF for U-232= **1.0116811**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
72	15947	1465	814

U-238 counts	U-234 counts	U-232 counts
5721	6776	1273

Bkgd	Bkgd	Bkgd	bkgd
0	1	10	12

bkgd	bkgd	bkgd
8	15	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
72	15946	1359.0333	802

U-238* counts	U-234* counts	U-232sp counts
5713	6761	1250.394

U-238(dpm/g)= **1869.8773** ± **57.94631**
U-234(dpm/g)= **2212.89** ± **67.59738**

Th-232(dpm/g)= **12.418177** ± **1.499028**
Th-230(dpm/g)= **2750.2812** ± **75.08326**

U-234/U-238= **1.1834413** ± **0.021248**
Th-230/U-234= **1.2428459** ± **0.018023**
Th-230/Th-232= **221.47222** ± **26.15961**
U234/Th-232= **178.19766** ± **22.18873**

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

U(ppb)= **2506483.8**

Th(ppb)= **50822.915**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= **2559.3248** **255.65752** **25.5523**

WP421 U3. <HN

MCB # 1 ACQ 01-15-95 AT 21:32:15 RT : 30001.8 LT : 30000.0
No detector description was entered
NOPI-421 U 1/17/95

ROI # 1-1 RANGE : 41 = 3.89keV to 166 = 4.20keV
AREA : Gross = 5848 Net = 5721 +/- 91
CENTROID : 142.00 = 4.14keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

ROI # 1-2 RANGE : 254 = 4.42keV to 391 = 4.76keV
AREA : Gross = 6914 Net = 6776 +/- 99
CENTROID : 370.95 = 4.71keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

ROI # 1-3 RANGE : 499 = 5.03keV to 612 = 5.31keV
AREA : Gross = 1301 Net = 1273 +/- 42
CENTROID : 588.97 = 5.25keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.15keV

ID : No close library match

NP421Th3.CHW

MCB # 1 ACQ 01-16-95 AT 22:19:55 RT : 25004.9 LT : 25000.0
No detector description was entered
NOPI-421 Th 1/18/95

ROI # 9-1 RANGE : 18 = 392.32keV to 72 = 404.80keV
AREA : Gross = 154 Net = 72 +/- 27
Could not properly fit the peak.

ROI # 9-2 RANGE : 193 = 432.77keV to 372 = 474.14keV
AREA : Gross = 16154 Net = 15947 +/- 148
CENTROID : 341.27 = 467.04keV
SHAPE : Fwhm = 9.73keV Fwtm = 16.82keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.638 +/- 0.01

ROI # 9-3 RANGE : 586 = 523.60keV to 689 = 547.40keV
AREA : Gross = 1570 Net = 1465 +/- 56
CENTROID : 665.03 = 541.86keV
SHAPE : Fwhm = 4.52keV Fwtm = 15.41keV

ID : No close library match

ROI # 9-4 RANGE : 717 = 553.88keV to 801 = 573.29keV
AREA : Gross = 1040 Net = 814 +/- 62
CENTROID : 776.34 = 567.59keV
SHAPE : Fwhm = 4.32keV Fwtm = 14.17keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP422U3.CHN
NP422TH3.CHNSample # **NOPI-422**
Analyst **JDP**Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3056	0.299	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th=	592.66	(mins.)
Days btwn. sep. and count.=	12	(days)
CF for Th-228=	0.9879646	

Counting time for U=	500.03	(mins.)
Days btwn. sep. and count.=	12	(days)
CF for U-232=	1.0116811	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
87	13837	2220	1433

U-238 counts	U-234 counts	U-232 counts
3070	3306	1060

Bkgd	Bkgd	Bkgd	bkgd
6	14	12	6

bkgd	bkgd	bkgd
1	4	1

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
81	13823	2077.3455	1427

U-238* counts	U-234* counts	U-232sp counts
3069	3302	1046.7725

U-238(dpm/g)=	1280.4462	±	45.61569
U-234(dpm/g)=	1377.6583	±	48.62717

Th-232(dpm/g)=	9.753325	±	1.065959
Th-230(dpm/g)=	1664.4471	±	38.05439

U-234/U-238=	1.0759205	±	0.026967
Th-230/U-234=	1.2081712	±	0.023388
Th-230/Th-232=	170.65432	±	18.35349
U234/Th-232=	141.25012	±	16.22261

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

U(ppb)= **1716378.7**Th(ppb)= **39916.681**

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	2559.3248	255.65752	25.5523

NP422u3.CHW

MCB # 1 ACQ 01-16-95 AT 01:23:05 RT : 30002.1 LT : 30000.0
No detector description was entered
NOPI-422 U 1/17/95

ROI # 8-1 RANGE : 35 = 398.86keV to 152 = 425.56keV
AREA : Gross = 3217 Net = 3070 +/- 76
CENTROID : 122.62 = 418.85keV
SHAPE : Fwhm = 9.18keV Fwtm = 14.21keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.102 +/- 0.00

ROI # 8-2 RANGE : 279 = 454.55keV to 401 = 482.39keV
AREA : Gross = 3384 Net = 3306 +/- 69
CENTROID : 374.36 = 476.31keV
SHAPE : Fwhm = 8.34keV Fwtm = 15.20keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.110 +/- 0.00

ROI # 8-3 RANGE : 544 = 515.03keV to 640 = 536.94keV
AREA : Gross = 1084 Net = 1060 +/- 38
CENTROID : 615.38 = 531.32keV
SHAPE : Fwhm = 3.37keV Fwtm = 12.60keV

ID : No close library match

NP422Th3.CHW

MCB # 1 ACQ 01-16-95 AT 03:09:25 RT : 35559.6 LT : 35554.3
No detector description was entered
NOPI-422 Th 1/17/95

ROI # 14-1 RANGE : 18 = 393.39keV to 67 = 404.67keV
AREA : Gross = 126 Net = 87 +/- 19
CENTROID : 52.61 = 401.35keV
SHAPE : Fwhm = 1.08keV Fwtm = 2.92keV

ID : No close library match

ROI # 14-2 RANGE : 202 = 435.74keV to 368 = 473.95keV
AREA : Gross = 13977 Net = 13837 +/- 133
CENTROID : 335.53 = 466.48keV
SHAPE : Fwhm = 7.81keV Fwtm = 17.23keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.389 +/- 0.00

ROI # 14-3 RANGE : 560 = 518.15keV to 685 = 546.92keV
AREA : Gross = 2470 Net = 2220 +/- 85
CENTROID : 657.71 = 540.64keV
SHAPE : Fwhm = 4.95keV Fwtm = 15.72keV

ID : No close library match

ROI # 14-4 RANGE : 696 = 549.45keV to 796 = 572.47keV
AREA : Gross = 1637 Net = 1433 +/- 68
CENTROID : 767.94 = 566.01keV
SHAPE : Fwhm = 6.22keV Fwtm = 13.87keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP423U3.CHN
NP423TH3.CHN

Sample # **NOPI-423**
Analyst **JDP**

Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.3347	0.2981	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th= **1122.25** (mins.)
Days btwn. sep. and count.= **13** (days)
CF for Th-228= **0.986805**

Counting time for U= **500.03** (mins.)
Days btwn. sep. and count.= **12** (days)
CF for U-232= **1.0116811**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
94	6736	3127	1584

U-238 counts	U-234 counts	U-232 counts
3101	3465	3554

Bkgd	Bkgd	Bkgd	bkgd
5	12	25	25

bkgd	bkgd	bkgd
1	8	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
89	6724	2970.7462	1559

U-238* counts	U-234* counts	U-232sp counts
3100	3457	3501.1033

U-238(dpm/g)= **352.01564** ± **8.650205**
U-234(dpm/g)= **392.55422** ± **9.371885**

Th-232(dpm/g)= **6.8216476** ± **0.714096**
Th-230(dpm/g)= **515.37931** ± **11.15235**

U-234/U-238= **1.1151613** ± **0.027567**
Th-230/U-234= **1.3128869** ± **0.027447**
Th-230/Th-232= **75.550562** ± **7.846628**
U234/Th-232= **57.545367** ± **6.178579**

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

U(ppb)= **471860.64**

Th(ppb)= **27918.431**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP423u3.cHW

MCB # 1 ACQ 01-15-95 AT 21:32:16 RT : 30001.8 LT : 30000.0
No detector description was entered
NOPI-423 U 1/17/95

ROI # 5-1 RANGE : 61 = 3.91keV to 169 = 4.20keV
AREA : Gross = 3193 Net = 3101 +/- 68
CENTROID : 144.31 = 4.13keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.16keV

ID : No close library match

ROI # 5-2 RANGE : 259 = 4.44keV to 383 = 4.77keV
AREA : Gross = 3591 Net = 3465 +/- 77
CENTROID : 359.09 = 4.71keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.18keV

ID : No close library match

ROI # 5-3 RANGE : 470 = 5.00keV to 589 = 5.32keV
AREA : Gross = 3614 Net = 3554 +/- 69
CENTROID : 566.30 = 5.26keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.16keV

ID : No close library match

NP423Th.CHW

MCB # 1 ACQ 01-16-95 AT 03:09:26 RT : 67335.1 LT : 67332.6
No detector description was entered
NOPI-423 Th 1/17/95

ROI # 15-1 RANGE : 15 = 394.06keV to 78 = 408.52keV
AREA : Gross = 167 Net = 94 +/- 28
CENTROID : 39.25 = 399.63keV
SHAPE : Fwhm = 1.35keV Fwtm = 4.51keV

ID : No close library match

ROI # 15-2 RANGE : 215 = 439.96keV to 361 = 473.46keV
AREA : Gross = 6829 Net = 6736 +/- 94
CENTROID : 334.06 = 467.28keV
SHAPE : Fwhm = 9.40keV Fwtm = 17.29keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.100 +/- 0.00

ROI # 15-3 RANGE : 565 = 520.28keV to 682 = 547.13keV
AREA : Gross = 3392 Net = 3127 +/- 90
CENTROID : 656.15 = 541.20keV
SHAPE : Fwhm = 5.56keV Fwtm = 16.92keV

ID : No close library match

ROI # 15-4 RANGE : 696 = 550.34keV to 792 = 572.37keV
AREA : Gross = 1802 Net = 1584 +/- 70
CENTROID : 765.58 = 566.31keV
SHAPE : Fwhm = 7.82keV Fwtm = 13.69keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP424U.CHN
NP424TH.CHN

Sample # **NOPI-424**
Analyst **JDP**

Sep. date **1/23/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5122	0.9995	454.83	1/22/93	731	446.15055

Th-228/U-232= **0.5817043**

Counting time for Th= **971.5** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for Th-228= **0.997684**

Counting time for U= **1155.647** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.0023159**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
137	1188	8042	1171

U-238 counts	U-234 counts	U-232 counts
1717	1810	26055

Bkgd	Bkgd	Bkgd	bkgd
15	44	25	47

bkgd	bkgd	bkgd
6	14	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
122	1144	7853.8999	1124

U-238* counts	U-234* counts	U-232sp counts
1711	1796	25984.822

U-238(dpm/g)= **57.326433** ± **1.428328**
U-234(dpm/g)= **60.174327** ± **1.462702**

Th-232(dpm/g)= **7.86686** ± **0.677812**
Th-230(dpm/g)= **73.767933** ± **2.292864**

U-234/U-238= **1.0496786** ± **0.035362**
Th-230/U-234= **1.2259038** ± **0.045775**
Th-230/Th-232= **9.3770492** ± **0.846069**
U234/Th-232= **7.6490908** ± **0.684775**

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

U(ppb)= **76843.423**

Th(ppb)= **32196.091**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2598.0684 259.52771 25.939115

NP424 U.C.H.N

MCB # 1 ACQ 01-25-95 AT 13:11:41 RT : 69338.8 LT : 69331.9
No detector description was entered
NOPI-424 U 1/26/95

ROI # 7-1 RANGE : 80 to 157
AREA : Gross = 1756 Net = 1717 +/- 47
CENTROID : 130.62
SHAPE : Fwhm = 19.15 Channels Fwtm = 46.05 Channels

ROI # 7-2 RANGE : 329 to 407
AREA : Gross = 1967 Net = 1810 +/- 61
CENTROID : 379.17
SHAPE : Fwhm = 16.97 Channels Fwtm = 48.16 Channels

ROI # 7-3 RANGE : 547 to 637
AREA : Gross = 26310 Net = 26055 +/- 172
CENTROID : 614.69
SHAPE : Fwhm = 17.66 Channels Fwtm = 51.23 Channels

MCB # 1 ACQ 01-25-95 AT 16:49:44 RT : 58290.3 LT : 58283.8
No detector description was entered
NOPI-424 Th 1/26/95

ROI # 16-1 RANGE : 24 = 393.82keV to 71 = 404.56keV
AREA : Gross = 168 Net = 137 +/- 19
CENTROID : 57.38 = 401.45keV
SHAPE : Fwhm = 0.86keV Fwtm = 4.21keV

ID : No close library match

ROI # 16-2 RANGE : 253 = 446.12keV to 369 = 472.60keV
AREA : Gross = 1190 Net = 1188 +/- 35
CENTROID : 347.14 = 467.61keV
SHAPE : Fwhm = 3.94keV Fwtm = 14.53keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.020 +/- 0.00

ROI # 16-3 RANGE : 572 = 518.96keV to 698 = 547.73keV
AREA : Gross = 8249 Net = 8042 +/- 111
CENTROID : 669.67 = 541.26keV
SHAPE : Fwhm = 4.66keV Fwtm = 15.13keV

ID : No close library match

ROI # 16-4 RANGE : 716 = 551.84keV to 814 = 574.22keV
AREA : Gross = 1172 Net = 1171 +/- 34
CENTROID : 780.68 = 566.61keV
SHAPE : Fwhm = 5.20keV Fwtm = 11.72keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP425U3.CHN
NP425TH3.CHN

Sample # **NOPI-425**
Analyst **JDP**

Sep. date **1/4/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.2607	0.2978	454.83	1/22/93	712	446.37404

Th-228/U-232= **0.5727428**

Counting time for Th= **1123.3** (mins.)
Days btwn. sep. and count.= **13** (days)
CF for Th-228= **0.9868047**

Counting time for U= **500.03** (mins.)
Days btwn. sep. and count.= **12** (days)
CF for U-232= **1.0116811**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
92	5051	3034	1531

U-238 counts	U-234 counts	U-232 counts
3147	3487	4781

Bkgd	Bkgd	Bkgd	bkgd
26	110	35	20

bkgd	bkgd	bkgd
12	14	18

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
66	4941	2891.9481	1511

U-238* counts	U-234* counts	U-232sp counts
3135	3473	4708.0053

U-238(dpm/g)= **339.53393** ± **7.793944**
U-234(dpm/g)= **376.14078** ± **8.376552**

Th-232(dpm/g)= **6.6649312** ± **0.705324**
Th-230(dpm/g)= **498.96099** ± **11.46067**

U-234/U-238= **1.107815** ± **0.027238**
Th-230/U-234= **1.3265272** ± **0.029207**
Th-230/Th-232= **74.863636** ± **7.875835**
U234/Th-232= **56.435809** ± **6.103189**

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

U(ppb)= **455129.49**

Th(ppb)= **27277.05**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2559.3248 255.65752 25.5523

NP42543.CHN

MCB # 1 ACQ 01-15-95 AT 21:32:16 RT : 30001.8 LT : 30000.0
No detector description was entered
NOPI-425 U 1/17/95

ROI # 6-1 RANGE : 64 = 4.00keV to 158 = 4.25keV
AREA : Gross = 3148 Net = 3147 +/- 56
CENTROID : 134.73 = 4.19keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.14keV

ID : No close library match

ROI # 6-2 RANGE : 277 = 4.56keV to 380 = 4.84keV
AREA : Gross = 3513 Net = 3487 +/- 63
CENTROID : 353.29 = 4.76keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

ROI # 6-3 RANGE : 481 = 5.10keV to 584 = 5.37keV
AREA : Gross = 4782 Net = 4781 +/- 69
CENTROID : 559.36 = 5.31keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.13keV

ID : No close library match

NP425Th.c#W

MCB # 1 ACQ 01-16-95 AT 03:09:26 RT : 67398.2 LT : 67395.7
No detector description was entered
NOPI-425 Th 1/17/95

ROI # 16-1 RANGE : 20 = 392.91keV to 71 = 404.56keV
AREA : Gross = 105 Net = 92 +/- 14
Could not properly fit the peak.

ROI # 16-2 RANGE : 224 = 439.49keV to 373 = 473.52keV
AREA : Gross = 5052 Net = 5051 +/- 71
CENTROID : 344.66 = 467.05keV
SHAPE : Fwhm = 5.64keV Fwtm = 15.61keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.075 +/- 0.00

ROI # 16-3 RANGE : 567 = 517.82keV to 699 = 547.96keV
AREA : Gross = 3169 Net = 3034 +/- 77
CENTROID : 668.83 = 541.07keV
SHAPE : Fwhm = 4.92keV Fwtm = 16.49keV

ID : No close library match

ROI # 16-4 RANGE : 713 = 551.16keV to 808 = 572.85keV
AREA : Gross = 1627 Net = 1531 +/- 55
CENTROID : 784.93 = 567.58keV
SHAPE : Fwhm = 3.14keV Fwtm = 12.77keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP113U3.CHN
NP113TH3.CHN

Sample # **NOPI-113**
Analyst **JDP**

Sep. date **1/30/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4982	0.9914	454.83	1/22/93	738	446.0682

Th-228/U-232= **0.5849683**

Counting time for Th=	940.75	(mins.)	Counting time for U=	940.73	(mins.)
Days btwn. sep. and count.=	64	(days)	Days btwn. sep. and count.=	64	(days)
CF for Th-228=	0.9381802		CF for U-232=	1.0600729	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
260	118937	11988	3151

U-238 counts	U-234 counts	U-232 counts
6025	6045	1057

Bkgd	Bkgd	Bkgd	bkgd
4	7	14	25

bkgd	bkgd	bkgd
9	13	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
256	118930	12330.412	3126

U-238* counts	U-234* counts	U-232sp counts
6016	6032	989.55463

U-238(dpm/g)= **5396.529** ± **179.9602**
U-234(dpm/g)= **5410.882** ± **180.3943**

Th-232(dpm/g)= **10.78056** ± **0.675793**
Th-230(dpm/g)= **5008.327** ± **47.99236**

U-234/U-238= **1.00266** ± **0.018253**
Th-230/U-234= **0.925603** ± **0.012204**
Th-230/Th-232= **464.5703** ± **28.8429**
U234/Th-232= **501.9112** ± **35.63594**

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

U(ppb)= **7233797**

Th(ppb)= **44120.75**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2612.1641 260.93577 26.079847

NP113U3 CHW

MCB # 1 ACQ 04-03-95 AT 14:58:45 RT : 56444.1 LT : 56437.2
No detector description was entered
NOPI-113 U 4/4/95

ROI # 7-1 RANGE : 39 to 149
AREA : Gross = 6302 Net = 6025 +/- 104
CENTROID : 125.70
SHAPE : Fwhm = 40.23 Channels Fwtm = 69.15 Channels

ROI # 7-2 RANGE : 298 to 400
AREA : Gross = 6163 Net = 6045 +/- 89
CENTROID : 371.89
SHAPE : Fwhm = 40.46 Channels Fwtm = 68.78 Channels

ROI # 7-3 RANGE : 565 to 635
AREA : Gross = 1199 Net = 1057 +/- 51
CENTROID : 613.70
SHAPE : Fwhm = 19.92 Channels Fwtm = 52.34 Channels

NP113Th3 CHN

MCB # 1 ACQ 04-03-95 AT 14:58:44 RT : 56445.1 LT : 56438.2
No detector description was entered
NOPI-113 Th 4/4/95

ROI # 1-1 RANGE : 21 to 88
AREA : Gross = 326 Net = 260 +/- 31
CENTROID : 70.80
SHAPE : Fwhm = 3.30 Channels Fwtm = 38.68 Channels

ROI # 1-2 RANGE : 212 to 355
AREA : Gross = 119275 Net = 118937 +/- 356
CENTROID : 335.72
SHAPE : Fwhm = 16.31 Channels Fwtm = 49.19 Channels

ROI # 1-3 RANGE : 554 to 652
AREA : Gross = 12270 Net = 11988 +/- 128
CENTROID : 632.13
SHAPE : Fwhm = 15.51 Channels Fwtm = 51.90 Channels

ROI # 1-4 RANGE : 671 to 759
AREA : Gross = 5257 Net = 3151 +/- 180
CENTROID : 734.92
SHAPE : Fwhm = 20.84 Channels Fwtm = 46.99 Channels

U and Th Isotope Activities

FILENAME= NP114U.CHN
NP114TH.CHNSample # NOPI-114
Analyst JDP

Sep. date 1/23/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.5039	1.0041	454.83	1/22/93	731	446.15055	0.5817043

Counting time for Th=	959.05	(mins.)	Counting time for U=	1000.1	(mins.)
Days btwn. sep. and count.=	2	(days)	Days btwn. sep. and count.=	2	(days)
CF for Th-228=	0.9976883		CF for U-232=	1.0022639	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
194	98361	11539	3055

U-238 counts	U-234 counts	U-232 counts
4881	4822	974

Bkgd	Bkgd	Bkgd	bkgd
2	5	12	23

bkgd	bkgd	bkgd
2	4	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
192	98356	11200.64	3032

U-238* counts	U-234* counts	U-232sp counts
4879	4818	959.82709

$$\begin{aligned} \text{U-238(dpm/g)} &= 4519.099 \pm 158.5921 \\ \text{U-234(dpm/g)} &= 4462.5987 \pm 156.7686 \end{aligned}$$

$$\begin{aligned} \text{Th-232(dpm/g)} &= 8.8649179 \pm 0.641792 \\ \text{Th-230(dpm/g)} &= 4541.2389 \pm 44.68663 \end{aligned}$$

$$\begin{aligned} \text{U-234/U-238} &= 0.9874974 \pm 0.02005 \\ \text{Th-230/U-234} &= 1.0176221 \pm 0.015009 \\ \text{Th-230/Th-232} &= 512.27083 \pm 36.81515 \\ \text{U234/Th-232} &= 503.39989 \pm 40.50846 \end{aligned}$$

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

$$\text{U(ppb)} = 6057642.6$$

$$\text{Th(ppb)} = 36280.766$$

	Spike 25A	Spike 25B	Spide 25C
	10/9/92		
Th-228 (dpm/g)=	2598.0684	259.52771	25.939115

NP114U.CHW

MCB # 1 ACQ 01-25-95 AT 13:11:41 RT : 60006.0 LT : 60000.0
No detector description was entered
NOPI-114 U 1/26/95

ROI # 9-1 RANGE : 39 = 397.17keV to 156 = 424.22keV
AREA : Gross = 5088 Net = 4881 +/- 94
CENTROID : 134.74 = 419.30keV
SHAPE : Fwhm = 6.86keV Fwtm = 14.10keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.081 +/- 0.00

ROI # 9-2 RANGE : 292 = 455.65keV to 409 = 482.69keV
AREA : Gross = 4958 Net = 4822 +/- 86
CENTROID : 382.42 = 476.55keV
SHAPE : Fwhm = 8.92keV Fwtm = 15.58keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.080 +/- 0.00

ROI # 9-3 RANGE : 575 = 521.06keV to 640 = 536.08keV
AREA : Gross = 1073 Net = 974 +/- 44
CENTROID : 620.51 = 531.58keV
SHAPE : Fwhm = 5.37keV Fwtm = 11.91keV

ID : No close library match

MCB # 1 ACQ 01-25-95 AT 16:49:43 RT : 57542.9 LT : 57536.3
No detector description was entered
NOPI-114 Th 1/26/95

ROI # 2-1 RANGE : 33 = 3.87keV to 94 = 4.02keV
AREA : Gross = 254 Net = 194 +/- 27
CENTROID : 75.57 = 3.97keV
SHAPE : Fwhm = 0.01keV Fwtm = 0.06keV

ID : No close library match

ROI # 2-2 RANGE : 211 = 4.32keV to 365 = 4.71keV
AREA : Gross = 98515 Net = 98361 +/- 320
CENTROID : 343.76 = 4.66keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 2-3 RANGE : 559 = 5.20keV to 664 = 5.47keV
AREA : Gross = 11681 Net = 11539 +/- 118
CENTROID : 639.27 = 5.41keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.14keV

ID : No close library match

ROI # 2-4 RANGE : 681 = 5.52keV to 803 = 5.83keV
AREA : Gross = 3327 Net = 3055 +/- 91
CENTROID : 746.21 = 5.68keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.18keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP115U.CHN
NP115TH.CHNSample # NOPI-115
Analyst JDP

Sep. date 1/30/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.4982	0.99	454.83	1/22/93	738	446.06824	0.5849683

Counting time for Th=	1333.468	(mins.)	Counting time for U=	1227.415	(mins.)
Days btwn. sep. and count.=	4	(days)	Days btwn. sep. and count.=	2	(days)
CF for Th-228=	0.9955824		CF for U-232=	1.0023399	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
118	45514	7466	3113

U-238 counts	U-234 counts	U-232 counts
5344	5175	1568

Bkgd	Bkgd	Bkgd	bkgd
4	17	27	69

bkgd	bkgd	bkgd
7	6	11

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
114	45497	7195.9603	3044

U-238* counts	U-234* counts	U-232sp counts
5337	5169	1553.3653

U-238(dpm/g)=	3045.4844	±	87.46856
U-234(dpm/g)=	2949.6175	±	85.02841

Th-232(dpm/g)=	8.2145009	±	0.762159
Th-230(dpm/g)=	3278.3785	±	40.93536

U-234/U-238=	0.9685216	±	0.018889
Th-230/U-234=	1.1114588	±	0.016305
Th-230/Th-232=	399.09649	±	36.78741
U234/Th-232=	359.07447	±	34.88665

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

U(ppb)= 4082330.5

Th(ppb)= 33618.854

	Spike 25A	Spike 25B	Spide 25C
	10/9/92		
Th-228 (dpm/g)=	2612.1641	260.93577	26.079847

NP115U.CHA

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 73644.9 LT : 73640.1
No detector description was entered
NOPI-115 U 2/2/95

ROI # 8-1 RANGE : 46 = 401.37keV to 155 = 426.24keV
AREA : Gross = 5509 Net = 5344 +/- 91
CENTROID : 127.82 = 420.04keV
SHAPE : Fwhm = 6.68keV Fwtm = 14.18keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.073 +/- 0.00

ROI # 8-2 RANGE : 308 = 461.16keV to 407 = 483.76keV
AREA : Gross = 5325 Net = 5175 +/- 87
CENTROID : 376.79 = 476.86keV
SHAPE : Fwhm = 7.05keV Fwtm = 14.30keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.070 +/- 0.00

ROI # 8-3 RANGE : 565 = 519.82keV to 643 = 537.62keV
AREA : Gross = 1738 Net = 1568 +/- 60
CENTROID : 616.79 = 531.64keV
SHAPE : Fwhm = 4.52keV Fwtm = 11.98keV

ID : No close library match

NP 115Th.c Hw

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-115 Th 2/6/95

ROI # 7-1 RANGE : 32 to 62
AREA : Gross = 150 Net = 118 +/- 16
CENTROID : 45.35
SHAPE : Fwhm = 3.57 Channels Fwtm = 24.94 Channels

ROI # 7-2 RANGE : 190 to 368
AREA : Gross = 45871 Net = 45514 +/- 236
CENTROID : 340.17
SHAPE : Fwhm = 20.81 Channels Fwtm = 60.11 Channels

ROI # 7-3 RANGE : 580 to 685
AREA : Gross = 7625 Net = 7466 +/- 101
CENTROID : 660.85
SHAPE : Fwhm = 19.06 Channels Fwtm = 67.34 Channels

ROI # 7-4 RANGE : 713 to 827
AREA : Gross = 3344 Net = 3113 +/- 85
CENTROID : 772.56
SHAPE : Fwhm = 29.29 Channels Fwtm = 80.81 Channels

U and Th Isotope Activities

FILENAME= NP116U.CHN
NP116TH.CHN

Sample # NOPI-116
Analyst JDP

Sep. date 1/30/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5051	0.9876	454.83	1/22/93	738	446.06824

Th-228/U-232= 0.5849683

Counting time for Th= 1333.468 (mins.)
Days btwn. sep. and count.= 4 (days)
CF for Th-228= 0.9955824

Counting time for U= 833.398 (mins.)
Days btwn. sep. and count.= 2 (days)
CF for U-232= 1.0022081

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
167	55000	10119	3179

U-238 counts	U-234 counts	U-232 counts
7388	7809	2391

Bkgd	Bkgd	Bkgd	bkgd
2	17	23	69

bkgd	bkgd	bkgd
2	4	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
165	54983	9810.2363	3110

U-238* counts	U-234* counts	U-232sp counts
7386	7805	2377.7497

U-238(dpm/g)= 2709.2444 ± 63.74442
U-234(dpm/g)= 2862.937 ± 66.91514

Th-232(dpm/g)= 8.581077 ± 0.669481
Th-230(dpm/g)= 2859.4749 ± 30.93072

U-234/U-238= 1.0567289 ± 0.017151
Th-230/U-234= 0.9987907 ± 0.012078
Th-230/Th-232= 333.2303 ± 25.82525
U234/Th-232= 333.63376 ± 27.1725

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

U(ppb)= 3631616.5

Th(ppb)= 35119.112

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2612.1641 260.93577 26.079847

NP116U.CHW

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 50003.9 LT : 50000.0
No detector description was entered
NOPI-116 U 2/2/95

ROI # 9-1 RANGE : 44 = 398.33keV to 159 = 424.91keV
AREA : Gross = 10520 Net = 7388 +/- 255
CENTROID : 131.14 = 418.47keV
SHAPE : Fwhm = 6.57keV Fwtm = 14.57keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.148 +/- 0.01

ROI # 9-2 RANGE : 286 = 454.26keV to 408 = 482.46keV
AREA : Gross = 10105 Net = 7809 +/- 229
CENTROID : 379.26 = 475.82keV
SHAPE : Fwhm = 10.24keV Fwtm = 17.30keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.156 +/- 0.00

ROI # 9-3 RANGE : 560 = 517.59keV to 646 = 537.47keV
AREA : Gross = 3189 Net = 2391 +/- 115
CENTROID : 616.98 = 530.76keV
SHAPE : Fwhm = 8.45keV Fwtm = 13.69keV

ID : No close library match

NOPI116Th.cta

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-116 Th 2/6/95

ROI # 8-1 RANGE : 32 = 398.17keV to 71 = 407.07keV
AREA : Gross = 377 Net = 167 +/- 37
CENTROID : 46.53 = 401.49keV
SHAPE : Fwhm = 1.64keV Fwtm = 2.03keV

ID : No close library match

ROI # 8-2 RANGE : 168 = 429.21keV to 367 = 474.63keV
AREA : Gross = 56031 Net = 55000 +/- 297
CENTROID : 339.16 = 468.28keV
SHAPE : Fwhm = 5.51keV Fwtm = 14.61keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.688 +/- 0.00

ROI # 8-3 RANGE : 556 = 517.77keV to 689 = 548.12keV
AREA : Gross = 10588 Net = 10119 +/- 142
CENTROID : 661.90 = 541.94keV
SHAPE : Fwhm = 4.68keV Fwtm = 15.71keV

ID : No close library match

ROI # 8-4 RANGE : 710 = 552.92keV to 833 = 580.99keV
AREA : Gross = 3469 Net = 3179 +/- 94
CENTROID : 773.05 = 567.31keV
SHAPE : Fwhm = 6.24keV Fwtm = 18.96keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP117U.CHN
NP117TH.CHN

Sample # **NOPI-117**
Analyst **JDP**

Sep. date **1/30/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5071	0.9938	454.83	1/22/93	738	446.06824

Th-228/U-232= **0.5849683**

Counting time for Th= **1333.468** (mins.)
Days btwn. sep. and count.= **4** (days)
CF for Th-228= **0.9955824**

Counting time for U= **833.398** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.0022081**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
279	46444	19028	5811

U-238 counts	U-234 counts	U-232 counts
6688	6788	5602

Bkgd	Bkgd	Bkgd	bkgd
1	5	36	88

bkgd	bkgd	bkgd
1	1	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
278	46439	18493.606	5723

U-238* counts	U-234* counts	U-232sp counts
6687	6787	5581.6751

U-238(dpm/g)= **1047.3057** ± **18.96836**
U-234(dpm/g)= **1062.9675** ± **19.18727**

Th-232(dpm/g)= **7.687094** ± **0.463576**
Th-230(dpm/g)= **1284.1042** ± **11.05266**

U-234/U-238= **1.0149544** ± **0.017487**
Th-230/U-234= **1.2080371** ± **0.015698**
Th-230/Th-232= **167.04676** ± **10.03082**
U234/Th-232= **138.2795** ± **8.704593**

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

U(ppb)= **1403864.7**

Th(ppb)= **31460.377**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2612.1641 260.93577 26.079847

NP117U CHN

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 50003.9 LT : 50000.0
No detector description was entered
NOPI-117 U 2/2/95

ROI # 10-1 RANGE : 38 = 403.12keV to 149 = 428.82keV
AREA : Gross = 6744 Net = 6688 +/- 88
CENTROID : 117.00 = 421.41keV
SHAPE : Fwhm = 7.97keV Fwtm = 15.77keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.134 +/- 0.00

ROI # 10-2 RANGE : 282 = 459.63keV to 396 = 486.03keV
AREA : Gross = 6926 Net = 6788 +/- 96
CENTROID : 365.37 = 478.93keV
SHAPE : Fwhm = 8.91keV Fwtm = 15.40keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.136 +/- 0.00

ROI # 10-3 RANGE : 544 = 520.30keV to 630 = 540.22keV
AREA : Gross = 6108 Net = 5602 +/- 112
CENTROID : 603.51 = 534.08keV
SHAPE : Fwhm = 9.08keV Fwtm = 14.62keV

ID : No close library match

NP117 Th. C tw

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-117 Th 2/6/95

ROI # 9-1 RANGE : 20 = 392.78keV to 75 = 405.50keV
AREA : Gross = 439 Net = 279 +/- 40
CENTROID : 57.46 = 401.44keV
SHAPE : Fwhm = 2.65keV Fwtm = 6.51keV

ID : No close library match

ROI # 9-2 RANGE : 230 = 441.32keV to 376 = 475.06keV
AREA : Gross = 46613 Net = 46444 +/- 225
CENTROID : 346.02 = 468.13keV
SHAPE : Fwhm = 4.57keV Fwtm = 13.12keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.581 +/- 0.00

ROI # 9-3 RANGE : 589 = 524.29keV to 695 = 548.79keV
AREA : Gross = 19295 Net = 19028 +/- 153
CENTROID : 667.14 = 542.35keV
SHAPE : Fwhm = 4.46keV Fwtm = 14.70keV

ID : No close library match

ROI # 9-4 RANGE : 710 = 552.26keV to 834 = 580.92keV
AREA : Gross = 6123 Net = 5811 +/- 110
CENTROID : 777.00 = 567.74keV
SHAPE : Fwhm = 6.52keV Fwtm = 13.90keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP118U.CHN
NP118TH.CHN

Sample # **NOPI-118**
Analyst **JDP**

Sep. date **1/30/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5027	0.9974	454.83	1/22/93	738	446.06824

Th-228/U-232= **0.5849683**

Counting time for Th= **1333.468** (mins.)
Days btwn. sep. and count.= **4** (days)
CF for Th-228= **0.9955824**

Counting time for U= **833.398** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.0022081**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
264	29905	17222	4523

U-238 counts	U-234 counts	U-232 counts
5994	6496	7182

Bkgd	Bkgd	Bkgd	bkgd
1	2	30	105

bkgd	bkgd	bkgd
1	5	15

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
263	29903	16770.091	4418

U-238* counts	U-234* counts	U-232sp counts
5993	6491	7151.2094

U-238(dpm/g)= **741.69708** ± **12.97589**
U-234(dpm/g)= **803.32984** ± **13.75497**

Th-232(dpm/g)= **8.1192221** ± **0.503519**
Th-230(dpm/g)= **923.15247** ± **8.83069**

U-234/U-238= **1.0830969** ± **0.019398**
Th-230/U-234= **1.1491574** ± **0.01573**
Th-230/Th-232= **113.69962** ± **7.028548**
U234/Th-232= **98.941725** ± **6.365516**

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

U(ppb)= **994210.53**

Th(ppb)= **33228.914**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2612.1641 260.93577 26.079847

NP118U.CHN

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 50003.9 LT : 50000.0
No detector description was entered
NOPI-118 U 2/2/95

ROI # 11-1 RANGE : 62 = 401.93keV to 169 = 426.58keV
AREA : Gross = 5994 Net = 5994 +/- 77
CENTROID : 137.82 = 419.40keV
SHAPE : Fwhm = 6.76keV Fwtm = 13.23keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.120 +/- 0.00

ROI # 11-2 RANGE : 305 = 457.91keV to 416 = 483.48keV
AREA : Gross = 6606 Net = 6496 +/- 92
CENTROID : 389.47 = 477.37keV
SHAPE : Fwhm = 7.12keV Fwtm = 13.45keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.130 +/- 0.00

ROI # 11-3 RANGE : 573 = 519.65keV to 654 = 538.31keV
AREA : Gross = 7538 Net = 7182 +/- 108
CENTROID : 626.38 = 531.95keV
SHAPE : Fwhm = 5.25keV Fwtm = 12.93keV

ID : No close library match

WP118Th. (HW)

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-118 Th 2/6/95

ROI # 10-1 RANGE : 10 = 396.63keV to 60 = 408.21keV
AREA : Gross = 392 Net = 264 +/- 35
CENTROID : 40.70 = 403.75keV
SHAPE : Fwhm = 0.90keV Fwtm = 5.01keV

ID : No close library match

ROI # 10-2 RANGE : 219 = 445.04keV to 364 = 478.62keV
AREA : Gross = 29997 Net = 29905 +/- 179
CENTROID : 332.26 = 471.26keV
SHAPE : Fwhm = 4.49keV Fwtm = 13.09keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.374 +/- 0.00

ROI # 10-3 RANGE : 559 = 523.77keV to 683 = 552.49keV
AREA : Gross = 17472 Net = 17222 +/- 149
CENTROID : 652.47 = 545.42keV
SHAPE : Fwhm = 4.21keV Fwtm = 14.87keV

ID : No close library match

ROI # 10-4 RANGE : 707 = 558.05keV to 829 = 586.30keV
AREA : Gross = 4814 Net = 4523 +/- 101
CENTROID : 763.98 = 571.24keV
SHAPE : Fwhm = 5.12keV Fwtm = 11.42keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP119U.CHN
NP119TH.CHN

Sample # **NOPI-119**
Analyst **JDP**

Sep. date **1/23/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.506	1.002	454.83	1/22/93	731	446.15055

Th-228/U-232= **0.5817043**

Counting time for Th= **960.16** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for Th-228= **0.9976879**

Counting time for U= **500.05** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.0020966**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
292	27130	17197	2322

U-238 counts	U-234 counts	U-232 counts
3567	3707	3907

Bkgd	Bkgd	Bkgd	bkgd
2	2	3	1

bkgd	bkgd	bkgd
1	1	5

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
290	27128	16820.548	2321

U-238* counts	U-234* counts	U-232sp counts
3566	3706	3893.8362

U-238(dpm/g)= **809.10019** ± **18.73724**
U-234(dpm/g)= **840.8652** ± **19.27968**

Th-232(dpm/g)= **8.8605116** ± **0.522906**
Th-230(dpm/g)= **828.85503** ± **8.079072**

U-234/U-238= **1.0392597** ± **0.024375**
Th-230/U-234= **0.9857169** ± **0.01726**
Th-230/Th-232= **93.544828** ± **5.503678**
U234/Th-232= **94.900299** ± **6.008409**

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

U(ppb)= **1084561.3**

Th(ppb)= **36262.733**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2598.0684 259.52771 25.939115

NP1194.CHW

MCB # 1 ACQ 01-25-95 AT 13:11:41 RT : 30002.9 LT : 30000.0
No detector description was entered
NOPI-119 U 1/26/95

ROI # 10-1 RANGE : 33 = 401.96keV to 146 = 428.13keV
AREA : Gross = 3568 Net = 3567 +/- 60
CENTROID : 118.37 = 421.73keV
SHAPE : Fwhm = 8.11keV Fwtm = 15.08keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.119 +/- 0.00

ROI # 10-2 RANGE : 278 = 458.70keV to 395 = 485.79keV
AREA : Gross = 3707 Net = 3707 +/- 61
CENTROID : 366.94 = 479.30keV
SHAPE : Fwhm = 7.74keV Fwtm = 15.48keV

ID : No close library match

ROI # 10-3 RANGE : 563 = 524.70keV to 630 = 540.22keV
AREA : Gross = 4732 Net = 3907 +/- 111
CENTROID : 604.29 = 534.26keV
SHAPE : Fwhm = 6.35keV Fwtm = 11.93keV

ID : No close library match

NPI19 Th. CHW

MCB # 1 ACQ 01-25-95 AT 16:49:43 RT : 57609.7 LT : 57603.1
No detector description was entered
NOPI-119 Th 1/26/95

ROI # 3-1 RANGE : 55 = 3.89keV to 114 = 4.03keV
AREA : Gross = 292 Net = 292 +/- 17
CENTROID : 96.29 = 3.99keV
SHAPE : Fwhm = 0.01keV Fwtm = 0.10keV

ID : No close library match

ROI # 3-2 RANGE : 271 = 4.41keV to 389 = 4.69keV
AREA : Gross = 27225 Net = 27130 +/- 170
CENTROID : 368.76 = 4.64keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.12keV

ID : No close library match

ROI # 3-3 RANGE : 569 = 5.12keV to 700 = 5.43keV
AREA : Gross = 17412 Net = 17197 +/- 147
CENTROID : 674.52 = 5.37keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 3-4 RANGE : 720 = 5.48keV to 834 = 5.76keV
AREA : Gross = 2455 Net = 2322 +/- 69
CENTROID : 782.11 = 5.63keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.12keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP120U.CHN
NP120TH.CHN

Sample # **NOPI-120**
Analyst **JDP**

Sep. date **1/23/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5216	1.0061	454.83	1/22/93	731	446.15055

Th-228/U-232= **0.5817043**

Counting time for Th= **833.36** (mins.)
Days btwn. sep. and count.= **3** (days)
CF for Th-228= **0.9967422**

Counting time for U= **500.05** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.0020966**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
271	22848	14048	3392

U-238 counts	U-234 counts	U-232 counts
3118	3141	3560

Bkgd	Bkgd	Bkgd	bkgd
3	7	13	24

bkgd	bkgd	bkgd
0	3	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
268	22841	13633.785	3368

U-238* counts	U-234* counts	U-232sp counts
3118	3138	3542.5726

U-238(dpm/g)= **757.42973** ± **18.57815**
U-234(dpm/g)= **762.28816** ± **18.66079**

Th-232(dpm/g)= **9.8402391** ± **0.60349**
Th-230(dpm/g)= **838.66008** ± **8.991751**

U-234/U-238= **1.0064144** ± **0.025442**
Th-230/U-234= **1.1001877** ± **0.020936**
Th-230/Th-232= **85.227612** ± **5.207822**
U234/Th-232= **77.466427** ± **5.115419**

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

U(ppb)= **1015299.4**

Th(ppb)= **40272.388**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2598.0684 259.52771 25.939115

NP1204.CHW

MCB # 1 ACQ 01-25-95 AT 13:11:42 RT : 30002.9 LT : 30000.0
No detector description was entered
NOPI-120 U 1/26/95

ROI # 11-1 RANGE : 54 = 400.08keV to 164 = 425.43keV
AREA : Gross = 3119 Net = 3118 +/- 56
CENTROID : 137.13 = 419.24keV
SHAPE : Fwhm = 6.58keV Fwtm = 14.61keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.104 +/- 0.00

ROI # 11-2 RANGE : 289 = 454.22keV to 419 = 484.17keV
AREA : Gross = 3142 Net = 3141 +/- 56
CENTROID : 385.98 = 476.57keV
SHAPE : Fwhm = 8.26keV Fwtm = 14.21keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.105 +/- 0.00

ROI # 11-3 RANGE : 569 = 518.73keV to 650 = 537.39keV
AREA : Gross = 3914 Net = 3560 +/- 90
CENTROID : 625.46 = 531.74keV
SHAPE : Fwhm = 7.94keV Fwtm = 14.23keV

ID : No close library match

MCB # 1 ACQ 01-26-95 AT 09:16:09 RT : 50001.7 LT : 50000.0
No detector description was entered
NOPI-120 Th 1/27/95

ROI # 1-1 RANGE : 37 = 3.88keV to 84 = 4.00keV
AREA : Gross = 272 Net = 271 +/- 17
CENTROID : 73.09 = 3.97keV
SHAPE : Fwhm = 0.02keV Fwtm = 0.04keV

ID : No close library match

ROI # 1-2 RANGE : 219 = 4.33keV to 355 = 4.67keV
AREA : Gross = 22985 Net = 22848 +/- 161
CENTROID : 335.28 = 4.62keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-3 RANGE : 553 = 5.16keV to 656 = 5.42keV
AREA : Gross = 14324 Net = 14048 +/- 136
CENTROID : 631.65 = 5.36keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.14keV

ID : No close library match

ROI # 1-4 RANGE : 682 = 5.48keV to 787 = 5.74keV
AREA : Gross = 3568 Net = 3392 +/- 80
CENTROID : 736.66 = 5.62keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.10keV

ID : No close library match

U and Th isotope Activities

FILENAME= NP121U.CHN
NP121TH.CHN

Sample # **NOPI-121**
Analyst **JDP**

Sep. date **1/30/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.508	1.0012	454.83	1/22/93	738	446.06824

Th-228/U-232= **0.5849683**

Counting time for Th= **1333.468** (mins.)
Days btwn. sep. and count.= **4** (days)
CF for Th-228= **0.9955824**

Counting time for U= **1230.87** (mins.)
Days btwn. sep. and count.= **2** (days)
CF for U-232= **1.002341**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
265	1650	14460	3846

U-238 counts	U-234 counts	U-232 counts
1468	2138	31756

Bkgd	Bkgd	Bkgd	bkgd
1	3	54	34

bkgd	bkgd	bkgd
18	16	18

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
264	1647	14002.989	3812

U-238* counts	U-234* counts	U-232sp counts
1450	2122	31663.874

U-238(dpm/g)= **40.258945** ± **1.074762**
U-234(dpm/g)= **58.916884** ± **1.31639**

Th-232(dpm/g)= **9.6955825** ± **0.601028**
Th-230(dpm/g)= **60.487214** ± **1.571756**

U-234/U-238= **1.4634483** ± **0.049605**
Th-230/U-234= **1.0266533** ± **0.033642**
Th-230/Th-232= **6.2386364** ± **0.412866**
U234/Th-232= **6.0766729** ± **0.400413**

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

U(ppb)= **53965.248**

Th(ppb)= **39680.363**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= **2612.1641** **260.93577** **26.079847**

NP121U.CHW

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 73852.3 LT : 73847.4
No detector description was entered
NOPI-121 U 2/2/95

ROI # 12-1 RANGE : 61 = 401.61keV to 161 = 424.76keV
AREA : Gross = 1704 Net = 1468 +/- 72
CENTROID : 139.67 = 419.82keV
SHAPE : Fwhm = 7.69keV Fwtm = 13.51keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.020 +/- 0.00

ROI # 12-2 RANGE : 304 = 457.87keV to 413 = 483.10keV
AREA : Gross = 2399 Net = 2138 +/- 82
CENTROID : 386.39 = 476.94keV
SHAPE : Fwhm = 5.12keV Fwtm = 12.70keV

ID : No close library match

ROI # 12-3 RANGE : 559 = 516.91keV to 657 = 539.60keV
AREA : Gross = 34560 Net = 31756 +/- 274
CENTROID : 623.85 = 531.92keV
SHAPE : Fwhm = 9.53keV Fwtm = 15.08keV

ID : No close library match

NP121 Th rthw

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-121 Th 2/6/95

ROI # 11-1 RANGE : 15 = 391.10keV to 79 = 405.84keV
AREA : Gross = 306 Net = 265 +/- 26
CENTROID : 54.74 = 400.26keV
SHAPE : Fwhm = 0.73keV Fwtm = 7.61keV

ID : No close library match

ROI # 11-2 RANGE : 278 = 451.69keV to 376 = 474.27keV
AREA : Gross = 1650 Net = 1650 +/- 41
CENTROID : 351.97 = 468.73keV
SHAPE : Fwhm = 4.22keV Fwtm = 12.78keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.021 +/- 0.00

ROI # 11-3 RANGE : 581 = 521.50keV to 701 = 549.14keV
AREA : Gross = 14727 Net = 14460 +/- 140
CENTROID : 672.80 = 542.64keV
SHAPE : Fwhm = 4.70keV Fwtm = 14.86keV

ID : No close library match

ROI # 11-4 RANGE : 719 = 553.29keV to 808 = 573.79keV
AREA : Gross = 3922 Net = 3846 +/- 70
CENTROID : 782.70 = 567.96keV
SHAPE : Fwhm = 5.89keV Fwtm = 12.39keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP122U.CHN
NP122TH.CHNSample # NOPI-122
Analyst JDP

Sep. date 1/30/95

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.511	1.0018	454.83	1/22/93	738	446.06824

Th-228/U-232= 0.5849683

Counting time for Th=	1333.468	(mins.)
Days btwn. sep. and count.=	4	(days)
CF for Th-228=	0.9955824	

Counting time for U=	1231.333	(mins.)
Days btwn. sep. and count.=	2	(days)
CF for U-232=	1.0023412	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
318	4025	16848	3855

U-238 counts	U-234 counts	U-232 counts
3094	3929	29373

Bkgd	Bkgd	Bkgd	bkgd
16	19	32	45

bkgd	bkgd	bkgd
26	15	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
302	4006	16385.79	3810

U-238* counts	U-234* counts	U-232sp counts
3068	3914	29294.416

U-238(dpm/g)=	91.586601	±	1.731086
U-234(dpm/g)=	116.84158	±	1.984803

Th-232(dpm/g)=	9.4282982	±	0.533679
Th-230(dpm/g)=	125.06544	±	2.194182

U-234/U-238=	1.2757497	±	0.030664
Th-230/U-234=	1.0703847	±	0.024005
Th-230/Th-232=	13.264901	±	0.772685
U234/Th-232=	12.392647	±	0.73238

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

U(ppb)= 122767.59

Th(ppb)= 38586.469

Spike 25A	Spike 25B	Spide 25C
10/9/92		
Th-228 (dpm/g)=	2612.1641	260.93577 26.079847

MCB # 1 ACQ 02-01-95 AT 14:30:58 RT : 73880.0 LT : 73875.1
No detector description was entered
NOPI-122 U 2/2/95

ROI # 13-1 RANGE : 47 = 401.31keV to 151 = 425.34keV
AREA : Gross = 3320 Net = 3094 +/- 83
CENTROID : 124.00 = 419.10keV
SHAPE : Fwhm = 7.56keV Fwtm = 12.89keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.042 +/- 0.00

ROI # 13-2 RANGE : 297 = 459.08keV to 405 = 484.03keV
AREA : Gross = 4216 Net = 3929 +/- 94
CENTROID : 374.02 = 476.87keV
SHAPE : Fwhm = 7.72keV Fwtm = 13.97keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.053 +/- 0.00

ROI # 13-3 RANGE : 539 = 514.99keV to 641 = 538.56keV
AREA : Gross = 31552 Net = 29373 +/- 254
CENTROID : 608.86 = 531.13keV
SHAPE : Fwhm = 9.22keV Fwtm = 14.16keV

ID : No close library match

NP122 Th. CHW

MCB # 1 ACQ 02-03-95 AT 09:14:48 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-122 Th 2/6/95

ROI # 12-1 RANGE : 12 = 390.26keV to 77 = 405.31keV
AREA : Gross = 504 Net = 318 +/- 47
CENTROID : 55.89 = 400.42keV
SHAPE : Fwhm = 3.75keV Fwtm = 7.47keV

ID : No close library match

ROI # 12-2 RANGE : 259 = 447.45keV to 372 = 473.61keV
AREA : Gross = 4157 Net = 4025 +/- 80
CENTROID : 344.87 = 467.33keV
SHAPE : Fwhm = 5.57keV Fwtm = 15.71keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.050 +/- 0.00

ROI # 12-3 RANGE : 547 = 514.13keV to 700 = 549.55keV
AREA : Gross = 17310 Net = 16848 +/- 168
CENTROID : 666.43 = 541.78keV
SHAPE : Fwhm = 6.05keV Fwtm = 17.31keV

ID : No close library match

ROI # 12-4 RANGE : 711 = 552.10keV to 808 = 574.56keV
AREA : Gross = 4085 Net = 3855 +/- 86
CENTROID : 777.62 = 567.52keV
SHAPE : Fwhm = 7.76keV Fwtm = 14.94keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP123U.CHN
NP123TH.CHN

Sample # **NOPI-123**
Analyst **JDP**

Sep. date **1/30/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.513	1.0045	454.83	1/22/93	738	446.06824	0.5849683

Counting time for Th=	1333.468 (mins.)	Counting time for U=	1231.752 (mins.)
Days btwn. sep. and count.=	4 (days)	Days btwn. sep. and count.=	2 (days)
CF for Th-228=	0.9955824	CF for U-232=	1.0023413

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts	U-238 counts	U-234 counts	U-232 counts
262	2856	15196	3659	3149	3523	36907

| Bkgd |
|------|------|------|------|------|------|------|
| 8 | 26 | 20 | 11 | 20 | 17 | 26 |

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts	U-238* counts	U-234* counts	U-232sp counts
254	2830	14795.137	3648	3129	3506	36794.851

U-238(dpm/g)=	74.276665 ± 1.37894
U-234(dpm/g)=	83.225947 ± 1.467574

Th-232(dpm/g)=	8.7716425 ± 0.546566
Th-230(dpm/g)=	97.731293 ± 1.993208

U-234/U-238=	1.1204858 ± 0.027478
Th-230/U-234=	1.1742888 ± 0.029568
Th-230/Th-232=	11.141732 ± 0.719219
U234/Th-232=	9.4880686 ± 0.614424

Decay constant (m-1)	U-238 2.948E-16	Th-232 9.413E-17
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U(ppb)= 99564.423

Th(ppb)= 35899.025

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	2612.1641	260.93577	26.079847

NP123u.CHW

MCB # 1 ACQ 02-01-95 AT 14:30:59 RT : 73905.1 LT : 73900.2
No detector description was entered
NOPI-123 U 2/2/95

ROI # 14-1 RANGE : 75 = 406.51keV to 153 = 424.46keV
AREA : Gross = 3151 Net = 3149 +/- 56
CENTROID : 131.27 = 419.46keV
SHAPE : Fwhm = 4.34keV Fwtm = 10.95keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.043 +/- 0.00

ROI # 14-2 RANGE : 324 = 463.82keV to 404 = 482.24keV
AREA : Gross = 3564 Net = 3523 +/- 64
CENTROID : 381.49 = 477.06keV
SHAPE : Fwhm = 4.36keV Fwtm = 10.71keV

ID : No close library match

ROI # 14-3 RANGE : 558 = 517.69keV to 646 = 537.94keV
AREA : Gross = 37382 Net = 36907 +/- 209
CENTROID : 618.18 = 531.54keV
SHAPE : Fwhm = 3.83keV Fwtm = 11.71keV

ID : No close library match

NOPI-123 Th. (HW)

MCB # 1 ACQ 02-03-95 AT 09:14:49 RT : 80008.1 LT : 80000.0
No detector description was entered
NOPI-123 Th 2/6/95

ROI # 13-1 RANGE : 12 = 393.22keV to 64 = 405.24keV
AREA : Gross = 403 Net = 262 +/- 37
CENTROID : 49.43 = 401.87keV
SHAPE : Fwhm = 0.63keV Fwtm = 6.24keV

ID : No close library match

ROI # 13-2 RANGE : 250 = 448.22keV to 361 = 473.86keV
AREA : Gross = 3078 Net = 2856 +/- 82
CENTROID : 334.86 = 467.82keV
SHAPE : Fwhm = 4.71keV Fwtm = 14.49keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.036 +/- 0.00

ROI # 13-3 RANGE : 536 = 514.30keV to 685 = 548.73keV
AREA : Gross = 15533 Net = 15196 +/- 153
CENTROID : 655.47 = 541.90keV
SHAPE : Fwhm = 4.99keV Fwtm = 15.93keV

ID : No close library match

ROI # 13-4 RANGE : 704 = 553.12keV to 796 = 574.37keV
AREA : Gross = 3845 Net = 3659 +/- 80
CENTROID : 766.05 = 567.45keV
SHAPE : Fwhm = 6.45keV Fwtm = 13.60keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP307U.CHN
NP307TH.CHN

Sample # **NOPI-307**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5032	0.9973	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
244	12578	12662	4945

U-238 counts	U-234 counts	U-232 counts
6106	8101	14970

Bkgd	Bkgd	Bkgd	bkgd
4	6	14	22

bkgd	bkgd	bkgd
2	5	3

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
240	12572	12236.979	4923

U-238* counts	U-234* counts	U-232sp counts
6104	8096	14877.764

U-238(dpm/g)= **362.68452** ± **5.507238**
U-234(dpm/g)= **481.04422** ± **6.634955**

Th-232(dpm/g)= **10.166096** ± **0.657058**
Th-230(dpm/g)= **532.53399** ± **6.704016**

U-234/U-238= **1.3263434** ± **0.022478**
Th-230/U-234= **1.1070375** ± **0.015771**
Th-230/Th-232= **52.383333** ± **3.38587**
U234/Th-232= **47.318481** ± **3.127168**

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

U(ppb)= **486161.78**

Th(ppb)= **41605.997**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP3074.CHN

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 40006.7 LT : 40000.0
No detector description was entered
NOPI-307 U 2/9/95

ROI # 2-1 RANGE : 63 = 3.94keV to 169 = 4.21keV
AREA : Gross = 6130 Net = 6103 +/- 81
CENTROID : 151.55 = 4.17keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.13keV

ID : No close library match

ROI # 2-2 RANGE : 287 = 4.51keV to 401 = 4.80keV
AREA : Gross = 8244 Net = 8101 +/- 103
CENTROID : 383.55 = 4.76keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

ROI # 2-3 RANGE : 541 = 5.16keV to 624 = 5.37keV
AREA : Gross = 16088 Net = 14970 +/- 172
CENTROID : 599.42 = 5.31keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

NP307TH.CHW

MCB # 1 ACQ 02-09-95 AT 11:40:14 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-307 Th 2/10/95

ROI # 1-1 RANGE : 27 = 3.86keV to 90 = 4.01keV
AREA : Gross = 276 Net = 244 +/- 24
CENTROID : 62.52 = 3.94keV
SHAPE : Fwhm = 0.01keV Fwtm = 0.05keV

ID : No close library match

ROI # 1-2 RANGE : 230 = 4.36keV to 357 = 4.68keV
AREA : Gross = 12686 Net = 12578 +/- 122
CENTROID : 335.51 = 4.62keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-3 RANGE : 511 = 5.06keV to 655 = 5.41keV
AREA : Gross = 13120 Net = 12662 +/- 153
CENTROID : 631.30 = 5.36keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.15keV

ID : No close library match

ROI # 1-4 RANGE : 667 = 5.44keV to 758 = 5.67keV
AREA : Gross = 5419 Net = 4945 +/- 108
CENTROID : 729.87 = 5.60keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.16keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP397U.CHN
NP397TH.CHN

Sample # **NOPI-397**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.2934	0.9995	454.83	1/22/93	741	446.03297	0.5863609

Counting time for Th=	833.47	(mins.)	Counting time for U=	666.778	(mins.)
Days btwn. sep. and count.=	7	(days)	Days btwn. sep. and count.=	6	(days)
CF for Th-228=	0.9927949		CF for U-232=	1.005998	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
170	12435	10952	5261

U-238 counts	U-234 counts	U-232 counts
10842	14313	22747

Bkgd	Bkgd	Bkgd	bkgd
1	2	9	12

bkgd	bkgd	bkgd
2	5	1

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
169	12433	10573.202	5249

U-238* counts	U-234* counts	U-232sp counts
10840	14308	22610.384

U-238(dpm/g)= $\frac{728.46889}{\pm 8.501446}$
U-234(dpm/g)= $\frac{961.52517}{\pm 10.25855}$

Th-232(dpm/g)= $\frac{14.240816}{\pm 1.100665}$
Th-230(dpm/g)= $\frac{1047.669}{\pm 13.72909}$

U-234/U-238= $\frac{1.3199262}{\pm 0.016805}$
Th-230/U-234= $\frac{1.0895908}{\pm 0.013357}$
Th-230/Th-232= $\frac{73.568047}{\pm 5.68085}$
U234/Th-232= $\frac{67.518968}{\pm 5.267989}$

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

U(ppb)= $\frac{976478.75}{\pm}$

Th(ppb)= $\frac{58282.288}{\pm}$

	Spike 25A	Spike 25B	Spide 25C
	10/9/92		
Th-228 (dpm/g)=	2618.176	261.53631	26.13987

WP397U.CHW

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 40006.7 LT : 40000.0
No detector description was entered
NOPI-397 U 2/9/95

ROI # 3-1 RANGE : 65 = 3.91keV to 195 = 4.22keV
AREA : Gross = 11169 Net = 10842 +/- 133
CENTROID : 172.03 = 4.17keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.19keV

ID : No close library match

ROI # 3-2 RANGE : 302 = 4.48keV to 429 = 4.78keV
AREA : Gross = 14467 Net = 14313 +/- 132
CENTROID : 397.59 = 4.71keV
SHAPE : Fwhm = 0.11keV Fwtm = 0.20keV

ID : No close library match

ROI # 3-3 RANGE : 551 = 5.08keV to 651 = 5.32keV
AREA : Gross = 24565 Net = 22747 +/- 227
CENTROID : 624.53 = 5.25keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.18keV

ID : No close library match

NP397Th.CHW

MCB # 1 ACQ 02-09-95 AT 11:40:14 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-397 Th 2/10/95

ROI # 2-1 RANGE : 43 = 3.89keV to 91 = 4.01keV
AREA : Gross = 170 Net = 170 +/- 13
CENTROID : 79.13 = 3.98keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.09keV

ID : No close library match

ROI # 2-2 RANGE : 234 = 4.38keV to 363 = 4.71keV
AREA : Gross = 12597 Net = 12435 +/- 126
CENTROID : 345.08 = 4.66keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 2-3 RANGE : 517 = 5.10keV to 667 = 5.48keV
AREA : Gross = 11217 Net = 10952 +/- 132
CENTROID : 640.98 = 5.41keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.14keV

ID : No close library match

ROI # 2-4 RANGE : 676 = 5.50keV to 765 = 5.73keV
AREA : Gross = 5531 Net = 5261 +/- 95
CENTROID : 741.11 = 5.67keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.13keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP398U.CHN
NP398TH.CHN

Sample # **NOPI-398**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5054	0.9959	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
456	34503	16959	6760

U-238 counts	U-234 counts	U-232 counts
10935	12765	10800

Bkgd	Bkgd	Bkgd	bkgd
1	2	3	1

bkgd	bkgd	bkgd
4	6	16

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
455	34501	16263.23	6759

U-238* counts	U-234* counts	U-232sp counts
10931	12759	10719.704

U-238(dpm/g)= **896.24053** ± **12.15859**
U-234(dpm/g)= **1046.1196** ± **13.67706**

Th-232(dpm/g)= **14.418401** ± **0.684221**
Th-230(dpm/g)= **1093.295** ± **10.25303**

U-234/U-238= **1.1672308** ± **0.015209**
Th-230/U-234= **1.0450957** ± **0.010827**
Th-230/Th-232= **75.826374** ± **3.574283**
U234/Th-232= **72.554481** ± **3.571332**

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

U(ppb)= **1201368.9**

Th(ppb)= **59009.076**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP398U.cHW

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 40006.7 LT : 40000.0
No detector description was entered
NOPI-398 U 2/9/95

ROI # 5-1 RANGE : 25 = 3.81keV to 165 = 4.19keV
AREA : Gross = 11478 Net = 10935 +/- 152
CENTROID : 134.87 = 4.11keV
SHAPE : Fwhm = 0.14keV Fwtm = 0.27keV

ID : No close library match

ROI # 5-2 RANGE : 234 = 4.37keV to 384 = 4.77keV
AREA : Gross = 13166 Net = 12765 +/- 150
CENTROID : 348.17 = 4.68keV
SHAPE : Fwhm = 0.14keV Fwtm = 0.25keV

ID : No close library match

ROI # 5-3 RANGE : 489 = 5.06keV to 590 = 5.33keV
AREA : Gross = 12958 Net = 10800 +/- 213
CENTROID : 554.33 = 5.23keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.21keV

ID : No close library match

NP398Th.cth

MCB # 1 ACQ 02-09-95 AT 11:40:14 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-398 Th 2/10/95

ROI # 3-1 RANGE : 50 = 3.88keV to 118 = 4.04keV
AREA : Gross = 491 Net = 456 +/- 29
CENTROID : 100.48 = 4.00keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.11keV

ID : No close library match

ROI # 3-2 RANGE : 250 = 4.36keV to 390 = 4.69keV
AREA : Gross = 34622 Net = 34503 +/- 193
CENTROID : 367.64 = 4.64keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.14keV

ID : No close library match

ROI # 3-3 RANGE : 560 = 5.10keV to 695 = 5.42keV
AREA : Gross = 17387 Net = 16959 +/- 162
CENTROID : 672.78 = 5.37keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.15keV

ID : No close library match

ROI # 3-4 RANGE : 705 = 5.45keV to 805 = 5.69keV
AREA : Gross = 7618 Net = 6760 +/- 143
CENTROID : 773.68 = 5.61keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.14keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP399U.CHN
NP399TH.CHN

Sample # **NOPI-399**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5059	0.9976	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
282	11685	13914	5102

U-238 counts	U-234 counts	U-232 counts
5621	11745	26496

Bkgd	Bkgd	Bkgd	bkgd
3	3	3	49

bkgd	bkgd	bkgd
6	11	15

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
279	11682	13463.205	5053

U-238* counts	U-234* counts	U-232sp counts
5615	11734	26323.115

U-238(dpm/g)= **187.61658** ± **2.755128**
U-234(dpm/g)= **392.07354** ± **4.346258**

Th-232(dpm/g)= **10.687583** ± **0.642853**
Th-230(dpm/g)= **447.49943** ± **5.615179**

U-234/U-238= **2.0897596** ± **0.033893**
Th-230/U-234= **1.1413661** ± **0.014913**
Th-230/Th-232= **41.870968** ± **2.523287**
U234/Th-232= **36.684959** ± **2.243744**

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

U(ppb)= **251491.32**

Th(ppb)= **43740.246**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP399U.cdw

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 40006.7 LT : 40000.0
No detector description was entered
NOPI-399 U 2/9/95

ROI # 6-1 RANGE : 46 = 3.95keV to 155 = 4.24keV
AREA : Gross = 5748 Net = 5621 +/- 88
CENTROID : 129.40 = 4.17keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.17keV

ID : No close library match

ROI # 6-2 RANGE : 255 = 4.51keV to 378 = 4.83keV
AREA : Gross = 11776 Net = 11745 +/- 111
CENTROID : 345.51 = 4.74keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.17keV

ID : No close library match

ROI # 6-3 RANGE : 493 = 5.13keV to 578 = 5.36keV
AREA : Gross = 28431 Net = 26496 +/- 229
CENTROID : 556.99 = 5.30keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.15keV

ID : No close library match

NP399TH.CHW

MCB # 1 ACQ 02-09-95 AT 11:40:14 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-399 Th 2/10/95

ROI # 5-1 RANGE : 27 = 3.82keV to 94 = 4.00keV
AREA : Gross = 282 Net = 282 +/- 17
CENTROID : 73.38 = 3.94keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.11keV

ID : No close library match

ROI # 5-2 RANGE : 233 = 4.37keV to 348 = 4.68keV
AREA : Gross = 11800 Net = 11685 +/- 117
CENTROID : 325.71 = 4.62keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.15keV

ID : No close library match

ROI # 5-3 RANGE : 504 = 5.10keV to 626 = 5.42keV
AREA : Gross = 14205 Net = 13914 +/- 140
CENTROID : 603.42 = 5.36keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.16keV

ID : No close library match

ROI # 5-4 RANGE : 638 = 5.45keV to 723 = 5.68keV
AREA : Gross = 5377 Net = 5102 +/- 94
CENTROID : 695.39 = 5.61keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.17keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP400U.CHN
NP400TH.CHN

Sample # **NOPI-400**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4939	0.9969	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
288	37444	13734	5282

U-238 counts	U-234 counts	U-232 counts
8097	22636	13517

Bkgd	Bkgd	Bkgd	bkgd
4	16	27	71

bkgd	bkgd	bkgd
2	3	6

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
284	37428	13244.29	5211

U-238* counts	U-234* counts	U-232sp counts
8095	22633	13430.445

U-238(dpm/g)= **542.63275** ± **7.625553**
U-234(dpm/g)= **1517.1596** ± **16.49162**

Th-232(dpm/g)= **11.319682** ± **0.673976**
Th-230(dpm/g)= **1491.8065** ± **14.88211**

U-234/U-238= **2.7959234** ± **0.036205**
Th-230/U-234= **0.9832891** ± **0.008279**
Th-230/Th-232= **131.78873** ± **7.795533**
U234/Th-232= **134.02847** ± **8.111981**

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

U(ppb)= **727374.03**

Th(ppb)= **46327.189**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP400d.CHW

MCB # 1 ACQ 02-08-95 AT 15:59:27 RT : 40006.6 LT : 40000.0
No detector description was entered
NOPI-400 U 2/9/95

ROI # 9-1 RANGE : 32 = 395.56keV to 161 = 425.37keV
AREA : Gross = 8248 Net = 8097 +/- 106
CENTROID : 130.38 = 418.29keV
SHAPE : Fwhm = 12.17keV Fwtm = 19.99keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.202 +/- 0.00

ROI # 9-2 RANGE : 273 = 451.26keV to 412 = 483.38keV
AREA : Gross = 23056 Net = 22636 +/- 179
CENTROID : 376.05 = 475.07keV
SHAPE : Fwhm = 12.55keV Fwtm = 20.93keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.566 +/- 0.00

ROI # 9-3 RANGE : 561 = 517.82keV to 649 = 538.16keV
AREA : Gross = 17196 Net = 13517 +/- 254
CENTROID : 613.09 = 529.86keV
SHAPE : Fwhm = 10.71keV Fwtm = 15.33keV

ID : No close library match

NP400Th.CHN

MCB # 1 ACQ 02-09-95 AT 11:40:15 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-400 Th 2/10/95

ROI # 6-1 RANGE : 19 = 3.88keV to 80 = 4.04keV
AREA : Gross = 349 Net = 288 +/- 29
CENTROID : 58.55 = 3.99keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.10keV

ID : No close library match

ROI # 6-2 RANGE : 192 = 4.34keV to 338 = 4.72keV
AREA : Gross = 37645 Net = 37444 +/- 205
CENTROID : 312.64 = 4.66keV
SHAPE : Fwhm = 0.07keV Fwtm = 0.15keV

ID : No close library match

ROI # 6-3 RANGE : 511 = 5.18keV to 614 = 5.45keV
AREA : Gross = 14306 Net = 13734 +/- 152
CENTROID : 593.15 = 5.40keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.17keV

ID : No close library match

ROI # 6-4 RANGE : 631 = 5.50keV to 711 = 5.71keV
AREA : Gross = 5768 Net = 5282 +/- 107
CENTROID : 685.40 = 5.64keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.15keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP401U.CHN
NP401TH.CHN

Sample # **NOPI-401**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5033	0.9958	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
143	5516	9320	3960

U-238 counts	U-234 counts	U-232 counts
2723	5269	19600

Bkgd	Bkgd	Bkgd	bkgd
4	8	13	23

bkgd	bkgd	bkgd
4	5	6

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
139	5508	9025.3695	3937

U-238* counts	U-234* counts	U-232sp counts
2719	5264	19477.176

U-238(dpm/g)= **123.19565** ± **2.519531**
U-234(dpm/g)= **238.5075** ± **3.70117**

Th-232(dpm/g)= **7.9694252** ± **0.67153**
Th-230(dpm/g)= **315.79564** ± **5.364688**

U-234/U-238= **1.9360059** ± **0.045693**
Th-230/U-234= **1.3240491** ± **0.025506**
Th-230/Th-232= **39.625899** ± **3.356362**
U234/Th-232= **29.927817** ± **2.564224**

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

U(ppb)= **165138.05**

Th(ppb)= **32615.852**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP4014.CTN

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 40006.6 LT : 40000.0
No detector description was entered
NOPI-401 U 2/9/95

ROI # 7-1 RANGE : 73 to 155
AREA : Gross = 2765 Net = 2723 +/- 57
CENTROID : 132.15
SHAPE : Fwhm = 23.21 Channels Fwtm = 49.81 Channels

ROI # 7-2 RANGE : 313 to 409
AREA : Gross = 5478 Net = 5269 +/- 92
CENTROID : 381.84
SHAPE : Fwhm = 21.48 Channels Fwtm = 56.39 Channels

ROI # 7-3 RANGE : 558 to 644
AREA : Gross = 20151 Net = 19600 +/- 164
CENTROID : 617.65
SHAPE : Fwhm = 21.79 Channels Fwtm = 54.09 Channels

NP401Th.CHN

MCB # 1 ACQ 02-09-95 AT 11:40:15 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-401 Th 2/10/95

ROI # 7-1 RANGE : 17 to 68
AREA : Gross = 195 Net = 143 +/- 23
CENTROID : 54.00
SHAPE : Fwhm = 2.68 Channels Fwtm = 17.64 Channels

ROI # 7-2 RANGE : 266 to 362
AREA : Gross = 5612 Net = 5516 +/- 83
CENTROID : 338.87
SHAPE : Fwhm = 22.68 Channels Fwtm = 60.26 Channels

ROI # 7-3 RANGE : 585 to 686
AREA : Gross = 9540 Net = 9320 +/- 113
CENTROID : 659.39
SHAPE : Fwhm = 19.31 Channels Fwtm = 63.59 Channels

ROI # 7-4 RANGE : 705 to 793
AREA : Gross = 4138 Net = 3960 +/- 80
CENTROID : 766.33
SHAPE : Fwhm = 30.69 Channels Fwtm = 54.62 Channels

U and Th Isotope Activities

FILENAME= NP402U.CHN
NP402TH.CHN

Sample # **NOPI-402**
Analyst **JDP**

Sep. date **2/2/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5014	1.0014	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th= **833.47** (mins.)
Days btwn. sep. and count.= **7** (days)
CF for Th-228= **0.9927949**

Counting time for U= **666.778** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.005998**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
219	6303	10135	3590

U-238 counts	U-234 counts	U-232 counts
5006	6280	19158

Bkgd	Bkgd	Bkgd	bkgd
1	7	15	38

bkgd	bkgd	bkgd
4	4	5

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
218	6296	9785.8229	3552

U-238* counts	U-234* counts	U-232sp counts
5002	6276	19038.806

U-238(dpm/g)= **234.04222** ± **3.714994**
U-234(dpm/g)= **293.65233** ± **4.269923**

Th-232(dpm/g)= **11.636286** ± **0.794757**
Th-230(dpm/g)= **336.06449** ± **5.390904**

U-234/U-238= **1.2546981** ± **0.023773**
Th-230/U-234= **1.1444298** ± **0.020405**
Th-230/Th-232= **28.880734** ± **1.985194**
U234/Th-232= **25.235915** ± **1.762239**

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

U(ppb)= **313722.74**

Th(ppb)= **47622.932**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2618.176 261.53631 26.13987

NP402U.CHN

MCB # 1 ACQ 02-08-95 AT 15:59:26 RT : 50006.8 LT : 50000.0
No detector description was entered
NOPI-402 U 2/9/95

ROI # 8-1 RANGE : 33 = 398.40keV to 156 = 426.47keV
AREA : Gross = 5285 Net = 5006 +/- 102
CENTROID : 126.29 = 419.69keV
SHAPE : Fwhm = 8.93keV Fwtm = 17.59keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.100 +/- 0.00

ROI # 8-2 RANGE : 297 = 458.65keV to 408 = 483.99keV
AREA : Gross = 6728 Net = 6280 +/- 119
CENTROID : 375.51 = 476.57keV
SHAPE : Fwhm = 10.50keV Fwtm = 17.24keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.126 +/- 0.00

ROI # 8-3 RANGE : 544 = 515.03keV to 647 = 538.54keV
AREA : Gross = 20458 Net = 19158 +/- 201
CENTROID : 611.55 = 530.44keV
SHAPE : Fwhm = 9.91keV Fwtm = 16.70keV

ID : No close library match

NP40ZTA.CHN

MCB # 1 ACQ 02-09-95 AT 11:40:15 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-402 Th 2/10/95

ROI # 8-1 RANGE : 11 = 393.38keV to 65 = 405.70keV
AREA : Gross = 337 Net = 219 +/- 34
Could not properly fit the peak.

ROI # 8-2 RANGE : 247 = 447.24keV to 365 = 474.17keV
AREA : Gross = 6619 Net = 6303 +/- 111
CENTROID : 335.06 = 467.34keV
SHAPE : Fwhm = 6.39keV Fwtm = 16.44keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.126 +/- 0.00

ROI # 8-3 RANGE : 555 = 517.54keV to 685 = 547.21keV
AREA : Gross = 10691 Net = 10135 +/- 147
CENTROID : 658.92 = 541.26keV
SHAPE : Fwhm = 5.91keV Fwtm = 16.71keV

ID : No close library match

ROI # 8-4 RANGE : 709 = 552.69keV to 794 = 572.09keV
AREA : Gross = 4133 Net = 3590 +/- 104
CENTROID : 762.16 = 564.82keV
SHAPE : Fwhm = 9.02keV Fwtm = 16.43keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP403U.CHN
NP403TH.CHN

Sample # **NOPI-403**
Analyst **JDP**

Sep. date **2/2/95**

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	0.9985	454.83	1/22/93	741	446.03297

Th-228/U-232= **0.5863609**

Counting time for Th=	833.47	(mins.)	Counting time for U=	666.778	(mins.)
Days btwn. sep. and count.=	7	(days)	Days btwn. sep. and count.=	6	(days)
CF for Th-228=	0.9927949		CF for U-232=	1.005998	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
140	5341	9940	3880

U-238 counts	U-234 counts	U-232 counts
6673	6637	26659

Bkgd	Bkgd	Bkgd	bkgd
0.5	2	22	50

bkgd	bkgd	bkgd
7	5	4

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
139.5	5339	9646.0159	3830

U-238* counts	U-234* counts	U-232sp counts
6666	6632	26496.077

U-238(dpm/g)= **222.35884** ± **3.043705**
U-234(dpm/g)= **221.2247** ± **3.034742**

Th-232(dpm/g)= **7.4948327** ± **0.637874**
Th-230(dpm/g)= **286.84525** ± **4.866526**

U-234/U-238= **0.9948995** ± **0.017247**
Th-230/U-234= **1.296624** ± **0.023835**
Th-230/Th-232= **38.272401** ± **3.276727**
U234/Th-232= **29.516963** ± **2.544568**

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

U(ppb)= **298061.71**

Th(ppb)= **30673.524**

	Spike 25A 10/9/92	Spike 25B	Spide 25C
Th-228 (dpm/g)=	2618.176	261.53631	26.13987

NP403 u.ctn

MCB # 1 ACQ 02-08-95 AT 10:06:16 RT : 40004.1 LT : 40000.0
No detector description was entered
NOPI-403 U 2/9/95

ROI # 1-1 RANGE : 73 = 3.97keV to 162 = 4.19keV
AREA : Gross = 6822 Net = 6673 +/- 94
CENTROID : 139.93 = 4.14keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-2 RANGE : 294 = 4.52keV to 387 = 4.75keV
AREA : Gross = 7499 Net = 6637 +/- 139
CENTROID : 367.15 = 4.70keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-3 RANGE : 520 = 5.08keV to 610 = 5.30keV
AREA : Gross = 27918 Net = 26659 +/- 211
CENTROID : 585.36 = 5.24keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.14keV

ID : No close library match

NP403TH.CHN

MCB # 1 ACQ 02-09-95 AT 11:40:15 RT : 50008.2 LT : 50000.0
No detector description was entered
NOPI-403 Th 2/10/95

ROI # 9-1 RANGE : 22 = 393.25keV to 72 = 404.80keV
AREA : Gross = 184 Net = 140 +/- 22
CENTROID : 44.73 = 398.50keV
SHAPE : Fwhm = 0.93keV Fwtm = 5.14keV

ID : No close library match

ROI # 9-2 RANGE : 233 = 442.01keV to 369 = 473.45keV
AREA : Gross = 5444 Net = 5341 +/- 87
CENTROID : 342.83 = 467.40keV
SHAPE : Fwhm = 6.67keV Fwtm = 15.95keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.107 +/- 0.00

ROI # 9-3 RANGE : 568 = 519.44keV to 690 = 547.64keV
AREA : Gross = 10263 Net = 9940 +/- 127
CENTROID : 664.14 = 541.66keV
SHAPE : Fwhm = 5.51keV Fwtm = 15.93keV

ID : No close library match

ROI # 9-4 RANGE : 707 = 551.57keV to 802 = 573.52keV
AREA : Gross = 4328 Net = 3880 +/- 103
CENTROID : 768.95 = 565.88keV
SHAPE : Fwhm = 9.14keV Fwtm = 15.45keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP404U.CHN
NP404TH.CHN

Sample # **NOPI-404**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5068	1.0001	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **500.055** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0059424**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
337	5149	18282	4679

U-238 counts	U-234 counts	U-232 counts
2220	2242	18990

Bkgd	Bkgd	Bkgd	bkgd
5	7	15	26

bkgd	bkgd	bkgd
5	3	4

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
332	5142	17784.235	4653

U-238* counts	U-234* counts	U-232sp counts
2215	2239	18873.843

U-238(dpm/g)= **103.28054** ± **2.316594**
U-234(dpm/g)= **104.3996** ± **2.331385**

Th-232(dpm/g)= **9.6788334** ± **0.532077**
Th-230(dpm/g)= **149.90531** ± **2.365043**

U-234/U-238= **1.0108352** ± **0.030266**
Th-230/U-234= **1.43588** ± **0.036332**
Th-230/Th-232= **15.487952** ± **0.870854**
U234/Th-232= **10.786383** ± **0.64002**

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

U(ppb)= **138442.77**

Th(ppb)= **39611.815**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP404u.CHW

MCB # 1 ACQ 02-14-95 AT 11:10:29 RT : 30003.3 LT : 30000.0
No detector description was entered
NOPI-404 U 2/15/95

ROI # 1-1 RANGE : 85 = 4.00keV to 162 = 4.19keV
AREA : Gross = 2239 Net = 2220 +/- 49
CENTROID : 145.23 = 4.15keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.10keV

ID : No close library match

ROI # 1-2 RANGE : 333 = 4.62keV to 388 = 4.75keV
AREA : Gross = 2438 Net = 2242 +/- 62
CENTROID : 373.90 = 4.72keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.10keV

ID : No close library match

ROI # 1-3 RANGE : 533 = 5.11keV to 611 = 5.31keV
AREA : Gross = 19846 Net = 18990 +/- 171
CENTROID : 591.84 = 5.26keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.11keV

ID : No close library match

MCB # 1 ACQ 02-13-95 AT 16:19:34 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-404 Th 2/14/95

ROI # 1-1 RANGE : 15 = 3.83keV to 89 = 4.01keV
AREA : Gross = 337 Net = 337 +/- 18
CENTROID : 68.25 = 3.96keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.09keV

ID : No close library match

ROI # 1-2 RANGE : 253 = 4.42keV to 355 = 4.67keV
AREA : Gross = 5201 Net = 5149 +/- 77
CENTROID : 335.38 = 4.62keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.13keV

ID : No close library match

ROI # 1-3 RANGE : 554 = 5.16keV to 654 = 5.41keV
AREA : Gross = 18667 Net = 18282 +/- 156
CENTROID : 631.20 = 5.36keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.14keV

ID : No close library match

ROI # 1-4 RANGE : 680 = 5.48keV to 755 = 5.66keV
AREA : Gross = 5021 Net = 4679 +/- 93
CENTROID : 730.67 = 5.60keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.13keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP405U.CHN
NP405TH.CHN

Sample # **NOPI-405**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.502	1.0012	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **333.377** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0058869**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
349	13299	18573	4815

U-238 counts	U-234 counts	U-232 counts
4453	4263	11276

Bkgd	Bkgd	Bkgd	bkgd
2	4	13	19

bkgd	bkgd	bkgd
1	3	2

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
347	13295	18056.174	4796

U-238* counts	U-234* counts	U-232sp counts
4452	4260	11208.019

U-238(dpm/g)= **353.29842** ± **6.252999**
U-234(dpm/g)= **338.06183** ± **6.078165**

Th-232(dpm/g)= **10.070109** ± **0.544081**
Th-230(dpm/g)= **385.82737** ± **4.382755**

U-234/U-238= **0.9568733** ± **0.020504**
Th-230/U-234= **1.1412923** ± **0.020087**
Th-230/Th-232= **38.314121** ± **2.077644**
U234/Th-232= **33.570821** ± **1.911602**

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

U(ppb)= **473580.14**

Th(ppb)= **41213.158**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP4054.c.hw

MCB # 1 ACQ 02-14-95 AT 11:10:29 RT : 20002.6 LT : 20000.0
No detector description was entered
NOPI-405 U 2/15/95

ROI # 2-1 RANGE : 56 = 3.92keV to 170 = 4.21keV
AREA : Gross = 4816 Net = 4453 +/- 105
CENTROID : 148.54 = 4.16keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.21keV

ID : No close library match

ROI # 2-2 RANGE : 282 = 4.50keV to 401 = 4.80keV
AREA : Gross = 4642 Net = 4263 +/- 107
CENTROID : 376.85 = 4.74keV
SHAPE : Fwhm = 0.13keV Fwtm = 0.20keV

ID : No close library match

ROI # 2-3 RANGE : 515 = 5.09keV to 616 = 5.35keV
AREA : Gross = 12568 Net = 11276 +/- 179
CENTROID : 592.99 = 5.29keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.21keV

ID : No close library match

NP405Th.cHN

MCB # 1 ACQ 02-13-95 AT 16:19:35 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-405 Th 2/14/95

ROI # 2-1 RANGE : 17 = 3.82keV to 97 = 4.03keV
AREA : Gross = 389 Net = 349 +/- 29
CENTROID : 82.19 = 3.99keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.08keV

ID : No close library match

ROI # 2-2 RANGE : 241 = 4.39keV to 363 = 4.71keV
AREA : Gross = 13396 Net = 13299 +/- 123
CENTROID : 343.21 = 4.66keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.14keV

ID : No close library match

ROI # 2-3 RANGE : 517 = 5.10keV to 663 = 5.47keV
AREA : Gross = 19184 Net = 18573 +/- 181
CENTROID : 639.69 = 5.41keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.15keV

ID : No close library match

ROI # 2-4 RANGE : 681 = 5.52keV to 767 = 5.73keV
AREA : Gross = 5424 Net = 4815 +/- 114
CENTROID : 739.21 = 5.66keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.14keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP406U.CHN
NP406TH.CHN

Sample # **NOPI-406**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.4989	1.0026	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **1000.087** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.006109**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
259	1308	13664	3353

U-238 counts	U-234 counts	U-232 counts
1246	1262	33917

Bkgd	Bkgd	Bkgd	bkgd
1	1	21	72

bkgd	bkgd	bkgd
2	7	21

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
258	1307	13282.745	3281

U-238* counts	U-234* counts	U-232sp counts
1244	1255	33690.187

U-238(dpm/g)= **33.092489** ± **0.954563**
U-234(dpm/g)= **33.385108** ± **0.957097**

Th-232(dpm/g)= **10.25556** ± **0.643261**
Th-230(dpm/g)= **51.953555** ± **1.503704**

U-234/U-238= **1.0088424** ± **0.04029**
Th-230/U-234= **1.5561895** ± **0.061404**
Th-230/Th-232= **5.0658915** ± **0.344538**
U234/Th-232= **3.2553178** ± **0.224501**

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

U(ppb)= **44358.947**

Th(ppb)= **41972.14**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP406U.CHW

MCB # 1 ACQ 02-14-95 AT 11:10:30 RT : 60005.2 LT : 60000.0
No detector description was entered
NOPI-406 U 2/15/95

ROI # 5-1 RANGE : 112 = 4.04keV to 165 = 4.19keV
AREA : Gross = 1273 Net = 1246 +/- 38
CENTROID : 147.60 = 4.14keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.10keV

ID : No close library match

ROI # 5-2 RANGE : 331 = 4.63keV to 379 = 4.76keV
AREA : Gross = 1449 Net = 1262 +/- 51
CENTROID : 363.60 = 4.72keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.10keV

ID : No close library match

ROI # 5-3 RANGE : 516 = 5.13keV to 590 = 5.33keV
AREA : Gross = 34792 Net = 33917 +/- 210
CENTROID : 566.84 = 5.26keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.12keV

ID : No close library match

NP406Th.cttn

MCB # 1 ACQ 02-13-95 AT 16:19:36 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-406 Th 2/14/95

ROI # 10-1 RANGE : 10 = 396.63keV to 64 = 409.14keV
AREA : Gross = 261 Net = 259 +/- 17
CENTROID : 40.25 = 403.64keV
SHAPE : Fwhm = 1.66keV Fwtm = 4.38keV

ID : No close library match

ROI # 10-2 RANGE : 261 = 454.76keV to 354 = 476.30keV
AREA : Gross = 1309 Net = 1308 +/- 36
CENTROID : 334.34 = 471.75keV
SHAPE : Fwhm = 3.61keV Fwtm = 13.24keV

ID : No close library match

ROI # 10-3 RANGE : 561 = 524.24keV to 681 = 552.03keV
AREA : Gross = 13906 Net = 13664 +/- 135
CENTROID : 653.78 = 545.72keV
SHAPE : Fwhm = 4.99keV Fwtm = 15.81keV

ID : No close library match

ROI # 10-4 RANGE : 703 = 557.12keV to 791 = 577.50keV
AREA : Gross = 3647 Net = 3353 +/- 86
CENTROID : 761.86 = 570.75keV
SHAPE : Fwhm = 7.14keV Fwtm = 14.61keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP407U.CHN
NP407TH.CHN

Sample # **NOPI-407**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5098	1.0009	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **333.377** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0058869**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
290	10259	14635	3764

U-238 counts	U-234 counts	U-232 counts
4587	4155	12074

Bkgd	Bkgd	Bkgd	bkgd
3	5	58	174

bkgd	bkgd	bkgd
4	5	7

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
287	10254	14176.248	3590

U-238* counts	U-234* counts	U-232sp counts
4583	4150	11996.378

U-238(dpm/g)= **334.49439** ± **5.801622**
U-234(dpm/g)= **302.8915** ± **5.447809**

Th-232(dpm/g)= **10.442987** ± **0.619279**
Th-230(dpm/g)= **373.10939** ± **4.80435**

U-234/U-238= **0.9055204** ± **0.019393**
Th-230/U-234= **1.2318252** ± **0.022652**
Th-230/Th-232= **35.728223** ± **2.12748**
U234/Th-232= **29.004296** ± **1.797354**

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

U(ppb)= **448374.22**

Th(ppb)= **42739.21**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP407 U.C.H.N

MCB # 1 ACQ 02-14-95 AT 11:10:30 RT : 20002.6 LT : 20000.0
No detector description was entered
NOPI-407 U 2/15/95

ROI # 6-1 RANGE : 33 = 3.92keV to 152 = 4.23keV
AREA : Gross = 4828 Net = 4587 +/- 96
CENTROID : 124.71 = 4.16keV
SHAPE : Fwhm = 0.15keV Fwtm = 0.23keV

ID : No close library match

ROI # 6-2 RANGE : 261 = 4.52keV to 369 = 4.81keV
AREA : Gross = 4574 Net = 4155 +/- 107
CENTROID : 338.65 = 4.73keV
SHAPE : Fwhm = 0.14keV Fwtm = 0.21keV

ID : No close library match

ROI # 6-3 RANGE : 468 = 5.07keV to 578 = 5.36keV
AREA : Gross = 12889 Net = 12074 +/- 162
CENTROID : 542.17 = 5.26keV
SHAPE : Fwhm = 0.13keV Fwtm = 0.22keV

ID : No close library match

NP407Th.ctw

MCB # 1 ACQ 02-13-95 AT 16:19:35 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-407 Th 2/14/95

ROI # 5-1 RANGE : 29 = 3.82keV to 92 = 3.99keV
AREA : Gross = 291 Net = 290 +/- 17
CENTROID : 71.59 = 3.94keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.08keV

ID : No close library match

ROI # 5-2 RANGE : 223 = 4.34keV to 350 = 4.68keV
AREA : Gross = 10385 Net = 10259 +/- 113
CENTROID : 324.99 = 4.62keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.16keV

ID : No close library match

ROI # 5-3 RANGE : 511 = 5.11keV to 627 = 5.43keV
AREA : Gross = 14927 Net = 14635 +/- 142
CENTROID : 603.57 = 5.36keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.16keV

ID : No close library match

ROI # 5-4 RANGE : 640 = 5.46keV to 723 = 5.68keV
AREA : Gross = 4079 Net = 3764 +/- 89
CENTROID : 697.80 = 5.62keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.16keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP408U.CHN
NP408TH.CHN

Sample # **NOPI-408**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5007	0.9987	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **1248.873** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0061918**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
345	559	18682	4990

U-238 counts	U-234 counts	U-232 counts
429	432	31514

Bkgd	Bkgd	Bkgd	bkgd
5	10	32	113

bkgd	bkgd	bkgd
6	10	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
340	549	18149.337	4877

U-238* counts	U-234* counts	U-232sp counts
423	422	31310.133

U-238(dpm/g)= **12.017419** ± **0.584142**
U-234(dpm/g)= **11.989009** ± **0.580762**

Th-232(dpm/g)= **9.8172288** ± **0.5334**
Th-230(dpm/g)= **15.851937** ± **0.680422**

U-234/U-238= **0.9976359** ± **0.067999**
Th-230/U-234= **1.3222058** ± **0.084701**
Th-230/Th-232= **1.6147059** ± **0.110551**
U234/Th-232= **1.2212213** ± **0.088895**

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

U(ppb)= **16108.793**

Th(ppb)= **40178.215**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP4084 (HW)

MCB # 1 ACQ 02-14-95 AT 11:10:30 RT : 74932.4 LT : 74926.6
No detector description was entered
NOPI-408 U 2/15/95

ROI # 7-1 RANGE : 90 to 153
AREA : Gross = 430 Net = 429 +/- 21
CENTROID : 123.92
SHAPE : Fwhm = 4.98 Channels Fwtm = 39.30 Channels

ROI # 7-2 RANGE : 344 to 398
AREA : Gross = 507 Net = 432 +/- 32
CENTROID : 381.02
SHAPE : Fwhm = 16.05 Channels Fwtm = 42.95 Channels

ROI # 7-3 RANGE : 563 to 642
AREA : Gross = 32501 Net = 31514 +/- 209
CENTROID : 616.15
SHAPE : Fwhm = 18.11 Channels Fwtm = 49.84 Channels

NP408Th.ctw

MCB # 1 ACQ 02-13-95 AT 16:19:35 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-408 Th 2/14/95

ROI # 6-1 RANGE : 17 = 3.88keV to 79 = 4.04keV
AREA : Gross = 361 Net = 345 +/- 22
CENTROID : 63.34 = 4.00keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.06keV

ID : No close library match

ROI # 6-2 RANGE : 265 = 4.53keV to 337 = 4.72keV
AREA : Gross = 577 Net = 559 +/- 28
CENTROID : 312.13 = 4.66keV
SHAPE : Fwhm = 0.03keV Fwtm = 0.11keV

ID : No close library match

ROI # 6-3 RANGE : 523 = 5.21keV to 618 = 5.46keV
AREA : Gross = 19195 Net = 18682 +/- 162
CENTROID : 594.32 = 5.40keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.15keV

ID : No close library match

ROI # 6-4 RANGE : 637 = 5.51keV to 715 = 5.72keV
AREA : Gross = 5386 Net = 4990 +/- 99
CENTROID : 687.57 = 5.65keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.14keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP409U.CHN
NP409TH.CHN

Sample # **NOPI-409**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5018	1.0016	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **1249.688** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0061921**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
78	250	10223	2672

U-238 counts	U-234 counts	U-232 counts
265	270	32074

Bkgd	Bkgd	Bkgd	bkgd
4	9	20	31

bkgd	bkgd	bkgd
4	3	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
74	241	10042.549	2641

U-238* counts	U-234* counts	U-232sp counts
261	267	31864.69

U-238(dpm/g)= **7.2910949** ± **0.449735**
U-234(dpm/g)= **7.4587062** ± **0.455829**

Th-232(dpm/g)= **3.8642457** ± **0.439206**
Th-230(dpm/g)= **12.584908** ± **0.805613**

U-234/U-238= **1.0229885** ± **0.088459**
Th-230/U-234= **1.6872777** ± **0.148094**
Th-230/Th-232= **3.2567568** ± **0.422382**
U234/Th-232= **1.9301842** ± **0.249085**

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

U(ppb)= **9773.3744**

Th(ppb)= **15814.901**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP409U.CITN

MCB # 1 ACQ 02-14-95 AT 11:10:30 RT : 74981.3 LT : 74975.5
No detector description was entered
NOPI-409 U 2/15/95

ROI # 8-1 RANGE : 91 = 411.64keV to 149 = 424.87keV
AREA : Gross = 501 Net = 265 +/- 48
CENTROID : 134.27 = 421.51keV
SHAPE : Fwhm = 1.73keV Fwtm = 6.81keV

ID : No close library match

ROI # 8-2 RANGE : 348 = 470.29keV to 395 = 481.02keV
AREA : Gross = 546 Net = 270 +/- 47
CENTROID : 378.04 = 477.15keV
SHAPE : Fwhm = 1.30keV Fwtm = 6.73keV

ID : No close library match

ROI # 8-3 RANGE : 559 = 518.45keV to 644 = 537.85keV
AREA : Gross = 34008 Net = 32074 +/- 241
CENTROID : 617.62 = 531.83keV
SHAPE : Fwhm = 4.31keV Fwtm = 12.01keV

ID : No close library match

NP 409Th.CHW

MCB # 1 ACQ 02-13-95 AT 16:19:35 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-409 Th 2/14/95

ROI # 7-1 RANGE : 24 to 67
AREA : Gross = 166 Net = 78 +/- 25
CENTROID : 40.39
SHAPE : Fwhm = 2.99 Channels Fwtm = 7.40 Channels

ROI # 7-2 RANGE : 285 to 359
AREA : Gross = 325 Net = 250 +/- 33
CENTROID : 331.33
SHAPE : Fwhm = 2.62 Channels Fwtm = 34.68 Channels

ROI # 7-3 RANGE : 546 to 689
AREA : Gross = 11352 Net = 10223 +/- 190
CENTROID : 660.52
SHAPE : Fwhm = 23.51 Channels Fwtm = 79.87 Channels

ROI # 7-4 RANGE : 715 to 800
AREA : Gross = 3145 Net = 2672 +/- 95
CENTROID : 770.33
SHAPE : Fwhm = 29.13 Channels Fwtm = 60.47 Channels

U and Th Isotope Activities

FILENAME= NP410U.CHN
NP410TH.CHN

Sample # **NOPI-410**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5067	1.003	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **1250.137** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0061923**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
169	319	9409	2684

U-238 counts	U-234 counts	U-232 counts
313	324	34312

Bkgd	Bkgd	Bkgd	bkgd
1	7	14	47

bkgd	bkgd	bkgd
2	2	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
168	312	9136.4644	2637

U-238* counts	U-234* counts	U-232sp counts
311	322	34088.913

U-238(dpm/g)= **8.053702** ± **0.457294**
U-234(dpm/g)= **8.3385596** ± **0.465435**

Th-232(dpm/g)= **9.5630065** ± **0.742193**
Th-230(dpm/g)= **17.759869** ± **1.011078**

U-234/U-238= **1.0353698** ± **0.082058**
Th-230/U-234= **2.1298486** ± **0.167991**
Th-230/Th-232= **1.8571429** ± **0.176692**
U234/Th-232= **0.8719601** ± **0.083358**

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

U(ppb)= **10795.614**

Th(ppb)= **39137.779**

Spike 25A 10/9/92
Th-228 (dpm/g)= **2630.1478** Spike 25B **262.7322** Spide 25C **26.259396**

NP410M.CHW

MCB # 1 ACQ 02-14-95 AT 11:10:31 RT : 75008.2 LT : 75002.4
No detector description was entered
NOPI-410 U 2/15/95

ROI # 9-1 RANGE : 97 = 410.58keV to 152 = 423.29keV
AREA : Gross = 369 Net = 313 +/- 28
CENTROID : 139.78 = 420.47keV
SHAPE : Fwhm = 2.82keV Fwtm = 6.34keV

ID : No close library match

ROI # 9-2 RANGE : 358 = 470.90keV to 404 = 481.53keV
AREA : Gross = 472 Net = 324 +/- 37
CENTROID : 384.59 = 477.05keV
SHAPE : Fwhm = 3.92keV Fwtm = 4.83keV

ID : No close library match

ROI # 9-3 RANGE : 570 = 519.90keV to 649 = 538.16keV
AREA : Gross = 35086 Net = 34312 +/- 209
CENTROID : 623.31 = 532.22keV
SHAPE : Fwhm = 3.70keV Fwtm = 11.28keV

ID : No close library match

NP410Th.CHW

MCB # 1 ACQ 02-13-95 AT 16:19:36 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-410 Th 2/14/95

ROI # 8-1 RANGE : 23 = 396.12keV to 68 = 406.39keV
AREA : Gross = 214 Net = 169 +/- 22
CENTROID : 51.57 = 402.64keV
SHAPE : Fwhm = 1.24keV Fwtm = 4.25keV

ID : No close library match

ROI # 8-2 RANGE : 267 = 451.81keV to 365 = 474.17keV
AREA : Gross = 416 Net = 319 +/- 43
CENTROID : 328.00 = 465.73keV
SHAPE : Fwhm = 1.45keV Fwtm = 6.54keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.005 +/- 0.00

ROI # 8-3 RANGE : 590 = 525.53keV to 687 = 547.67keV
AREA : Gross = 10144 Net = 9409 +/- 144
CENTROID : 663.45 = 542.29keV
SHAPE : Fwhm = 4.96keV Fwtm = 15.86keV

ID : No close library match

ROI # 8-4 RANGE : 713 = 553.60keV to 804 = 574.37keV
AREA : Gross = 2928 Net = 2684 +/- 79
CENTROID : 772.61 = 567.20keV
SHAPE : Fwhm = 6.47keV Fwtm = 14.02keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP411U.CHN
NP411TH.CHN

Sample # **NOPI-411**
Analyst **JDP**

Sep. date **2/8/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5059	0.9994	454.83	1/22/93	747	445.96244

Th-228/U-232= **0.5891353**

Counting time for Th= **1000.097** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for Th-228= **0.9947095**

Counting time for U= **1250.602** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for U-232= **1.0061924**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
188	471	12212	3224

U-238 counts	U-234 counts	U-232 counts
406	413	30041

Bkgd	Bkgd	Bkgd	bkgd
1	3	25	57

bkgd	bkgd	bkgd
2	1	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
187	468	11896.075	3167

U-238* counts	U-234* counts	U-232sp counts
404	412	29844.192

U-238(dpm/g)= **11.925991** ± **0.595863**
U-234(dpm/g)= **12.162149** ± **0.60256**

Th-232(dpm/g)= **8.1587926** ± **0.599604**
Th-230(dpm/g)= **20.418797** ± **0.95882**

U-234/U-238= **1.019802** ± **0.071272**
Th-230/U-234= **1.6788806** ± **0.113178**
Th-230/Th-232= **2.5026738** ± **0.215902**
U234/Th-232= **1.4906801** ± **0.132122**

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

U(ppb)= **15986.238**

Th(ppb)= **33390.862**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2630.1478 262.7322 26.259396

NP411U.CHW

MCB # 1 ACQ 02-14-95 AT 11:10:31 RT : 75036.1 LT : 75030.3
No detector description was entered
NOPI-411 U 2/15/95

ROI # 10-1 RANGE : 80 = 412.85keV to 142 = 427.20keV
AREA : Gross = 437 Net = 406 +/- 26
CENTROID : 119.27 = 421.94keV
SHAPE : Fwhm = 1.15keV Fwtm = 8.12keV

ID : No close library match

ROI # 10-2 RANGE : 344 = 473.98keV to 392 = 485.10keV
AREA : Gross = 529 Net = 413 +/- 35
CENTROID : 373.12 = 480.73keV
SHAPE : Fwhm = 1.94keV Fwtm = 5.77keV

ID : No close library match

ROI # 10-3 RANGE : 556 = 523.08keV to 632 = 540.68keV
AREA : Gross = 30734 Net = 30041 +/- 196
CENTROID : 607.94 = 535.11keV
SHAPE : Fwhm = 4.08keV Fwtm = 11.45keV

ID : No close library match

NP411Th-CHW

MCB # 1 ACQ 02-13-95 AT 16:19:36 RT : 60005.8 LT : 60000.0
No detector description was entered
NOPI-411 Th 2/14/95

ROI # 9-1 RANGE : 29 = 394.86keV to 75 = 405.50keV
AREA : Gross = 218 Net = 188 +/- 20
CENTROID : 55.29 = 400.94keV
SHAPE : Fwhm = 2.04keV Fwtm = 9.31keV

ID : No close library match

ROI # 9-2 RANGE : 277 = 452.18keV to 366 = 472.75keV
AREA : Gross = 472 Net = 471 +/- 22
CENTROID : 345.17 = 467.94keV
SHAPE : Fwhm = 3.57keV Fwtm = 14.93keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.008 +/- 0.00

ROI # 9-3 RANGE : 582 = 522.67keV to 691 = 547.87keV
AREA : Gross = 12651 Net = 12212 +/- 141
CENTROID : 666.81 = 542.28keV
SHAPE : Fwhm = 4.89keV Fwtm = 14.69keV

ID : No close library match

ROI # 9-4 RANGE : 717 = 553.88keV to 805 = 574.22keV
AREA : Gross = 3402 Net = 3224 +/- 75
CENTROID : 773.78 = 567.00keV
SHAPE : Fwhm = 8.79keV Fwtm = 13.23keV

ID : No close library match

U and Th Isotope Activities

FILENAME= NP212U.CHN
NP212TH.CHN

Sample # **NOPI-212**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5356	0.498	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **1515.7** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.0053211**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
322	515	7545	1479

U-238 counts	U-234 counts	U-232 counts
913	1151	27414

Bkgd	Bkgd	Bkgd	bkgd
3	5	13	22

bkgd	bkgd	bkgd
11	10	12

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
319	510	7182.436	1457

U-238* counts	U-234* counts	U-232sp counts
902	1141	27256.963

U-238(dpm/g)= **13.719066** ± **0.461534**
U-234(dpm/g)= **17.354163** ± **0.522152**

Th-232(dpm/g)= **10.915185** ± **0.621124**
Th-230(dpm/g)= **17.45061** ± **0.794776**

U-234/U-238= **1.2649667** ± **0.056061**
Th-230/U-234= **1.0055576** ± **0.053309**
Th-230/Th-232= **1.5987461** ± **0.113582**
U234/Th-232= **1.58991** ± **0.102342**

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

U(ppb)= **18389.772**

Th(ppb)= **44671.738**

Spike 25A 10/9/92 Spike 25B Spike 25C
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

NP212U.CHW

MCB # 1 ACQ 02-21-95 AT 11:24:50 RT : 90942.0 LT : 90936.6
No detector description was entered
NOPI-212 U 2/22/95

ROI # 1-1 RANGE : 102 to 162
AREA : Gross = 914 Net = 913 +/- 30
CENTROID : 146.52
SHAPE : Fwhm = 13.73 Channels Fwtm = 37.21 Channels

ROI # 1-2 RANGE : 325 to 386
AREA : Gross = 1231 Net = 1151 +/- 44
CENTROID : 374.37
SHAPE : Fwhm = 10.68 Channels Fwtm = 38.12 Channels

ROI # 1-3 RANGE : 538 to 611
AREA : Gross = 28364 Net = 27414 +/- 195
CENTROID : 592.48
SHAPE : Fwhm = 12.36 Channels Fwtm = 40.50 Channels

NP212Th.CHW

MCB # 1 ACQ 02-22-95 AT 13:02:48 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-212 Th 2/23/95

ROI # 1-1 RANGE : 31 to 85
AREA : Gross = 336 Net = 322 +/- 21
CENTROID : 72.01
SHAPE : Fwhm = 13.47 Channels Fwtm = 36.27 Channels

ROI # 1-2 RANGE : 295 to 350
AREA : Gross = 543 Net = 515 +/- 27
CENTROID : 337.20
SHAPE : Fwhm = 10.86 Channels Fwtm = 29.95 Channels

ROI # 1-3 RANGE : 564 to 652
AREA : Gross = 7633 Net = 7545 +/- 94
CENTROID : 632.23
SHAPE : Fwhm = 14.23 Channels Fwtm = 54.69 Channels

ROI # 1-4 RANGE : 685 to 756
AREA : Gross = 1540 Net = 1479 +/- 46
CENTROID : 733.07
SHAPE : Fwhm = 24.81 Channels Fwtm = 43.54 Channels

U and Th Isotope Activities

FILENAME= NP214U.CHN
NP214TH.CHN

Sample # **NOPI-214**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5085	0.4975	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **1516.213** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.0053213**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
227	2138	6223	1356
Bkgd	Bkgd	Bkgd	bkgd
1	2	9	16

U-238 counts	U-234 counts	U-232 counts
2408	3716	22132
bkgd	bkgd	bkgd
3	9	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
226	2136	5955.4269	1340

U-238* counts	U-234* counts	U-232sp counts
2405	3707	22006.896

U-238(dpm/g)= **47.672194** ± **1.022972**
U-234(dpm/g)= **73.480591** ± **1.302681**

Th-232(dpm/g)= **9.8134361** ± **0.663114**
Th-230(dpm/g)= **92.749997** ± **2.325087**

U-234/U-238= **1.5413721** ± **0.040324**
Th-230/U-234= **1.262238** ± **0.034263**
Th-230/Th-232= **9.4513274** ± **0.659769**
U234/Th-232= **7.4877536** ± **0.523086**

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

U(ppb)= **63902.364**

Th(ppb)= **40162.693**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

NP214U.CHW

MCB # 1 ACQ 02-21-95 AT 11:24:50 RT : 90972.8 LT : 90967.4
No detector description was entered
NOPI-214 U 2/22/95

ROI # 2-1 RANGE : 113 to 173
AREA : Gross = 2456 Net = 2408 +/- 53
CENTROID : 154.45
SHAPE : Fwhm = 10.70 Channels Fwtm = 36.83 Channels

ROI # 2-2 RANGE : 332 to 400
AREA : Gross = 3854 Net = 3716 +/- 72
CENTROID : 383.17
SHAPE : Fwhm = 11.57 Channels Fwtm = 34.79 Channels

ROI # 2-3 RANGE : 549 to 619
AREA : Gross = 22735 Net = 22132 +/- 169
CENTROID : 601.14
SHAPE : Fwhm = 12.53 Channels Fwtm = 41.29 Channels

NP214TH-CHW

MCB # 1 ACQ 02-22-95 AT 13:02:49 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-214 Th 2/23/95

ROI # 2-1 RANGE : 39 to 94
AREA : Gross = 241 Net = 227 +/- 19
Could not properly fit the peak.

ROI # 2-2 RANGE : 279 to 361
AREA : Gross = 2159 Net = 2138 +/- 49
CENTROID : 342.68
SHAPE : Fwhm = 19.71 Channels Fwtm = 49.31 Channels

ROI # 2-3 RANGE : 558 to 657
AREA : Gross = 6448 Net = 6223 +/- 99
CENTROID : 638.82
SHAPE : Fwhm = 18.03 Channels Fwtm = 56.92 Channels

ROI # 2-4 RANGE : 686 to 762
AREA : Gross = 1431 Net = 1356 +/- 47
CENTROID : 741.03
SHAPE : Fwhm = 28.37 Channels Fwtm = 46.80 Channels

U and Th Isotope Activities

FILENAME= NP215U.CHN
NP215TH.CHN

Sample # **NOPI-215**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5176	0.4991	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **1517.463** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.0053217**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
173	2932	6272	1060

U-238 counts	U-234 counts	U-232 counts
5022	5124	20119

Bkgd	Bkgd	Bkgd	bkgd
4	8	17	26

bkgd	bkgd	bkgd
3	6	14

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
169	2924	6070.003	1034

U-238* counts	U-234* counts	U-232sp counts
5019	5118	19998.574

U-238(dpm/g)= **107.89919** ± **1.702033**
U-234(dpm/g)= **110.0275** ± **1.721726**

Th-232(dpm/g)= **7.0960151** ± **0.54689**
Th-230(dpm/g)= **122.77366** ± **2.746683**

U-234/U-238= **1.019725** ± **0.020248**
Th-230/U-234= **1.1158451** ± **0.025839**
Th-230/Th-232= **17.301775** ± **1.353681**
U234/Th-232= **15.505534** ± **1.219395**

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

U(ppb)= **144633.86**

Th(ppb)= **29041.314**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

NP215U.CHW

MCB # 1 ACQ 02-21-95 AT 11:24:51 RT : 91047.8 LT : 91042.4
No detector description was entered
NOPI-215 U 2/22/95

ROI # 9-1 RANGE : 66 to 161
AREA : Gross = 5198 Net = 5022 +/- 88
CENTROID : 135.95
SHAPE : Fwhm = 20.28 Channels Fwtm = 51.67 Channels

ROI # 9-2 RANGE : 311 to 410
AREA : Gross = 5374 Net = 5124 +/- 95
CENTROID : 384.32
SHAPE : Fwhm = 22.30 Channels Fwtm = 51.67 Channels

ROI # 9-3 RANGE : 562 to 646
AREA : Gross = 21253 Net = 20119 +/- 187
CENTROID : 621.73
SHAPE : Fwhm = 18.34 Channels Fwtm = 51.87 Channels

NP215 Th. CHW

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-215 Th 2/23/95

ROI # 7-1 RANGE : 23 to 72
AREA : Gross = 205 Net = 173 +/- 20
CENTROID : 53.66
SHAPE : Fwhm = 1.99 Channels Fwtm = 20.44 Channels

ROI # 7-2 RANGE : 276 to 363
AREA : Gross = 3020 Net = 2932 +/- 64
CENTROID : 337.56
SHAPE : Fwhm = 17.20 Channels Fwtm = 60.58 Channels

ROI # 7-3 RANGE : 573 to 685
AREA : Gross = 6385 Net = 6272 +/- 91
CENTROID : 657.06
SHAPE : Fwhm = 19.56 Channels Fwtm = 64.13 Channels

ROI # 7-4 RANGE : 718 to 794
AREA : Gross = 1163 Net = 1060 +/- 48
CENTROID : 767.37
SHAPE : Fwhm = 23.45 Channels Fwtm = 50.87 Channels

U and Th Isotope Activities

FILENAME= NP216U.CHN
NP216TH.CHN

Sample # **NOPI-216**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5183	0.4977	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **416.695** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.0049547**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
146	10718	6006	1227

U-238 counts	U-234 counts	U-232 counts
3773	3935	3043

Bkgd	Bkgd	Bkgd	bkgd
1	7	13	40

bkgd	bkgd	bkgd
9	10	14

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
145	10711	5822.2034	1187

U-238* counts	U-234* counts	U-232sp counts
3764	3925	3014.0662

U-238(dpm/g)= **534.6751** ± **13.02748**
U-234(dpm/g)= **557.54511** ± **13.45929**

Th-232(dpm/g)= **6.3210683** ± **0.529456**
Th-230(dpm/g)= **466.93078** ± **7.526153**

U-234/U-238= **1.0427736** ± **0.02376**
Th-230/U-234= **0.8374762** ± **0.01561**
Th-230/Th-232= **73.868966** ± **6.154937**
U234/Th-232= **88.204253** ± **7.688744**

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

U(ppb)= **716707.17**

Th(ppb)= **25869.749**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

NP216U.CHW

MCB # 1 ACQ 02-21-95 AT 15:29:25 RT : 25001.7 LT : 25000.0
No detector description was entered
NOPI-216 U 2/22/95

ROI # 8-1 RANGE : 27 to 154
AREA : Gross = 4068 Net = 3773 +/- 99
CENTROID : 116.52
SHAPE : Fwhm = 57.37 Channels Fwtm = 98.04 Channels

ROI # 8-2 RANGE : 274 to 404
AREA : Gross = 4045 Net = 3935 +/- 79
CENTROID : 368.08
SHAPE : Fwhm = 54.39 Channels Fwtm = 100.29 Channels

ROI # 8-3 RANGE : 552 to 642
AREA : Gross = 3906 Net = 3043 +/- 124
CENTROID : 615.15
SHAPE : Fwhm = 46.70 Channels Fwtm = 65.21 Channels

NP 216Th.ctw

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-216 Th 2/23/95

ROI # 8-1 RANGE : 20 to 67
AREA : Gross = 274 Net = 146 +/- 32
CENTROID : 45.86
SHAPE : Fwhm = 4.84 Channels Fwtm = 14.88 Channels

ROI # 8-2 RANGE : 260 to 361
AREA : Gross = 11075 Net = 10718 +/- 128
CENTROID : 336.16
SHAPE : Fwhm = 24.61 Channels Fwtm = 62.29 Channels

ROI # 8-3 RANGE : 585 to 685
AREA : Gross = 6275 Net = 6006 +/- 101
CENTROID : 659.36
SHAPE : Fwhm = 20.22 Channels Fwtm = 68.73 Channels

ROI # 8-4 RANGE : 716 to 794
AREA : Gross = 1437 Net = 1227 +/- 61
CENTROID : 768.20
SHAPE : Fwhm = 23.63 Channels Fwtm = 49.54 Channels

U and Th Isotope Activities

FILENAME= NP216U3.CHN
NP216TH3.CHNSample # **NOPI-216**
Analyst **JDP**Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5183	0.4977	454.83	1/22/93	755	445.8684

Th-228/U-232= **0.5928116**

Counting time for Th=	940.75	(mins.)	Counting time for U=	940.73	(mins.)
Days btwn. sep. and count.=	47	(days)	Days btwn. sep. and count.=	47	(days)
CF for Th-228=	0.9541361		CF for U-232=	1.0445884	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
132	7564	4291	1312

U-238 counts	U-234 counts	U-232 counts
3351	3355	3353

Bkgd	Bkgd	Bkgd	bkgd
1	3	11	19

bkgd	bkgd	bkgd
5	6	8

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
131	7561	4282.9101	1293

U-238* counts	U-234* counts	U-232sp counts
3346	3349	3202.2181

U-238(dpm/g)= **447.3714** ± **10.92776**
 U-234(dpm/g)= **447.7725** ± **10.9343**

Th-232(dpm/g)= **7.763225** ± **0.686016**
 Th-230(dpm/g)= **448.0744** ± **8.563393**

U-234/U-238= **1.000897** ± **0.024445**
 Th-230/U-234= **1.000674** ± **0.020757**
 Th-230/Th-232= **57.71756** ± **5.067313**
 U234/Th-232= **57.67866** ± **5.287943**

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

U(ppb)= **599680.5**Th(ppb)= **31771.95**

Spike 25A	Spike 25B	Spide 25C
10/9/92		
Th-228 (dpm/g)= 2646.0026	264.31598	26.417691

NP216U3.CHN

MCB # 1 ACQ 04-03-95 AT 14:58:45 RT : 56444.0 LT : 56437.0
No detector description was entered
NOPI-216 U 4/4/95

ROI # 8-1 RANGE : 45 to 148
AREA : Gross = 3592 Net = 3351 +/- 85
CENTROID : 121.57
SHAPE : Fwhm = 39.02 Channels Fwtm = 76.08 Channels

ROI # 8-2 RANGE : 305 to 398
AREA : Gross = 3636 Net = 3355 +/- 86
CENTROID : 372.82
SHAPE : Fwhm = 41.00 Channels Fwtm = 66.47 Channels

ROI # 8-3 RANGE : 556 to 638
AREA : Gross = 3655 Net = 3353 +/- 85
CENTROID : 613.68
SHAPE : Fwhm = 36.69 Channels Fwtm = 66.61 Channels

NOPI-216 Th 3.0 HN

MCB # 1 ACQ 04-03-95 AT 14:58:44 RT : 56445.0 LT : 56438.0
No detector description was entered
NOPI-216 Th 4/4/95

ROI # 2-1 RANGE : 35 to 90
AREA : Gross = 132 Net = 132 +/- 11
CENTROID : 77.90
SHAPE : Fwhm = 1.40 Channels Fwtm = 20.32 Channels

ROI # 2-2 RANGE : 256 to 362
AREA : Gross = 7654 Net = 7564 +/- 95
CENTROID : 341.69
SHAPE : Fwhm = 19.04 Channels Fwtm = 53.22 Channels

ROI # 2-3 RANGE : 565 to 657
AREA : Gross = 4478 Net = 4291 +/- 84
CENTROID : 638.07
SHAPE : Fwhm = 16.28 Channels Fwtm = 55.42 Channels

ROI # 2-4 RANGE : 680 to 763
AREA : Gross = 1497 Net = 1312 +/- 61
CENTROID : 740.93
SHAPE : Fwhm = 31.09 Channels Fwtm = 51.40 Channels

U and Th Isotope Activities

FILENAME= NP217U.CHN
NP217TH.CHN

Sample # **NOPI-217**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5166	0.4949	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **1517.903** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.0053218**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
134	4005	6862	1123

U-238 counts	U-234 counts	U-232 counts
6778	6873	22363

Bkgd	Bkgd	Bkgd	bkgd
0	3	23	46

bkgd	bkgd	bkgd
1	7	36

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
134	4002	6690.3648	1077

U-238* counts	U-234* counts	U-232sp counts
6777	6866	22208.809

U-238(dpm/g)= **130.34128** ± **1.807251**
U-234(dpm/g)= **132.05301** ± **1.821248**

Th-232(dpm/g)= **5.071559** ± **0.442373**
Th-230(dpm/g)= **151.46551** ± **3.01191**

U-234/U-238= **1.0131327** ± **0.017343**
Th-230/U-234= **1.1470054** ± **0.022802**
Th-230/Th-232= **29.865672** ± **2.622807**
U234/Th-232= **26.037952** ± **2.299408**

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

U(ppb)= **174716.44**

Th(ppb)= **20755.978**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

MCB # 1 ACQ 02-21-95 AT 11:24:52 RT : 91074.2 LT : 91068.8
No detector description was entered
NOPI-217 U 2/22/95

ROI # 11-1 RANGE : 80 to 168
AREA : Gross = 6880 Net = 6778 +/- 90
CENTROID : 139.05
SHAPE : Fwhm = 26.78 Channels Fwtm = 52.68 Channels

ROI # 11-2 RANGE : 328 to 417
AREA : Gross = 7008 Net = 6873 +/- 94
CENTROID : 388.12
SHAPE : Fwhm = 28.69 Channels Fwtm = 54.88 Channels

ROI # 11-3 RANGE : 570 to 655
AREA : Gross = 23123 Net = 22363 +/- 180
CENTROID : 626.73
SHAPE : Fwhm = 23.56 Channels Fwtm = 55.82 Channels

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-217 Th 2/23/95

ROI # 9-1 RANGE : 22 to 68
AREA : Gross = 182 Net = 134 +/- 21
CENTROID : 46.86
SHAPE : Fwhm = 1.42 Channels Fwtm = 14.98 Channels

ROI # 9-2 RANGE : 269 to 366
AREA : Gross = 4103 Net = 4005 +/- 74
CENTROID : 343.41
SHAPE : Fwhm = 22.38 Channels Fwtm = 57.64 Channels

ROI # 9-3 RANGE : 592 to 686
AREA : Gross = 7081 Net = 6862 +/- 101
CENTROID : 663.19
SHAPE : Fwhm = 21.70 Channels Fwtm = 65.52 Channels

ROI # 9-4 RANGE : 728 to 798
AREA : Gross = 1334 Net = 1123 +/- 58
CENTROID : 779.17
SHAPE : Fwhm = 25.36 Channels Fwtm = 48.37 Channels

U and Th Isotope Activities

FILENAME= NP218U.CHN
NP218TH.CHN

Sample # **NOPI-218**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)
0.5283	0.4943	454.83	1/22/93	755	445.86842

Th-228/U-232= **0.5928116**

Counting time for Th= **833.375** (mins.)
Days btwn. sep. and count.= **6** (days)
CF for Th-228= **0.9937803**

Counting time for U= **1518.307** (mins.)
Days btwn. sep. and count.= **5** (days)
CF for U-232= **1.005322**

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
154	1678	6364	1101

U-238 counts	U-234 counts	U-232 counts
2566	2914	20833

Bkgd	Bkgd	Bkgd	bkgd
0	1	14	56

bkgd	bkgd	bkgd
16	17	18

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
154	1677	6180.0109	1045

U-238* counts	U-234* counts	U-232sp counts
2550	2897	20704.81

U-238(dpm/g)= **51.379** ± **1.07493**
U-234(dpm/g)= **58.370573** ± **1.154457**

Th-232(dpm/g)= **6.1626131** ± **0.50257**
Th-230(dpm/g)= **67.108456** ± **1.841612**

U-234/U-238= **1.1360784** ± **0.030756**
Th-230/U-234= **1.1496967** ± **0.035233**
Th-230/Th-232= **10.88961** ± **0.916893**
U234/Th-232= **9.4717244** ± **0.794825**

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

U(ppb)= **68871.165**

Th(ppb)= **25221.252**

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

MCB # 1 ACQ 02-21-95 AT 11:24:52 RT : 91098.4 LT : 91093.0
No detector description was entered
NOPI-218 U 2/22/95

ROI # 12-1 RANGE : 81 to 161
AREA : Gross = 2687 Net = 2566 +/- 64
CENTROID : 136.05
SHAPE : Fwhm = 22.24 Channels Fwtm = 51.13 Channels

ROI # 12-2 RANGE : 325 to 412
AREA : Gross = 3090 Net = 2914 +/- 73
CENTROID : 386.88
SHAPE : Fwhm = 22.94 Channels Fwtm = 51.89 Channels

ROI # 12-3 RANGE : 570 to 648
AREA : Gross = 22032 Net = 20833 +/- 188
CENTROID : 624.01
SHAPE : Fwhm = 19.74 Channels Fwtm = 51.90 Channels

NP218Th.CHN

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-218 Th 2/23/95

ROI # 10-1 RANGE : 24 to 59
AREA : Gross = 190 Net = 154 +/- 18
CENTROID : 39.92
SHAPE : Fwhm = 6.49 Channels Fwtm = 21.74 Channels

ROI # 10-2 RANGE : 263 to 349
AREA : Gross = 1721 Net = 1678 +/- 48
CENTROID : 327.64
SHAPE : Fwhm = 20.90 Channels Fwtm = 53.07 Channels

ROI # 10-3 RANGE : 578 to 674
AREA : Gross = 6476 Net = 6364 +/- 90
CENTROID : 648.31
SHAPE : Fwhm = 20.81 Channels Fwtm = 63.09 Channels

ROI # 10-4 RANGE : 711 to 783
AREA : Gross = 1103 Net = 1101 +/- 34
CENTROID : 759.37
SHAPE : Fwhm = 24.85 Channels Fwtm = 55.12 Channels

U and Th Isotope Activities

FILENAME= NP219U.CHN
NP219TH.CHN

Sample # **NOPI-219**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.5246	0.4953	454.83	1/22/93	755	445.86842	0.5928116

Counting time for Th=	833.375	(mins.)	Counting time for U=	1518.76	(mins.)
Days btwn. sep. and count.=	6	(days)	Days btwn. sep. and count.=	5	(days)
CF for Th-228=	0.9937803		CF for U-232=	1.0053221	

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
223	1201	5838	937

U-238 counts	U-234 counts	U-232 counts
1661	2032	19521

Bkgd	Bkgd	Bkgd	bkgd
0	2	32	22

bkgd	bkgd	bkgd
29	16	10

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
223	1199	5570.5393	915

U-238* counts	U-234* counts	U-232sp counts
1632	2016	19407.71

U-238(dpm/g)= 35.399132 ± 0.904774
U-234(dpm/g)= 43.728339 ± 1.019305

Th-232(dpm/g)= 9.9901288 ± 0.681646
Th-230(dpm/g)= 53.713742 ± 1.701913

U-234/U-238= 1.2352941 ± 0.040861
Th-230/U-234= 1.2283508 ± 0.044709
Th-230/Th-232= 5.3766816 ± 0.392054
U234/Th-232= 4.3771547 ± 0.315609

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

U(ppb)= 47450.894

Th(ppb)= 40885.83

Spike 25A Spike 25B Spide 25C
10/9/92
Th-228 (dpm/g)= 2646.0026 264.31598 26.417691

MCB # 1 ACQ 02-21-95 AT 11:24:52 RT : 91125.6 LT : 91120.1
No detector description was entered
NOPI-219 U 2/22/95

ROI # 13-1 RANGE : 83 to 149
AREA : Gross = 1805 Net = 1661 +/- 56
CENTROID : 126.88
SHAPE : Fwhm = 25.05 Channels Fwtm = 48.50 Channels

ROI # 13-2 RANGE : 325 to 396
AREA : Gross = 2266 Net = 2032 +/- 68
CENTROID : 374.33
SHAPE : Fwhm = 23.09 Channels Fwtm = 51.29 Channels

ROI # 13-3 RANGE : 561 to 637
AREA : Gross = 20868 Net = 19521 +/- 188
CENTROID : 610.97
SHAPE : Fwhm = 21.62 Channels Fwtm = 52.59 Channels

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-219 Th 2/23/95

ROI # 11-1 RANGE : 21 to 76
AREA : Gross = 251 Net = 223 +/- 21
CENTROID : 52.00
SHAPE : Fwhm = 1.83 Channels Fwtm = 32.69 Channels

ROI # 11-2 RANGE : 293 to 368
AREA : Gross = 1277 Net = 1201 +/- 46
CENTROID : 348.80
SHAPE : Fwhm = 21.37 Channels Fwtm = 58.42 Channels

ROI # 11-3 RANGE : 594 to 696
AREA : Gross = 6127 Net = 5838 +/- 103
CENTROID : 669.64
SHAPE : Fwhm = 21.61 Channels Fwtm = 69.95 Channels

ROI # 11-4 RANGE : 727 to 804
AREA : Gross = 1093 Net = 937 +/- 53
CENTROID : 782.18
SHAPE : Fwhm = 26.66 Channels Fwtm = 49.57 Channels

U and Th Isotope Activities

FILENAME= NP221U.CHN
NP221TH.CHN

Sample # **NOPI-221**
Analyst **JDP**

Sep. date **2/16/95**

Sample weight (g)	Spike weight (g)	U-232 (dpm/g)	Ref. date of spike	Days btwn ref. and sep.	U-232* (dpm/g)	Th-228/U-232=
0.5129	0.4953	454.83	1/22/93	755	445.86842	0.5928116

Counting time for Th=	833.375 (mins.)	Counting time for U=	1519.0617 (mins.)
Days btwn. sep. and count.=	6 (days)	Days btwn. sep. and count.=	5 (days)
CF for Th-228=	0.9937803	CF for U-232=	1.0053222

Th-232 counts	Th-230 counts	Th-228 counts	Ra-224 counts
190	2261	6018	1100

U-238 counts	U-234 counts	U-232 counts
4724	4568	23223

Bkgd	Bkgd	Bkgd	bkgd
5	8	14	26

bkgd	bkgd	bkgd
27	26	43

Th-232* counts	Th-230* counts	Th-228sp counts	Ra-224* counts
185	2253	5799.2988	1074

U-238* counts	U-234* counts	U-232sp counts
4697	4542	23057.284

U-238(dpm/g)=	87.711138 ± 1.399936
U-234(dpm/g)=	84.816689 ± 1.372812

Th-232(dpm/g)=	8.1424537 ± 0.599968
Th-230(dpm/g)=	99.161882 ± 2.446005

U-234/U-238=	0.9670002 ± 0.020066
Th-230/U-234=	1.1691317 ± 0.030063
Th-230/Th-232=	12.178378 ± 0.919886
U234/Th-232=	10.416601 ± 0.785836

Decay constant (m-1)	U-238 2.948E-16	Th-232 9.413E-17
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U(ppb)= 117572.71

Th(ppb)= 33323.993

	Spike 25A	Spike 25B	Spide 25C
	10/9/92		
Th-228 (dpm/g)=	2646.0026	264.31598	26.417691

MCB # 1 ACQ 02-21-95 AT 11:24:52 RT : 91143.7 LT : 91138.2
No detector description was entered
NOPI-221 U 2/22/95

ROI # 14-1 RANGE : 79 to 152
AREA : Gross = 4872 Net = 4724 +/- 80
CENTROID : 129.94
SHAPE : Fwhm = 22.73 Channels Fwtm = 48.17 Channels

ROI # 14-2 RANGE : 319 to 401
AREA : Gross = 4747 Net = 4568 +/- 83
CENTROID : 379.84
SHAPE : Fwhm = 20.92 Channels Fwtm = 49.79 Channels

ROI # 14-3 RANGE : 561 to 641
AREA : Gross = 23939 Net = 23223 +/- 179
CENTROID : 617.28
SHAPE : Fwhm = 17.64 Channels Fwtm = 51.45 Channels

NP22ITH.CHW

MCB # 1 ACQ 02-22-95 AT 13:02:50 RT : 50002.5 LT : 50000.0
No detector description was entered
NOPI-221 Th 2/23/95

ROI # 12-1 RANGE : 24 to 71
AREA : Gross = 276 Net = 190 +/- 28
CENTROID : 53.23
SHAPE : Fwhm = 4.13 Channels Fwtm = 26.23 Channels

ROI # 12-2 RANGE : 291 to 367
AREA : Gross = 2518 Net = 2261 +/- 73
CENTROID : 343.90
SHAPE : Fwhm = 23.60 Channels Fwtm = 57.92 Channels

ROI # 12-3 RANGE : 598 to 693
AREA : Gross = 6417 Net = 6018 +/- 110
CENTROID : 665.44
SHAPE : Fwhm = 22.29 Channels Fwtm = 70.15 Channels

ROI # 12-4 RANGE : 722 to 801
AREA : Gross = 1284 Net = 1100 +/- 58
CENTROID : 774.25
SHAPE : Fwhm = 13.86 Channels Fwtm = 54.77 Channels

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-294

Date 1/3/95

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 = OS-18

2. Record dry weight of sample = 3.414 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 25B

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 2.925 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH 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. Dissolve 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 # 49643A)

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 1/4/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/9/95

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 disappears 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 1/9/95

NP294U.CHN

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 50006.7 LT : 50000.0
No detector description was entered
NOPI-294 U 1/10/95

ROI # 9-1 RANGE : 46 = 398.79keV to 163 = 425.83keV
AREA : Gross = 5912 Net = 5796 +/- 89
CENTROID : 132.63 = 418.82keV
SHAPE : Fwhm = 7.35keV Fwtm = 12.98keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.116 +/- 0.00

ROI # 9-2 RANGE : 291 = 455.42keV to 411 = 483.15keV
AREA : Gross = 6977 Net = 6916 +/- 90
CENTROID : 379.54 = 475.88keV
SHAPE : Fwhm = 8.00keV Fwtm = 14.33keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.138 +/- 0.00

ROI # 9-3 RANGE : 526 = 509.73keV to 645 = 537.24keV
AREA : Gross = 8519 Net = 8398 +/- 103
CENTROID : 617.63 = 530.91keV
SHAPE : Fwhm = 8.67keV Fwtm = 13.23keV

ID : No close library match

NP2944.CHN

0:	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
10:	0	0	0	3	0	3	0	0
1	1	0	0	0	0	0	0	0
20:	1	1	0	0	1	0	0	0
0	4	2	0	0	0	0	0	0
30:	0	1	1	0	0	1	1	1
2	1	1	0	0	0	0	0	0
40:	0	1	1	0	0	1	1	1
0	1	3	0	0	0	0	0	0
50:	0	1	1	2	3	1	1	3
2	4	4	0	0	0	0	0	0
60:	5	5	1	2	3	6	8	8
4	3	5	0	0	0	0	0	0
70:	3	5	3	8	3	7	8	8
3	6	7	0	0	0	0	0	0
80:	3	7	13	11	5	12	9	9
10	21	18	0	0	0	0	0	0
90:	19	20	17	17	25	19	33	33
29	27	28	0	0	0	0	0	0
100:	35	54	55	41	56	48	66	66
61	62	66	0	0	0	0	0	0
110:	68	71	92	93	85	88	110	110
89	110	132	0	0	0	0	0	0
120:	107	124	105	109	147	126	150	150
137	147	150	0	0	0	0	0	0
130:	159	175	158	184	176	182	205	205
180	179	155	0	0	0	0	0	0
140:	156	140	138	89	80	68	71	71
61	31	20	0	0	0	0	0	0
150:	12	9	11	6	4	4	2	2
2	2	2	0	0	0	0	0	0
160:	1	1	0	1	1	2	4	4
1	0	1	0	0	0	0	0	0
170:	1	1	1	1	2	1	0	0
0	2	0	0	0	0	0	0	0
180:	2	0	2	1	0	0	2	2
1	1	2	0	0	0	0	0	0
190:	2	0	1	3	7	6	3	3
2	5	1	0	0	0	0	0	0
200:	2	6	1	6	7	4	5	5
7	6	4	0	0	0	0	0	0
210:	8	3	4	8	3	6	7	7
9	7	9	0	0	0	0	0	0
220:	3	7	5	11	2	9	9	9
8	8	3	0	0	0	0	0	0
230:	6	4	4	3	1	3	2	2
0	0	3	0	0	0	0	0	0
240:	1	1	0	1	2	0	0	0
1	1	2	0	0	0	0	0	0
250:	0	2	0	3	0	0	0	0
2	0	3	0	0	0	0	0	0
260:	1	1	0	2	1	0	0	0
3	1	0	0	0	0	0	0	0
270:	1	1	1	2	1	0	1	1
1	0	2	0	0	0	0	0	0
280:	1	0	0	1	1	2	3	3
2	1	2	0	0	0	0	0	0

5	4	2					
300:	5	5	5	5	6	5	6
3	3	9					
310:	6	5	3	9	7	8	5
2	8	7					
320:	4	3	11	4	7	10	8
7	11	11					
330:	13	15	16	15	12	17	20
16	22	24					
340:	36	26	35	30	39	37	43
51	56	39					
350:	51	63	60	66	66	90	87
87	105	88					
360:	100	95	120	92	118	147	125
140	129	141					
370:	130	151	133	130	149	169	138
148	193	202					
380:	172	193	182	198	201	182	196
193	179	155					
390:	155	150	102	105	89	70	40
39	37	20					
400:	17	4	3	2	2	3	3
1	1	1					
410:	0	1	0	0	2	1	0
1	0	0					
420:	0	1	0	1	1	0	0
0	0	0					
430:	2	1	0	1	0	0	0
1	0	0					
440:	1	0	0	0	0	1	0
1	1	0					
450:	1	1	0	0	0	1	0
0	0	1					
460:	0	0	1	1	0	0	0
0	0	2					
470:	0	1	0	1	0	1	0
1	0	1					
480:	0	0	0	1	0	0	0
0	1	0					
490:	1	0	0	0	1	1	1
0	0	0					
500:	0	1	3	0	0	0	0
2	1	0					
510:	1	0	3	4	0	3	2
0	0	0					
520:	0	1	3	3	4	1	2
0	2	1					
530:	4	1	1	4	0	3	1
3	3	2					
540:	3	3	4	1	2	6	6
6	2	10					

550:	9	3	4	7	8	8	7
7	8	8					
560:	4	9	7	9	8	11	13
20	16	12					
570:	19	18	27	16	23	42	31
23	42	33					
580:	51	48	52	59	62	57	69
74	95	83					
590:	96	106	147	124	139	122	141
128	168	153					
600:	133	128	135	128	164	137	157
174	169	154					
610:	180	179	185	187	189	228	232
270	263	228					
620:	237	277	240	219	236	218	169
162	158	113					
630:	119	61	67	42	29	16	17
5	3	4					
640:	1	6	1	0	1	2	1
3	1	4					
650:	1	2	3	3	2	2	8

660:	3	3	4	5	5	7	5
7	3	5					
670:	5	7	3	4	4	2	1
2	2	0					
680:	0	1	0	1	0	0	0
0	0	0					
690:	0	0	0	1	0	0	0
1	0	0					
700:	1	0	0	0	0	0	0
0	0	0					
710:	0	0	0	0	0	0	0
0	0	0					
720:	0	0	0	0	1	0	0
0	0	0					
730:	0	0	0	0	0	0	0
1	0	0					
740:	0	0	0	2	0	1	0
0	0	0					
750:	1	0	0	1	3	1	1
0	2	0					
760:	2	0	1	2	0	0	2
3	5	3					
770:	2	4	2	1	4	6	2
3	3	2					
780:	3	1	4	3	2	4	2
3	4	0					
790:	1	1	1	0	0	0	1
1	0	1					
800:	1	0	0	0	0	0	0
0	0	0					
810:	0	0	0	0	0	0	0
0	1	0					
820:	0	0	0	0	0	0	0
0	0	0					
830:	0	0	0	0	0	0	0
0	0	0					
840:	0	0	0	1	0	0	0
0	0	0					
850:	0	0	0	0	0	0	0
0	0	0					
860:	0	0	0	0	0	0	0
0	0	0					
870:	0	0	0	0	0	0	1
0	0	0					
880:	0	0	2	0	0	0	0
0	0	0					
890:	0	0	0	0	1	0	0
0	0	0					
900:	0	0	1	0	0	2	0
0	0	1					
910:	0	0	1	2	0	0	0
0	0	0					
920:	1	0	0	0	1	0	0
0	1	0					
930:	1	0	1	0	1	0	1
1	0	1					
940:	0	1	2	0	0	0	0
0	0	0					
950:	1	2	0	0	0	0	0
0	1	0					
960:	2	2	0	0	0	0	0
1	0	0					
970:	0	0	1	0	0	0	0
0	0	0					
980:	0	0	0	1	0	0	0

990:	0	0	0	0	1	1	0
0	0	0					
1000:	11	14	14	16	7	11	10
8	15	15					
1010:	8	8	15	9	12	8	16
15	12	14					
1020:	10	9	4	2			

NP294 Th. CHW

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 75008.3 LT : 75000.0
No detector description was entered
NOPI-294 Th 1/10/95

ROI # 10-1 RANGE : 12 = 397.10keV to 57 = 407.52keV
AREA : Gross = 187 Net = 86 +/- 28
CENTROID : 16.00 = 398.02keV
SHAPE : Fwhm = 0.35keV Fwtm = 0.94keV

ID : No close library match

ROI # 10-2 RANGE : 193 = 439.01keV to 353 = 476.07keV
AREA : Gross = 8342 Net = 8021 +/- 128
CENTROID : 321.22 = 468.71keV
SHAPE : Fwhm = 10.52keV Fwtm = 18.87keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.107 +/- 0.00

ROI # 10-3 RANGE : 522 = 515.21keV to 673 = 550.17keV
AREA : Gross = 5305 Net = 5077 +/- 103
CENTROID : 645.14 = 543.72keV
SHAPE : Fwhm = 7.38keV Fwtm = 18.11keV

ID : No close library match

ROI # 10-4 RANGE : 690 = 554.11keV to 788 = 576.81keV
AREA : Gross = 2378 Net = 2100 +/- 80
CENTROID : 762.19 = 570.83keV
SHAPE : Fwhm = 4.76keV Fwtm = 14.01keV

ID : No close library match

NP294Th.CHN

0:	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
10:	0	1	4	3	6	7	13	
7	6	3						
20:	6	3	5	2	6	3	5	
8	2	4						
30:	2	5	3	2	8	5	6	
1	7	9						
40:	4	5	3	5	2	3	6	
8	1	3						
50:	0	1	4	0	1	0	0	
0	0	0						
60:	1	0	1	0	1	0	0	
0	0	0						
70:	1	1	1	1	1	0	0	
0	0	0						
80:	1	0	1	1	1	1	0	
0	1	0						
90:	0	3	0	1	1	0	1	
0	1	2						
100:	1	0	1	0	2	1	0	
1	1	0						
110:	0	0	1	0	0	0	0	
0	1	0						
120:	1	0	0	1	0	0	0	
0	1	0						
130:	0	1	1	0	0	1	0	
1	0	0						
140:	0	0	1	2	0	1	1	
0	0	0						
150:	2	0	1	1	3	2	0	
2	0	1						
160:	3	1	0	0	1	1	0	
0	0	1						
170:	1	1	3	1	2	1	2	
1	1	4						
180:	1	2	0	1	0	1	4	
1	2	1						
190:	3	1	1	1	2	3	1	
1	3	4						
200:	5	3	4	6	5	4	0	
2	2	1						
210:	1	1	7	1	2	3	3	
7	2	1						
220:	3	7	2	1	4	2	5	
7	8	7						
230:	5	2	9	4	5	4	4	
3	7	2						
240:	4	3	8	8	12	8	11	
12	9	11						
250:	10	16	18	19	9	14	14	
12	14	25						
260:	17	13	11	15	36	21	39	
28	29	22						
270:	31	30	21	42	36	37	31	
49	45	51						
280:	42	52	52	58	55	45	58	

69	81	92					
290:	88	80	78	91	102	102	92
89	98	128					
300:	95	100	113	117	148	120	123
146	124	131					
310:	148	121	145	119	144	146	169
127	147	159					
320:	159	148	163	179	160	163	150
187	181	170					
330:	173	159	147	148	134	121	87
99	68	71					
340:	56	46	37	28	31	9	12
11	2	3					
350:	2	1	3	1	2	1	3
1	0	2					
360:	0	1	1	1	1	2	1
1	1	0					
370:	1	0	3	0	1	0	2
4	3	3					
380:	2	2	1	2	1	2	0
0	0	0					
390:	0	2	1	0	0	2	0
3	1	1					
400:	0	0	0	0	0	0	0
0	0	0					
410:	0	1	0	0	1	1	1
0	1	1					
420:	1	0	0	1	0	1	0
0	0	0					
430:	0	0	2	1	0	0	0
1	0	0					
440:	0	1	1	2	1	0	0
0	1	1					
450:	1	0	0	1	1	4	1
2	0	2					
460:	0	0	1	1	2	0	2
1	1	1					
470:	1	0	2	1	1	1	0
2	3	1					
480:	1	2	1	1	1	3	2
0	1	0					
490:	1	3	1	3	0	1	0
1	0	1					
500:	0	1	1	0	0	1	1
1	0	0					
510:	0	2	5	1	1	3	3
0	2	2					
520:	1	0	2	1	2	3	3
0	3	0					
530:	2	5	1	1	3	3	8
3	3	1					
540:	6	2	1	8	1	3	1
5	2	5					

550:	7	7	4	5	1	1	3
2	5	2					
560:	3	7	1	7	7	19	5
8	10	7					
570:	4	8	6	8	9	9	8
11	15	13					
580:	15	14	14	12	15	7	24
14	24	22					
590:	24	20	24	31	22	32	26
31	31	24					
600:	43	37	43	39	40	36	41
58	45	55					
610:	61	59	62	70	50	53	77
60	60	65					
620:	66	65	57	61	59	54	65
75	64	62					
630:	60	70	81	88	69	83	106
89	83	92					
640:	108	108	104	137	95	121	123
116	125	110					
650:	113	94	99	94	78	77	95
61	53	36					
660:	28	24	15	16	10	18	10
10	3	5					
670:	6	1	1	2	3	3	3
5	0	2					
680:	1	5	2	2	3	3	4
4	5	3					
690:	4	3	7	3	3	4	5
4	4	4					
700:	6	8	9	10	3	15	4
10	13	11					
710:	5	9	9	10	5	12	6
11	10	12					
720:	23	16	19	13	21	13	14
19	15	19					
730:	20	20	16	21	30	27	26
28	35	27					
740:	42	26	38	35	40	43	40
45	51	38					
750:	32	42	60	48	65	49	41
52	81	54					
760:	66	63	65	63	58	58	52
51	62	51					
770:	42	22	12	21	22	12	11

15	12	5						
780:	5	5	2	4	3	4	1	
1	1	2						
790:	3	4	4	3	7	2	1	
5	2	1						
800:	0	2	1	1	3	4	1	
1	1	0						
810:	3	2	1	1	0	1	2	
0	0	2						
820:	1	2	2	0	0	0	0	
2	2	0						
830:	1	1	2	0	0	0	0	
0	0	2						
840:	2	1	0	3	0	2	0	
1	0	0						
850:	2	0	0	1	3	1	1	
2	1	2						
860:	0	3	1	0	3	1	1	
5	2	3						
870:	2	4	1	4	2	4	6	
4	4	3						
880:	7	8	4	5	5	4	5	
10	4	5						
890:	5	4	6	6	8	4	3	
4	5	8						
900:	7	4	4	3	4	6	10	
8	7	8						
910:	7	8	9	4	10	10	7	
4	10	9						
920:	10	8	5	7	6	4	7	
8	3	7						
930:	3	7	4	4	5	1	4	
4	5	6						
940:	5	1	6	3	1	3	3	
4	3	2						
950:	1	6	3	1	1	3	1	
4	0	0						
960:	3	6	2	1	4	0	3	
2	5	3						
970:	3	4	4	3	4	3	4	
4	5	8						
980:	5	5	6	6	6	6	6	
4	9	3						
990:	11	5	8	7	6	4	12	
10	11	15						
1000:	64	87	91	79	82	85	91	
88	86	103						
1010:	87	82	102	88	83	83	68	
74	62	72						
1020:	70	84	78	13				

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-301

Date 1/3/95

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 = 05-18

2. Record dry weight of sample = .3205 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 204.88 pCi/g

Spike wt. .2975 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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. Dissolve 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 # 49643A)

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

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/ / / /

Separation Date 1/4/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/9/95

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 disappears 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 1/9/95

NP301U.CTN

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 50006.7 LT : 50000.0
No detector description was entered
NOPI-301 U 1/10/95

ROI # 11-1 RANGE : 37 = 396.17keV to 163 = 425.20keV
AREA : Gross = 9568 Net = 9167 +/- 131
CENTROID : 131.37 = 417.91keV
SHAPE : Fwhm = 9.32keV Fwtm = 17.06keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.183 +/- 0.00

ROI # 11-2 RANGE : 267 = 449.16keV to 414 = 483.02keV
AREA : Gross = 11204 Net = 11032 +/- 123
CENTROID : 381.52 = 475.54keV
SHAPE : Fwhm = 9.82keV Fwtm = 17.11keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.221 +/- 0.00

ROI # 11-3 RANGE : 522 = 507.90keV to 647 = 536.70keV
AREA : Gross = 3423 Net = 3297 +/- 76
CENTROID : 616.15 = 529.59keV
SHAPE : Fwhm = 9.88keV Fwtm = 17.61keV

ID : No close library match

14
5
3
1
119
18
10

163
37
1260
31
1260
3780
39060

4
9568
390
9178

NP3012.CTW

0:	0	0	0	0	0	0	0	0
0	0	1						
10:	0	0	2	3	0	4	5	
5	3	5						
20:	2	3	4	1	1	6	3	
2	3	4						
30:	3	6	6	9	4	5	1	
5	5	4						
40:	3	6	9	9	8	7	8	
4	10	5						
50:	11	8	11	4	16	9	12	
15	9	12						
60:	19	14	15	14	13	15	20	
26	20	31						
70:	20	29	22	30	22	25	19	
31	19	34						
80:	22	33	38	35	43	58	49	
51	47	46						
90:	45	60	50	43	59	46	66	
84	71	83						
100:	69	71	86	75	79	96	103	
94	85	115						
110:	108	108	143	133	142	133	131	
150	164	164						
120:	149	145	152	177	169	178	183	
204	195	180						
130:	207	238	195	216	189	202	211	
224	218	218						
140:	185	188	199	181	158	148	127	
104	112	104						
150:	79	63	50	38	24	16	10	
9	5	4						
160:	3	2	1	2	3	1	5	
1	2	1						
170:	2	2	2	0	1	2	3	
6	2	3						
180:	2	2	3	1	0	3	2	
3	5	9						
190:	2	2	3	6	5	2	4	
3	7	5						
200:	3	11	4	9	7	4	9	
5	4	2						
210:	9	7	8	10	6	7	6	
8	4	7						
220:	8	6	10	11	5	10	3	
6	7	2						
230:	4	6	9	9	5	7	2	
3	3	5						
240:	4	2	2	1	2	1	5	
4	1	1						
250:	1	2	4	4	1	4	3	
2	1	3						
260:	0	2	3	3	0	1	2	
1	3	1						
270:	2	4	2	4	8	2	8	
2	2	2						

200:	5	5	/	/	4	7	10
12	6	8					
290:	4	6	9	13	8	8	13
9	10	6					
300:	8	4	10	8	11	16	15
20	13	12					
310:	14	21	15	22	21	24	30
14	21	22					
320:	12	25	19	22	30	40	40
30	28	38					
330:	23	48	40	44	57	51	62
40	59	66					
340:	66	55	54	75	77	70	84
87	92	102					
350:	86	103	105	96	113	112	136
143	124	128					
360:	162	131	159	148	162	155	170
178	195	177					
370:	153	209	197	195	211	186	203
223	195	223					
380:	231	208	232	243	215	243	248
245	252	216					
390:	261	208	226	202	182	151	130
152	101	101					
400:	94	57	52	37	29	11	12
6	5	3					
410:	2	3	0	1	1	0	1
0	2	0					
420:	0	0	0	0	1	0	1
1	0	0					
430:	0	0	0	0	1	0	1
0	0	0					
440:	1	2	1	0	0	0	0
1	0	0					
450:	1	0	0	0	0	0	1
2	0	0					
460:	0	0	0	0	0	0	0
0	1	0					
470:	1	2	1	0	0	0	0
0	0	0					
480:	0	1	1	0	0	0	0
1	1	0					
490:	0	0	0	0	0	1	1
0	1	1					
500:	1	0	1	1	0	1	0
1	1	0					
510:	2	1	3	5	0	1	0
0	1	0					
520:	1	2	1	1	2	2	4
1	3	7					
530:	0	0	2	4	2	4	1
1	1	6					
540:	7	6	4	4	8	1	4
3	5	6					

550:	3	5	6	9	7	7	5
7	12	9					
560:	7	14	10	11	5	12	7
11	13	12					
570:	9	16	11	18	19	22	15
20	23	21					
580:	29	21	22	23	32	31	26
31	30	24					
590:	27	32	38	35	53	37	44
45	55	53					
600:	53	42	54	55	64	68	68
54	59	61					
610:	63	70	70	55	57	66	62
60	76	75					
620:	71	55	67	68	69	64	69
78	61	59					
630:	55	46	49	56	36	31	34
25	22	12					
640:	8	11	12	5	2	1	1
0	2	0					
650:	0	3	3	0	3	1	1
0	0	3					
660:	1	0	3	2	1	0	3
3	0	1					
670:	2	3	3	1	5	3	1
1	3	2					
680:	0	1	1	2	0	0	1
0	0	0					
690:	0	1	0	0	0	1	0
1	0	2					
700:	0	0	0	0	2	0	0
0	0	0					
710:	0	0	0	0	0	0	0
0	0	0					
720:	0	0	0	0	0	0	1
0	1	0					
730:	0	0	0	0	0	0	0
0	1	0					
740:	0	1	1	0	0	1	1
1	0	0					
750:	1	0	0	0	1	0	2
2	1	0					
760:	0	0	0	0	1	1	0
0	0	0					
770:	0	0	3	1	1	2	0
2	1	0					
780:	1	0	2	0	2	0	2
0	2	0					
790:	1	0	3	1	0	0	0
0	0	0					

800:	2	0	1	0		0	0
0	0	0					
810:	0	0	1	1	0	0	0
0	0	0					
820:	0	1	0	1	1	0	0
0	0	0					
830:	1	0	1	1	1	2	0
0	1	2					
840:	0	0	0	0	0	0	0
0	0	0					
850:	0	0	0	0	0	0	0
0	0	0					
860:	0	0	0	0	0	0	0
0	0	0					
870:	0	0	0	0	0	0	0
0	1	0					
880:	0	0	0	0	0	0	0
0	0	0					
890:	0	0	0	0	0	0	0
0	0	0					
900:	0	0	0	0	0	0	1
0	0	0					
910:	0	0	0	0	0	0	0
0	0	0					
920:	0	1	0	1	0	0	0
0	0	0					
930:	0	1	1	0	0	0	0
0	0	0					
940:	0	0	0	0	0	2	0
0	1	0					
950:	2	0	0	3	2	1	0
0	1	0					
960:	0	1	0	0	0	2	0
0	1	1					
970:	0	1	0	0	0	0	0
0	0	0					
980:	0	1	0	1	0	0	0
0	0	1					
990:	0	1	0	0	0	0	0
0	0	0					
1000:	8	14	12	10	14	12	11
14	17	13					
1010:	7	8	11	13	9	10	8
11	9	11					
1020:	13	15	8	5			

NP301Th.cHW

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 75008.3 LT : 75000.0
No detector description was entered
NOPI-301 Th 1/10/95

ROI # 12-1 RANGE : 11 = 390.03keV to 66 = 402.76keV
AREA : Gross = 314 Net = 90 +/- 44
CENTROID : 61.76 = 401.78keV
SHAPE : Fwhm = 0.41keV Fwtm = 1.14keV

ID : No close library match

ROI # 12-2 RANGE : 185 = 430.31keV to 377 = 474.77keV
AREA : Gross = 35742 Net = 34681 +/- 260
CENTROID : 333.82 = 464.77keV
SHAPE : Fwhm = 10.80keV Fwtm = 20.32keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.462 +/- 0.00

ROI # 12-3 RANGE : 568 = 518.99keV to 690 = 547.24keV
AREA : Gross = 5254 Net = 5009 +/- 99
CENTROID : 659.81 = 540.25keV
SHAPE : Fwhm = 7.10keV Fwtm = 18.25keV

ID : No close library match

ROI # 12-4 RANGE : 716 = 553.26keV to 815 = 576.18keV
AREA : Gross = 3057 Net = 2557 +/- 102
CENTROID : 774.72 = 566.85keV
SHAPE : Fwhm = 6.21keV Fwtm = 15.96keV

ID : No close library match

NP301TH.CHW

0:	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
10:	0	0	5	9	5	8	7	
1	11	6						
20:	3	5	4	7	4	3	6	
6	5	7						
30:	2	7	5	8	9	9	8	
4	8	10						
40:	4	6	8	6	5	5	6	
7	10	5						
50:	4	9	5	3	3	4	5	
3	5	5						
60:	4	8	10	2	5	2	3	
3	4	5						
70:	1	3	5	6	1	6	3	
0	2	4						
80:	2	2	4	3	2	5	3	
3	4	4						
90:	6	5	2	3	2	6	2	
6	5	2						
100:	4	1	7	3	2	5	2	
5	6	3						
110:	2	5	1	3	1	4	5	
4	2	4						
120:	5	1	5	4	4	6	4	
2	2	6						
130:	6	3	7	5	5	4	0	
5	3	2						
140:	8	3	4	2	3	8	4	
1	1	7						
150:	8	3	4	7	3	1	4	
10	4	10						
160:	5	5	3	6	7	8	7	
6	7	3						
170:	7	3	4	4	9	6	8	
3	3	9						
180:	6	7	5	10	2	5	3	
4	7	8						
190:	8	8	9	5	8	13	7	
9	10	10						
200:	6	9	7	9	9	12	6	
6	12	5						
210:	7	7	12	9	9	13	13	
13	12	8						
220:	10	14	8	16	17	17	8	
14	10	13						
230:	14	15	14	16	16	9	14	
24	12	17						
240:	18	14	18	20	26	16	20	
28	24	27						
250:	21	17	28	33	25	33	35	
39	37	40						
260:	33	45	48	46	48	37	45	
48	38	53						
270:	63	61	60	76	75	72	88	

280:	84	112	108	104	131	131	134
124	123	156					
290:	160	159	181	196	203	199	237
232	233	271					
300:	242	270	287	311	296	375	322
351	389	427					
310:	437	392	446	444	459	463	501
463	542	498					
320:	511	533	545	563	589	544	541
615	556	658					
330:	611	636	652	703	683	687	679
705	705	706					
340:	725	720	698	675	681	687	638
632	625	546					
350:	487	451	401	344	300	286	180
156	131	103					
360:	84	61	40	30	23	13	13
16	7	12					
370:	12	7	7	11	11	3	7
11	7	6					
380:	11	7	9	7	9	4	8
7	12	5					
390:	8	9	5	7	7	4	5
1	4	2					
400:	1	2	4	2	2	2	2
1	0	1					
410:	2	1	2	0	3	2	0
1	5	2					
420:	4	3	0	1	1	1	1
1	2	4					
430:	3	2	1	2	4	4	1
2	0	3					
440:	4	0	1	1	2	1	2
3	1	1					
450:	3	3	1	3	3	3	2
3	3	3					
460:	1	3	2	3	2	2	1
5	6	4					
470:	3	2	3	0	4	1	5
5	1	3					
480:	2	4	4	3	7	4	3
1	1	5					
490:	2	2	4	3	2	1	1
5	2	5					
500:	4	1	1	0	1	0	2
2	3	3					
510:	2	2	3	3	3	1	1
0	2	1					
520:	1	1	0	1	2	4	1
1	2	0					
530:	1	3	0	3	1	2	1
3	4	3					
540:	0	1	4	2	1	3	3
8	6	5					

550:	4	2	5	1	4	6	3
2	4	4					
560:	1	4	4	3	9	3	7
2	1	2					
570:	3	4	4	7	3	3	6
5	4	8					
580:	13	7	8	5	10	6	8
10	9	12					
590:	10	9	14	16	11	12	13
13	16	13					
600:	17	18	19	11	20	16	22
25	21	24					
610:	31	20	27	28	28	42	34
36	39	38					
620:	44	39	53	32	52	44	63
44	54	50					
630:	51	67	48	62	64	55	62
53	59	62					
640:	58	65	70	62	53	67	76
92	77	67					
650:	78	101	98	82	94	90	126
111	106	116					
660:	105	104	123	121	99	128	109
88	98	96					
670:	78	87	64	79	68	56	50
32	30	26					
680:	17	15	13	12	10	6	2
5	2	2					
690:	1	2	12	5	3	2	1
5	3	4					
700:	6	3	4	7	2	7	5
6	5	8					
710:	4	4	5	7	7	3	7
2	7	8					
720:	5	9	9	12	10	6	8
11	9	14					
730:	13	14	7	9	14	15	13
20	17	22					
740:	18	27	15	17	25	26	31
29	32	30					
750:	24	27	30	28	37	33	31
41	41	39					
760:	39	36	60	66	54	61	58
53	61	66					
770:	66	63	78	68	61	76	62
78	69	55					
780:	80	63	71	67	59	56	59
55	36	35					
790:	43	39	27	22	34	17	15
21	13	15					
800:	13	17	16	6	10	5	10
11	14	12					
810:	11	8	11	3	6	5	6
8	13	7					
820:	8	8	10	2	6	3	2
3	6	7					
830:	5	3	7	2	1	0	4
4	0	4					

850:	1	3	3	-	-	-	-	-
860:	1	3	3	2	4	7	2	4
870:	0	1	5	4	0	5	4	6
880:	6	5	4	5	3	8	4	1
890:	9	7	10	5	7	6	10	7
900:	9	4	9	5	8	13	13	9
910:	15	10	16	13	14	13	9	16
920:	15	16	14	9	13	9	16	12
930:	20	19	17	22	11	13	15	15
940:	8	12	11	16	13	16	12	11
950:	2	12	14	9	7	8	9	9
960:	2	16	14	2	4	4	2	3
970:	4	5	2	4	3	3	5	3
980:	2	1	2	0	2	2	1	1
990:	4	6	1	0	1	5	1	5
1000:	114	2	6	9	4	2	3	3
1010:	108	3	1	9	4	2	3	3
1020:	101	5	9	101	100	109	114	131
		94	101	101	100	109	114	131
		126	92					
		110	110	100	109	120	97	87
		101	105					
		101	101	95	24			

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-417

Date 1/3/95

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 = 05-18

2. Record dry weight of sample = .3068 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25 B

Reference Date 1/22/93

Reference Activity ^{232U} 204.88 pCi/g

Spike wt. .2972 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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 1/4/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/10/95

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 disappears 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 1/10⁹/95

NP417U.CHW

MCB # 1 ACQ 01-08-95 AT 22:08:56 RT : 50006.7 LT : 50000.0
No detector description was entered
NOPI-417 U 1/10/95

ROI # 1-1 RANGE : 36 = 3.88keV to 161 = 4.19keV
AREA : Gross = 9169 Net = 8917 +/- 118
CENTROID : 140.27 = 4.14keV
SHAPE : Fwhm = 0.08keV Fwtm = 0.16keV

ID : No close library match

ROI # 1-2 RANGE : 266 = 4.45keV to 388 = 4.75keV
AREA : Gross = 11756 Net = 11325 +/- 140
CENTROID : 366.76 = 4.70keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.17keV

ID : No close library match

ROI # 1-3 RANGE : 531 = 5.11keV to 605 = 5.29keV
AREA : Gross = 1547 Net = 1383 +/- 57
CENTROID : 587.88 = 5.25keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.12keV

ID : No close library match

NP417u.CHW

0:	0	0	0	0	0	0	0
0	0	0					
10:	0	0	1	1	1	3	2
6	3	7					
20:	6	3	1	2	8	5	2
3	2	0					
30:	2	3	8	1	2	2	4
1	4	7					
40:	2	2	6	3	7	7	6
7	8	12					
50:	7	1	5	8	8	9	7
6	10	10					
60:	10	7	13	15	12	7	12
10	8	13					
70:	17	17	16	12	10	8	12
17	23	22					
80:	18	21	17	20	25	16	22
25	32	19					
90:	33	30	33	35	42	38	53
49	52	46					
100:	45	51	56	42	60	40	59
70	59	65					
110:	73	82	86	85	77	96	103
113	128	128					
120:	151	123	139	144	156	188	155
157	175	202					
130:	196	196	226	205	207	188	227
205	214	228					
140:	218	222	244	242	264	237	277
257	231	206					
150:	181	139	94	56	39	13	4
2	6	1					
160:	1	1	1	1	1	2	1
2	2	3					
170:	0	2	1	2	1	2	3
6	2	0					
180:	3	3	1	5	6	2	3
6	0	0					
190:	6	2	2	2	5	5	9
4	2	3					
200:	4	6	5	10	5	7	18
6	10	6					
210:	9	10	6	11	14	13	10
9	11	5					
220:	14	9	11	10	10	15	6
10	10	3					
230:	8	4	4	5	3	6	3
2	3	4					
240:	1	8	3	3	6	1	6
5	5	5					
250:	4	3	4	1	4	3	10
3	7	3					
260:	3	10	4	5	5	5	3
4	13	10					
270:	5	7	5	10	9	9	11
10	8	12					
280:	5	16	7	16	11	6	12
20	12	9					

26	18	20						
300:	28	25	30	24	31	30	30	30
30	20	26						
310:	34	32	24	34	45	32	34	34
54	44	44						
320:	47	37	50	38	64	51	40	40
67	58	71						
330:	84	89	78	82	98	101	112	112
103	117	122						
340:	103	157	138	117	147	152	152	152
194	197	186						
350:	190	202	216	215	231	242	208	208
245	223	218						
360:	207	218	250	246	244	250	269	269
292	280	298						
370:	319	318	319	311	272	274	260	260
247	207	137						
380:	95	68	36	12	2	3	1	1
0	0	1						
390:	0	0	0	0	0	0	0	0
0	0	0						
400:	0	0	0	0	0	0	0	0
0	1	0						
410:	0	1	1	0	0	0	0	0
1	0	0						
420:	0	0	0	0	1	0	0	0
0	0	0						
430:	0	0	0	0	0	0	0	0
0	0	1						
440:	0	0	0	0	0	0	0	0
0	0	0						
450:	0	0	0	0	0	0	0	0
0	0	0						
460:	0	0	0	0	0	1	0	0
0	1	2						
470:	0	0	0	1	0	1	2	2
0	1	0						
480:	0	1	1	0	3	0	1	1
0	0	1						
490:	1	0	0	0	0	0	1	1
0	2	2						
500:	1	0	1	3	2	1	2	2
1	0	1						
510:	1	3	1	3	0	1	2	2
3	3	5						
520:	2	6	3	3	4	3	1	1
3	2	7						
530:	4	1	5	6	4	7	3	3
6	6	5						
540:	5	7	10	5	3	11	2	2
11	13	6						

550:	9	10	17	13	16	16	24
18	17	29					
560:	26	23	27	19	27	24	25
16	34	30					
570:	22	40	25	29	28	31	28
20	35	29					
580:	35	29	32	41	44	37	41
58	47	56					
590:	57	40	32	44	37	22	27
17	12	5					
600:	4	4	2	1	0	0	2
2	2	0					
610:	1	1	3	0	1	2	4
2	1	1					
620:	2	0	1	1	1	1	1
1	1	1					
630:	1	2	2	2	1	0	1
1	0	1					
640:	0	0	1	0	0	1	0
0	0	0					
650:	1	0	0	0	0	0	0
0	0	1					
660:	0	0	0	1	0	0	1
0	0	0					
670:	0	0	0	0	0	0	0
0	0	0					
680:	0	1	0	0	0	0	1
0	0	0					
690:	0	1	2	0	0	1	0
0	1	0					
700:	1	1	1	0	0	0	1
0	1	1					
710:	1	4	1	0	0	0	0
0	1	1					
720:	3	1	2	2	1	1	2
2	0	0					
730:	2	1	2	0	0	3	3
0	1	4					
740:	2	0	1	0	0	0	0
2	1	0					
750:	0	0	0	1	0	0	0
0	0	0					
760:	1	0	0	0	0	0	0
0	0	0					
770:	0	1	1	0	1	1	0
0	0	0					
780:	0	0	0	0	0	0	0
0	0	0					
790:	0	0	0	0	0	0	1
0	0	0					
800:	0	0	0	0	0	0	0
0	0	0					

820:	0	0	0	0	0	0	0
830:	0	0	0	0	0	0	0
840:	0	0	0	0	0	0	0
850:	0	0	0	0	0	0	0
860:	1	0	0	0	0	0	0
870:	0	0	0	0	0	1	1
880:	0	0	2	0	0	0	0
890:	0	0	0	0	0	0	2
900:	0	0	0	0	0	0	0
910:	1	0	0	0	0	0	0
920:	0	0	0	0	0	0	0
930:	0	1	0	0	0	0	0
940:	2	0	0	0	0	0	0
950:	0	1	0	1	2	3	0
960:	1	1	0	0	0	0	2
970:	1	1	1	1	0	1	0
980:	1	3	3	0	3	0	2
990:	0	1	1	0	0	0	0
1000:	0	3	7	2	2	2	1
1010:	4	1	3	1	0	4	1
1020:	3	1	3	1			

WP417 Th.CH N

MCB # 1 ACQ 01-09-95 AT 21:34:22 RT : 25902.0 LT : 25900.9
No detector description was entered
NOPI-417 Th 1/10/95

ROI # 11-1 RANGE : 14 = 390.87keV to 66 = 402.85keV
AREA : Gross = 93 Net = 28 +/- 23
Could not properly fit the peak.

ROI # 11-2 RANGE : 213 = 436.72keV to 375 = 474.04keV
AREA : Gross = 43146 Net = 42821 +/- 227
CENTROID : 339.52 = 465.86keV
SHAPE : Fwhm = 10.39keV Fwtm = 17.79keV

ID : SB-125 at 463.38keV
Corrected Rate = 1.653 +/- 0.01

ROI # 11-3 RANGE : 591 = 523.80keV to 690 = 546.61keV
AREA : Gross = 2749 Net = 2548 +/- 75
CENTROID : 666.27 = 541.14keV
SHAPE : Fwhm = 4.45keV Fwtm = 16.02keV

ID : No close library match

ROI # 11-4 RANGE : 723 = 554.21keV to 834 = 579.78keV
AREA : Gross = 2100 Net = 1932 +/- 70
CENTROID : 780.20 = 567.39keV
SHAPE : Fwhm = 7.04keV Fwtm = 19.99keV

ID : No close library match

NP417 Th. CHW

0:	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
10:	0	0	0	1	2	1	3	
4	0	1						
20:	3	0	2	1	0	1	2	
1	2	1						
30:	1	2	1	1	6	2	4	
2	2	1						
40:	2	2	4	1	2	1	1	
1	1	2						
50:	2	3	2	3	1	4	1	
2	0	2						
60:	2	3	2	2	0	1	0	
1	1	1						
70:	0	1	1	3	0	0	0	
1	0	1						
80:	1	0	1	2	0	1	0	
0	2	0						
90:	3	0	0	0	0	1	0	
2	0	0						
100:	1	0	0	0	0	0	1	
4	0	2						
110:	0	1	2	0	1	1	1	
1	1	1						
120:	3	1	1	0	0	2	1	
1	3	0						
130:	1	1	1	2	1	0	1	
2	2	0						
140:	2	1	2	1	2	0	0	
1	3	1						
150:	2	1	0	0	1	1	2	
0	3	1						
160:	3	0	2	0	1	1	1	
1	2	1						
170:	1	2	0	1	1	0	2	
1	2	2						
180:	1	3	4	0	1	1	2	
3	2	1						
190:	0	1	2	0	1	0	1	
4	2	2						
200:	5	3	3	1	3	1	3	
1	2	5						
210:	3	3	2	2	1	6	7	
2	1	3						
220:	4	6	6	4	5	7	5	
7	2	5						
230:	9	6	4	5	9	6	5	
8	7	10						
240:	8	14	9	8	8	14	11	
6	9	10						
250:	9	15	14	11	10	18	13	
16	19	28						
260:	16	23	25	26	25	28	32	
34	46	29						
270:	37	42	39	39	42	48	55	

280:	62	81	64	73	76	75	82
96	102	133					
290:	99	113	150	157	161	185	176
178	217	211					
300:	224	236	261	258	298	324	344
303	344	374					
310:	390	430	414	493	487	468	563
513	553	546					
320:	583	599	580	634	637	650	699
603	698	754					
330:	736	691	783	709	784	785	840
871	903	942					
340:	935	922	934	992	978	990	997
997	954	912					
350:	911	892	832	766	707	623	563
516	401	352					
360:	285	206	163	133	87	42	44
26	20	13					
370:	6	4	3	2	1	0	2
2	1	2					
380:	3	2	1	1	0	1	3
1	3	0					
390:	1	1	0	2	3	1	0
0	0	0					
400:	3	2	0	0	1	1	0
1	0	0					
410:	0	0	0	1	0	0	0
0	0	0					
420:	0	1	0	1	0	0	0
0	0	0					
430:	0	1	0	1	0	1	0
1	1	0					
440:	1	0	1	0	1	2	0
0	0	3					
450:	2	0	0	0	0	1	0
0	0	1					
460:	3	0	0	1	1	1	1
0	0	1					
470:	1	2	2	2	0	0	2
0	4	0					
480:	1	3	1	1	2	0	0
2	3	2					
490:	3	0	2	4	1	1	0
0	1	1					
500:	0	0	1	2	0	3	1
3	0	0					
510:	1	0	0	1	1	0	1
1	1	0					
520:	1	0	0	0	0	0	0
0	0	0					
530:	1	0	0	0	0	0	1
0	1	0					
540:	0	0	0	0	1	1	0
2	0	0					

550:	1	2	0	0	2	1	0
1	0	1					
560:	1	0	2	1	1	1	1
0	3	4					
570:	3	1	0	0	0	0	3
1	1	2					
580:	3	2	1	0	2	3	1
3	2	2					
590:	3	3	0	3	4	3	5
6	3	4					
600:	8	8	4	3	11	7	8
6	8	9					
610:	13	10	16	9	6	19	20
18	13	16					
620:	22	21	25	21	19	24	24
22	23	32					
630:	27	30	36	32	33	20	33
27	27	30					
640:	31	26	23	37	28	28	28
29	29	29					
650:	39	24	46	43	46	53	58
36	48	51					
660:	66	60	63	66	64	61	77
68	65	82					
670:	69	57	62	44	50	44	40
31	34	31					
680:	25	19	14	12	8	10	13
4	2	3					
690:	2	4	3	0	1	0	2
1	1	4					
700:	3	0	5	2	5	3	3
2	1	3					
710:	1	3	3	1	6	1	2
5	3	3					
720:	5	3	2	2	2	1	7
7	10	0					
730:	6	6	9	4	9	14	12
4	13	8					
740:	14	12	11	10	10	13	12
10	18	23					
750:	25	12	23	17	20	14	23
22	17	20					
760:	22	22	19	28	30	21	17
29	32	40					
770:	46	25	49	38	39	51	45
39	33	41					
780:	34	36	41	44	42	35	50
34	34	48					
790:	27	27	29	34	24	25	22
21	23	19					
800:	18	10	18	18	16	19	15
11	10	12					
810:	15	16	10	13	15	9	10
7	12	11					
820:	11	7	9	9	5	4	5
9	4	5					
830:	3	5	1	2	1	3	3
3	1	1					

850:	4	2	4	1	1	2	1	0
860:	0	2	2	2	2	5	4	2
870:	2	1	2	1	4	4	8	4
880:	6	6	3	5	3	4	3	8
890:	2	3	8	7	8	9	10	3
900:	15	12	9	14	6	23	16	15
910:	18	11	11	16	15	19	15	22
920:	15	13	20	18	9	10	15	14
930:	19	17	19	15	12	16	20	13
940:	15	15	13	12	9	8	11	10
950:	7	5	2	1	2	3	1	4
960:	2	4	3	3	3	0	1	1
970:	1	2	4	2	2	1	0	1
980:	2	0	1	2	0	2	3	2
990:	1	2	2	0	4	1	1	1
1000:	2	1	4	0	4	1	1	1
1010:	69	58	66	77	87	65	64	71
1020:	50	71	73	62	61	60	64	85
		76	59					
		67	74	54	16			

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-418

Date 1/3/95

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 = OS-18.

2. Record dry weight of sample = .4288 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 258

Reference Date 1/22/93

Reference Activity ^{232}U 20488 pCi/g

Spike wt. .4930 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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:

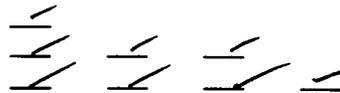
7. Add 1 ml of perchloric acid (HClO_4) to the sample solution in the teflon beaker. Evaporate to fumes of HClO_4 . Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).
8. Transfer solution to a 50 ml PP centrifuge tube. Add ~10 mg Fe carrier and coprecipitate 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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 1/4/95

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

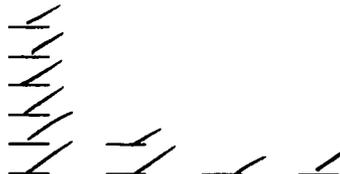
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/9/95

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 disappears 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 1/9/95

NP418 U.CTW

MCB # 1 ACQ 01-08-95 AT 22:08:56 RT : 50006.7 LT : 50000.0
No detector description was entered
NOPI-418 U 1/10/95

ROI # 5-1 RANGE : 39 = 3.85keV to 167 = 4.19keV
AREA : Gross = 8767 Net = 8590 +/- 111
CENTROID : 141.44 = 4.12keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.19keV

ID : No close library match

ROI # 5-2 RANGE : 252 = 4.42keV to 385 = 4.78keV
AREA : Gross = 11054 Net = 10901 +/- 119
CENTROID : 353.70 = 4.69keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.20keV

ID : No close library match

ROI # 5-3 RANGE : 491 = 5.06keV to 588 = 5.32keV
AREA : Gross = 1654 Net = 1556 +/- 55
CENTROID : 556.20 = 5.24keV
SHAPE : Fwhm = 0.09keV Fwtm = 0.18keV

ID : No close library match

NP418U.CHN

0:	0	0	0	0	0	0	0
0	0	0					
10:	0	0	0	0	1	1	1
0	4	1					
20:	2	1	1	3	2	2	0
0	1	0					
30:	1	0	0	1	2	1	1
2	1	0					
40:	1	5	2	5	2	1	1
3	5	4					
50:	5	2	7	2	2	2	9
8	0	6					
60:	2	9	4	13	6	8	6
10	13	10					
70:	6	10	12	14	15	10	18
17	13	21					
80:	18	10	15	12	9	16	15
19	31	21					
90:	22	36	32	29	29	30	39
42	34	45					
100:	36	51	38	48	37	57	44
54	70	59					
110:	70	75	73	71	88	92	103
85	96	122					
120:	108	115	111	134	142	144	156
167	165	159					
130:	157	170	164	195	182	167	161
211	209	191					
140:	211	221	209	192	227	226	239
253	213	207					
150:	206	205	169	163	114	110	62
61	40	26					
160:	19	9	7	5	1	0	2
0	5	3					
170:	1	1	1	2	0	2	4
1	2	0					
180:	1	2	3	2	3	3	7
0	3	1					
190:	6	7	1	4	8	7	2
6	7	1					
200:	4	9	8	4	7	6	6
6	12	8					
210:	5	5	6	9	12	13	6
8	17	11					
220:	13	14	9	9	11	8	7
6	6	9					
230:	7	2	1	2	2	4	2
3	0	0					
240:	2	1	1	2	2	2	1
1	3	1					
250:	3	3	1	2	4	1	3
5	4	5					
260:	3	5	3	4	6	2	7
6	6	8					
270:	6	7	8	5	6	7	5
6	7	12					
280:	10	17	15	14	12	17	18

1 /	20	10					
290:	24	29	11	25	23	23	28
31	24	34					
300:	26	35	26	29	42	37	42
47	45	60					
310:	47	51	52	55	71	53	61
66	56	85					
320:	92	88	87	105	94	82	101
108	138	152					
330:	124	132	139	115	153	157	165
168	173	176					
340:	163	184	195	198	202	207	212
226	222	220					
350:	199	226	210	238	240	243	230
224	273	252					
360:	275	241	226	268	241	225	222
192	157	151					
370:	109	100	75	49	30	20	10
8	4	3					
380:	0	1	2	0	0	0	1
0	0	0					
390:	0	0	0	0	0	0	0
0	0	0					
400:	0	0	0	0	0	0	0
0	0	0					
410:	0	0	0	0	0	0	0
0	0	0					
420:	0	0	0	0	0	0	0
1	0	0					
430:	0	0	0	0	1	2	0
0	0	1					
440:	0	0	0	1	1	0	0
0	0	1					
450:	0	0	0	2	0	0	0
0	0	0					
460:	0	1	1	0	0	1	0
2	0	3					
470:	2	0	1	1	0	0	1
2	1	0					
480:	3	0	0	1	0	2	1
1	0	1					
490:	1	1	0	1	2	2	0
2	3	3					
500:	0	3	2	2	6	5	1
1	3	8					
510:	2	6	2	4	8	6	10
6	6	12					
520:	9	10	15	6	13	8	12
11	15	12					
530:	11	15	16	9	17	14	22
23	20	30					
540:	16	25	22	31	32	34	43
35	21	36					

550:	37	31	35	25	38	38	31
35	49	25					
560:	43	32	47	46	42	39	47
38	21	43					
570:	38	42	36	18	24	10	16
9	6	4					
580:	3	4	4	0	2	2	3
1	0	1					
590:	1	0	3	0	0	0	2
2	1	2					
600:	4	3	6	0	0	5	3
2	3	1					
610:	4	3	4	1	3	3	1
1	2	1					
620:	0	1	1	0	0	0	0
0	0	1					
630:	0	1	0	0	0	0	0
0	0	0					
640:	0	0	0	0	0	0	1
0	0	0					
650:	0	0	1	0	0	0	0
0	1	1					
660:	0	0	0	0	0	0	0
0	0	0					
670:	0	0	1	0	0	3	2
0	0	1					
680:	0	0	1	0	0	0	1
0	1	1					
690:	0	0	1	2	2	0	2
1	2	1					
700:	2	2	3	0	3	2	1
1	4	3					
710:	1	2	1	1	1	1	0
0	0	1					
720:	1	1	1	0	0	0	0
1	0	1					
730:	1	0	0	0	0	0	1
0	0	0					
740:	0	0	0	0	0	0	1
0	0	1					
750:	0	0	0	0	0	0	0
0	0	0					
760:	0	0	0	1	0	0	0
0	0	0					
770:	0	0	0	0	0	0	0
0	0	1					
780:	0	0	0	0	0	0	0
0	0	0					
790:	1	0	0	0	1	0	0
0	0	0					
800:	0	0	0	0	0	0	0

0	0	0						
810:	0	0	2	0	0	0	0	0
1	0	0						
820:	0	0	0	0	0	0	0	0
0	1	0						
830:	0	1	2	0	0	1	0	0
0	0	1						
840:	0	0	1	0	0	0	0	1
0	0	0						
850:	0	1	0	1	0	1	0	0
0	0	0						
860:	0	0	0	0	0	0	0	0
0	0	0						
870:	0	0	0	0	0	0	0	0
0	0	1						
880:	0	1	0	0	0	0	0	0
0	0	0						
890:	0	2	0	0	0	0	0	0
0	0	0						
900:	1	0	0	1	0	0	0	1
1	0	0						
910:	0	2	0	1	0	0	0	1
1	0	1						
920:	0	2	0	0	0	1	0	1
1	3	1						
930:	0	1	3	4	3	3	0	2
1	0	4						
940:	2	1	0	0	0	0	0	0
0	0	0						
950:	1	0	0	0	1	0	0	0
0	0	0						
960:	0	0	0	0	0	0	0	0
0	0	0						
970:	0	0	0	0	0	0	0	0
0	0	0						
980:	0	0	0	0	0	0	0	0
0	0	0						
990:	0	0	0	0	0	0	0	0
0	0	0						
1000:	3	2	0	1	2	1	0	2
8	3	3						
1010:	3	7	2	8	2	4	0	1
3	5	3						
1020:	1	3	1	1				

NP418 Th.CTW

MCB # 1 ACQ 01-08-95 AT 22:08:56 RT : 60007.6 LT : 60000.0
No detector description was entered
NOPI-418 Th 1/10/95

ROI # 6-1 RANGE : 11 = 3.86keV to 66 = 4.01keV
AREA : Gross = 182 Net = 126 +/- 24
CENTROID : 57.00 = 3.98keV
SHAPE : Fwhm = 0.00keV Fwtm = 0.01keV

ID : No close library match

ROI # 6-2 RANGE : 138 = 4.20keV to 360 = 4.78keV
AREA : Gross = 98401 Net = 97621 +/- 355
CENTROID : 309.02 = 4.65keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.22keV

ID : No close library match

ROI # 6-3 RANGE : 484 = 5.11keV to 618 = 5.46keV
AREA : Gross = 7814 Net = 7166 +/- 145
CENTROID : 594.18 = 5.40keV
SHAPE : Fwhm = 0.12keV Fwtm = 0.20keV

ID : No close library match

ROI # 6-4 RANGE : 621 = 5.47keV to 740 = 5.79keV
AREA : Gross = 5019 Net = 4119 +/- 146
CENTROID : 693.03 = 5.66keV
SHAPE : Fwhm = 0.10keV Fwtm = 0.26keV

ID : No close library match

0:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
10:	1	0	1	0	4	2	6
5	2	3					
20:	1	5	1	4	2	0	2
4	3	1					
30:	2	2	3	2	5	4	2
3	2	3					
40:	2	3	1	2	7	6	2
3	3	3					
50:	3	6	8	5	4	1	4
9	4	9					
60:	10	2	3	3	0	2	3
6	2	3					
70:	5	3	4	1	4	11	1
1	0	2					
80:	1	2	2	2	4	3	2
1	3	4					
90:	1	2	2	2	4	4	3
2	2	3					
100:	2	1	3	7	3	2	3
1	2	4					
110:	2	6	2	3	8	2	2
5	1	6					
120:	3	3	3	5	5	5	6
5	3	6					
130:	7	3	7	2	7	5	5
4	4	3					
140:	4	9	8	4	4	1	4
6	8	2					
150:	7	8	7	3	2	5	7
9	4	5					
160:	10	5	4	4	8	12	8
9	7	9					
170:	6	12	12	8	20	12	14
13	16	13					
180:	17	20	19	18	17	10	12
13	16	27					
190:	18	20	21	19	19	24	28
31	28	33					
200:	25	29	29	38	27	28	25
40	40	36					
210:	39	29	42	45	50	50	40
60	50	54					
220:	48	70	57	61	79	76	85
72	74	88					
230:	83	106	102	114	106	112	126
114	124	139					
240:	124	152	161	162	162	167	161
171	180	208					
250:	240	236	205	272	258	292	264
323	327	330					
260:	304	370	421	411	439	453	486
518	540	562					
270:	605	622	666	692	697	802	763
782	863	843					
280:	891	982	960	1015	1111	1089	1178
1297	1279	1362					

1653	1660	1632					
300:	1661	1601	1646	1742	1678	1749	1722
1804	1784	1877					
310:	1840	1925	1985	1931	1962	1972	2103
1996	2040	2129					
320:	1933	1938	1787	1654	1541	1363	1178
1007	787	595					
330:	403	282	203	123	52	38	28
12	15	14					
340:	12	4	4	14	12	9	8
8	12	9					
350:	7	11	11	8	8	4	9
9	5	4					
360:	1	7	6	5	5	6	3
0	2	0					
370:	2	1	0	1	1	0	0
2	0	1					
380:	1	2	0	0	0	1	1
0	0	0					
390:	0	0	0	0	2	2	0
0	1	1					
400:	0	0	0	1	2	0	1
1	2	3					
410:	2	2	2	5	1	2	0
2	1	3					
420:	0	1	0	2	6	2	0
0	0	2					
430:	1	4	1	1	0	2	1
0	2	3					
440:	3	1	1	2	5	3	1
3	0	0					
450:	2	1	4	2	2	0	3
2	1	2					
460:	1	1	4	3	1	1	3
0	1	3					
470:	1	2	2	4	1	0	1
5	3	1					
480:	1	1	3	1	3	2	4
4	4	4					
490:	6	5	5	3	2	5	7
5	10	5					
500:	5	6	2	5	9	8	7
12	4	6					
510:	5	9	12	10	9	13	12
8	13	11					
520:	11	15	8	13	13	17	13
16	22	13					
530:	12	18	22	27	30	14	23
20	42	27					
540:	34	29	39	35	42	35	38
50	28	39					

550:	53	54	49	64	73	50	71
61	60	71					
560:	72	67	82	88	87	92	99
124	93	116					
570:	108	120	118	107	105	107	89
98	112	105					
580:	96	95	125	120	125	141	131
133	128	152					
590:	145	159	131	161	172	164	188
178	177	144					
600:	169	169	177	143	133	106	100
81	65	59					
610:	45	29	27	16	17	13	7
7	6	7					
620:	9	11	7	9	16	13	10
10	7	19					
630:	19	22	8	13	15	12	17
17	21	20					
640:	18	18	24	18	23	23	23
19	23	19					
650:	26	24	26	33	43	33	42
33	45	38					
660:	41	46	39	40	46	41	43
47	50	61					
670:	53	64	53	63	58	51	65
66	71	73					
680:	57	73	73	76	86	75	76
72	86	108					
690:	79	93	66	94	94	82	79
101	82	86					
700:	85	80	90	79	70	66	73
44	46	48					
710:	42	51	48	37	44	37	36
35	25	29					
720:	23	40	30	33	22	22	30
21	33	23					
730:	24	16	21	21	13	19	8
11	5	8					
740:	5	9	5	9	7	4	5
5	10	4					
750:	5	5	4	7	6	5	2
4	7	10					
760:	6	13	9	5	3	10	12
16	7	10					
770:	11	11	7	13	8	13	21
12	12	14					
780:	14	13	16	14	8	16	18
16	18	27					
790:	26	23	20	14	20	17	31
26	36	34					
800:	31	31	34	38	36	47	36
31	37	35					
810:	37	29	43	45	37	34	55
30	30	38					
820:	41	36	37	35	36	26	30
38	31	41					
830:	48	36	26	33	27	27	24
29	23	17					
840:	24	19	14	14	15	14	7
4	1	9					
850:	2	7	9	4	6	1	2

860:	3	6	3	6	5	4	6
5	1	5					
870:	6	7	5	9	3	3	8
12	5	5					
880:	7	6	7	9	4	9	10
8	12	6					
890:	15	6	12	10	15	11	12
15	9	13					
900:	15	12	15	14	17	29	20
17	18	13					
910:	22	17	14	21	19	23	19
21	20	27					
920:	27	35	28	22	19	27	20
9	16	18					
930:	15	18	12	24	15	18	18
14	15	3					
940:	6	6	2	2	1	2	0
0	0	1					
950:	0	0	1	0	2	0	2
2	1	0					
960:	0	0	1	1	0	1	1
0	1	0					
970:	1	2	0	0	1	0	0
1	0	0					
980:	0	0	1	3	0	1	1
1	1	0					
990:	1	1	0	2	3	1	1
0	2	3					
1000:	69	94	70	106	84	97	92
74	74	89					
1010:	93	89	91	78	81	88	83
84	76	93					
1020:	84	83	84	20			

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-419

Date 1/3/95

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 = 05-18.

2. Record dry weight of sample = .2608 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 204.88 pCi/g

Spike wt. .2984 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/9/95

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 disappears 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 1/9/95

NP49U.CTW

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 50006.7 LT : 50000.0
No detector description was entered
NOPI-419 U 1/10/95

ROI # 7-1 RANGE : 38 to 150
AREA : Gross = 7072 Net = 6469 +/- 131
CENTROID : 123.75
SHAPE : Fwhm = 39.22 Channels Fwtm = 75.84 Channels

ROI # 7-2 RANGE : 258 to 399
AREA : Gross = 11356 Net = 10880 +/- 147
CENTROID : 370.61
SHAPE : Fwhm = 37.85 Channels Fwtm = 85.44 Channels

ROI # 7-3 RANGE : 511 to 636
AREA : Gross = 5860 Net = 5590 +/- 105
CENTROID : 607.53
SHAPE : Fwhm = 46.43 Channels Fwtm = 80.37 Channels

NP419U.CHW

0:	0	0	0	0	0	0	0
0	0	0					
10:	0	1	2	6	7	10	6
5	11	6					
20:	7	9	5	8	4	8	9
7	11	7					
30:	5	9	10	7	12	8	8
5	6	7					
40:	9	8	17	18	11	10	20
9	12	22					
50:	16	15	18	19	19	20	10
21	17	22					
60:	23	26	21	23	25	25	27
37	25	20					
70:	21	35	27	30	38	33	27
56	39	49					
80:	42	40	47	44	44	40	40
56	58	44					
90:	52	47	54	54	80	63	69
91	75	77					
100:	76	86	89	93	102	110	92
112	98	89					
110:	101	102	137	115	127	138	125
140	148	136					
120:	117	139	114	133	135	154	147
150	150	182					
130:	152	125	141	141	126	107	101
85	86	85					
140:	55	46	44	24	20	20	8
9	1	5					
150:	4	8	4	4	1	3	3
2	1	3					
160:	3	0	4	5	1	4	5
5	3	2					
170:	1	3	0	6	1	2	5
4	6	2					
180:	2	7	5	7	1	5	7
5	5	2					
190:	6	5	5	8	1	2	6
6	4	5					
200:	0	8	5	9	3	5	3
5	9	5					
210:	6	10	7	5	11	6	7
8	5	6					
220:	6	6	4	5	5	3	6
3	4	4					
230:	3	2	3	1	5	7	3
3	3	6					
240:	5	3	2	4	2	2	0
4	7	5					
250:	9	1	3	3	4	7	7
5	5	5					
260:	8	8	7	10	3	8	7
8	5	12					
270:	8	11	15	4	6	6	14
9	11	11					
280:	14	12	13	15	18	15	9
6	20	24					
290:	15	20	24	13	13	12	20
22	14	25					

300:	22	28	30	22	20	28	37
22	27	26					
310:	27	31	33	45	42	36	47
39	49	35					
320:	53	46	37	48	53	55	62
63	54	66					
330:	62	78	72	77	81	96	86
98	78	91					
340:	100	107	96	107	108	102	129
110	138	112					
350:	139	155	177	154	178	144	153
179	173	197					
360:	169	200	204	183	193	195	188
225	202	203					
370:	215	188	203	239	207	252	243
228	245	233					
380:	211	226	197	200	190	163	146
104	106	91					
390:	64	65	45	24	18	11	13
2	0	0					
400:	3	0	1	1	2	0	2
2	1	2					
410:	0	1	3	0	0	0	0
1	1	0					
420:	1	1	0	0	0	0	0
0	0	0					
430:	1	0	1	0	1	1	1
1	1	0					
440:	1	0	1	1	0	2	0
1	2	1					
450:	0	2	0	0	6	2	1
0	0	1					
460:	3	0	2	0	2	1	1
1	0	5					
470:	4	1	2	1	2	2	0
1	5	2					
480:	2	0	4	1	0	2	4
0	3	1					
490:	1	3	4	6	3	4	6
2	3	2					
500:	3	4	7	6	1	4	7
2	4	2					
510:	3	3	2	5	7	4	5
9	6	6					
520:	5	7	7	11	8	6	8
13	5	8					
530:	10	10	7	6	8	9	8
6	11	13					
540:	12	13	22	10	7	14	22
18	19	12					

550:	22	20	20	20	27	26	27
32	26	26					
560:	32	22	25	28	31	32	41
29	34	45					
570:	34	58	41	50	39	51	40
49	63	61					
580:	62	70	67	69	74	66	73
77	75	91					
590:	101	98	84	93	108	96	97
101	113	91					
600:	85	102	85	103	111	98	106
129	112	120					
610:	113	105	132	120	148	110	96
111	110	101					
620:	103	87	66	73	56	37	30
28	25	16					
630:	9	4	5	2	2	0	1
2	1	0					
640:	1	3	1	2	3	1	1
2	2	0					
650:	0	0	1	2	4	2	5
2	2	2					
660:	5	2	3	1	1	1	2
3	3	1					
670:	1	0	1	0	0	0	0
0	0	0					
680:	0	0	0	0	0	0	1
1	0	0					
690:	0	0	0	0	0	0	0
0	0	0					
700:	0	0	0	0	0	0	1
0	0	0					
710:	0	0	0	0	0	0	1
1	0	0					
720:	0	0	0	1	0	0	0
0	1	0					
730:	1	0	0	0	0	0	0
0	1	0					
740:	0	0	0	2	0	0	1
1	2	1					
750:	0	0	0	0	2	2	1
3	0	1					
760:	2	1	2	0	1	3	2
0	1	1					
770:	1	3	1	1	3	0	1
2	1	0					
780:	3	1	0	1	2	0	1
0	0	0					
790:	0	0	0	1	0	0	0
0	0	2					
800:	1	0	0	1	0	0	0
0	0	0					
810:	0	0	0	0	0	0	1

820:	1	1	0	0	0	0	0
0	0	0					
830:	0	0	0	0	2	1	0
0	0	1					
840:	0	1	0	0	1	0	1
0	0	0					
850:	0	0	0	0	0	0	0
1	0	0					
860:	0	0	0	0	0	0	0
0	0	0					
870:	0	0	0	0	0	0	0
0	1	0					
880:	0	1	0	1	0	0	0
0	0	0					
890:	0	0	0	0	0	0	0
0	0	0					
900:	0	0	0	0	1	0	0
0	0	0					
910:	0	0	0	1	0	1	0
0	0	0					
920:	0	2	0	0	0	0	0
0	0	0					
930:	0	0	0	0	0	0	0
2	0	0					
940:	1	0	0	0	0	0	0
0	0	1					
950:	2	0	1	0	0	0	0
0	0	1					
960:	0	0	1	0	0	1	0
1	0	0					
970:	1	0	0	2	1	1	0
0	0	0					
980:	1	1	1	0	0	1	0
0	0	0					
990:	2	1	2	0	0	0	0
0	0	0					
1000:	7	14	7	8	8	12	5
11	16	12					
1010:	18	8	13	11	13	8	8
12	10	9					
1020:	11	9	4	2			

NP419TH.CHW

MCB # 1 ACQ 01-08-95 AT 22:08:57 RT : 75008.3 LT : 75000.0
No detector description was entered
NOPI-419 Th 1/10/95

ROI # 8-1 RANGE : 12 = 393.61keV to 63 = 405.25keV
AREA : Gross = 530 Net = 287 +/- 46
CENTROID : 39.87 = 399.97keV
SHAPE : Fwhm = 0.87keV Fwtm = 4.75keV

ID : No close library match

ROI # 8-2 RANGE : 187 = 433.55keV to 364 = 473.95keV
AREA : Gross = 21488 Net = 21043 +/- 184
CENTROID : 330.66 = 466.34keV
SHAPE : Fwhm = 10.76keV Fwtm = 18.28keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.281 +/- 0.00

ROI # 8-3 RANGE : 567 = 520.28keV to 684 = 546.98keV
AREA : Gross = 5206 Net = 4911 +/- 102
CENTROID : 656.10 = 540.61keV
SHAPE : Fwhm = 6.04keV Fwtm = 16.29keV

ID : No close library match

ROI # 8-4 RANGE : 697 = 549.95keV to 803 = 574.14keV
AREA : Gross = 2472 Net = 2150 +/- 87
CENTROID : 770.13 = 566.64keV
SHAPE : Fwhm = 7.06keV Fwtm = 14.24keV

ID : No close library match

NP 419 TH. CHW

0:	0	0	0	0	0	0	0
0	0	1					0
10:	0	0	5	5	12	15	9
19	19	8					
20:	13	14	13	14	8	4	8
8	7	12					
30:	17	6	11	11	14	9	12
8	12	21					
40:	18	14	11	15	18	10	15
10	9	9					
50:	13	9	4	11	8	5	7
7	4	8					
60:	5	4	2	0	5	5	7
5	4	5					
70:	6	4	6	9	3	3	3
4	8	0					
80:	3	5	2	4	4	8	3
6	2	2					
90:	1	4	4	3	7	3	5
5	2	1					
100:	5	5	3	2	2	4	4
10	3	5					
110:	3	2	6	2	4	4	4
1	9	3					
120:	3	5	2	4	2	0	5
2	7	3					
130:	3	6	3	6	4	4	5
7	8	2					
140:	4	4	1	4	2	8	4
7	3	4					
150:	3	3	6	7	13	6	1
8	4	9					
160:	9	6	3	8	2	2	6
4	4	9					
170:	9	3	4	9	1	7	13
5	6	4					
180:	3	7	5	5	7	7	4
2	5	4					
190:	6	8	4	6	16	6	9
4	9	3					
200:	9	8	5	8	8	8	8
5	9	6					
210:	9	9	9	4	6	7	4
3	11	13					
220:	8	11	6	10	12	10	11
10	13	14					
230:	12	17	9	6	8	14	8
15	14	10					
240:	17	9	11	18	14	13	13
14	16	13					
250:	18	12	29	21	22	21	27
19	34	27					
260:	21	20	31	38	25	27	26
31	43	35					
270:	36	49	36	45	46	55	45
66	63	64					
280:	58	67	85	92	72	103	95
89	91	118					
290:	118	133	133	131	155	146	175

300:	211	241	219	231	228	253	249
243	260	292					
310:	278	279	287	298	336	308	300
316	322	340					
320:	338	316	362	388	344	385	382
401	414	442					
330:	473	416	450	456	451	517	464
469	456	495					
340:	456	419	408	332	367	295	263
230	176	150					
350:	130	99	71	74	33	32	13
13	12	4					
360:	3	5	0	1	3	2	1
1	0	1					
370:	2	0	1	1	2	3	1
3	0	1					
380:	1	1	2	0	1	2	0
0	1	4					
390:	0	3	2	0	2	3	1
1	2	1					
400:	1	0	1	1	1	2	1
0	0	0					
410:	2	1	1	0	1	4	1
2	3	1					
420:	1	0	1	3	3	1	1
1	0	1					
430:	1	0	2	0	1	1	1
4	2	2					
440:	2	1	0	1	2	2	4
3	0	1					
450:	1	3	2	2	3	1	1
3	1	1					
460:	0	1	1	1	2	1	3
0	0	0					
470:	1	1	2	1	1	2	4
1	1	0					
480:	1	1	0	2	1	3	1
0	3	2					
490:	2	2	0	2	2	0	3
0	1	4					
500:	0	4	2	1	3	1	1
0	3	0					
510:	1	2	1	1	1	3	2
3	1	1					
520:	1	2	1	3	0	1	2
4	2	1					
530:	5	5	4	2	1	6	1
3	2	3					
540:	2	5	2	1	1	5	3
3	3	4					

550:	4	5	2	1	5	2	4
2	3	2					
560:	5	2	6	4	4	7	2
3	7	2					
570:	3	7	2	9	5	9	9
2	4	4					
580:	11	5	5	7	10	4	12
10	6	9					
590:	10	8	11	13	16	21	16
17	20	11					
600:	12	24	24	22	14	20	22
28	26	33					
610:	32	37	38	45	33	30	46
48	55	45					
620:	64	40	55	71	45	60	58
48	60	63					
630:	65	41	53	58	67	62	64
65	55	54					
640:	69	66	53	97	79	91	76
99	88	99					
650:	97	100	118	119	124	95	138
144	121	117					
660:	97	120	134	96	87	93	88
88	71	54					
670:	42	39	33	23	18	19	7
10	8	6					
680:	6	4	3	0	0	3	1
5	2	4					
690:	2	5	3	1	3	3	2
3	1	5					
700:	4	4	2	1	4	4	2
4	4	3					
710:	5	2	7	7	5	3	7
6	4	7					
720:	7	6	6	15	10	10	10
14	7	8					
730:	12	14	13	16	17	18	21
14	20	17					
740:	21	15	25	19	26	19	22
29	33	27					
750:	21	29	26	32	36	37	38
49	37	39					
760:	56	58	56	61	54	51	53
50	53	66					
770:	60	51	75	73	61	54	55
53	60	53					
780:	63	40	37	36	32	22	18
19	22	10					
790:	10	10	8	5	6	9	9
7	7	5					
800:	5	3	3	4	3	6	6
4	9	3					
810:	4	2	3	4	2	5	2
2	3	3					
820:	1	3	2	2	1	3	4
0	2	2					
830:	2	5	2	2	1	0	2
5	3	1					

2	3	2					
850:	2	0	3	4	0	0	0
0	1	2					
860:	2	2	0	1	2	2	2
3	0	1					
870:	0	1	4	4	2	1	3
3	5	1					
880:	1	3	5	4	6	6	5
6	5	9					
890:	5	4	7	9	4	4	9
5	8	8					
900:	5	7	10	7	8	6	12
7	12	8					
910:	3	4	10	10	6	6	13
7	7	11					
920:	8	3	12	5	10	5	8
12	9	7					
930:	15	6	8	12	9	5	7
8	4	8					
940:	6	8	7	4	9	6	6
8	4	3					
950:	2	6	4	5	6	8	2
2	6	6					
960:	2	1	3	1	2	1	2
2	2	2					
970:	1	3	1	1	3	3	4
2	6	1					
980:	4	3	2	1	5	3	2
2	1	5					
990:	2	3	4	6	3	2	3
9	4	7					
1000:	64	98	91	77	102	88	113
119	105	108					
1010:	103	99	99	93	114	96	97
89	112	105					
1020:	83	79	95	29			

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-420

Date 1/3/95

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 = OS-18

2. Record dry weight of sample = .3412 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232u}204.88 pCi/g

Spike wt. .2981 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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 11/4/95 1/10/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/12/95

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 disappears 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 1/12/95

NP420U.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-420 U 1/13/95

ROI # 7-1 RANGE : 26 to 154
AREA : Gross = 8466 Net = 8256 +/- 112
CENTROID : 123.35
SHAPE : Fwhm = 33.04 Channels Fwtm = 69.01 Channels

ROI # 7-2 RANGE : 271 to 400
AREA : Gross = 10384 Net = 10281 +/- 111
CENTROID : 370.39
SHAPE : Fwhm = 39.86 Channels Fwtm = 68.32 Channels

ROI # 7-3 RANGE : 530 to 634
AREA : Gross = 2813 Net = 2707 +/- 67
CENTROID : 608.20
SHAPE : Fwhm = 31.37 Channels Fwtm = 65.61 Channels

NP420U.CTN

0:	0	0	0	0	0	0	0	0
0	0	0						
10:	0	0	0	1	1	7	2	
3	3	4						
20:	4	2	3	3	3	0	2	
4	1	0						
30:	5	6	4	1	4	2	4	
4	6	5						
40:	4	5	10	4	6	4	3	
10	8	6						
50:	5	9	6	5	9	8	12	
5	7	8						
60:	10	15	17	17	14	12	18	
12	16	17						
70:	16	21	23	31	20	31	26	
25	32	37						
80:	32	32	49	31	44	42	37	
52	53	53						
90:	53	57	69	69	78	78	88	
80	92	91						
100:	82	113	114	123	107	119	113	
124	130	115						
110:	165	149	180	182	136	186	187	
187	170	183						
120:	214	198	177	218	210	211	237	
227	218	212						
130:	192	219	185	180	165	167	121	
112	91	65						
140:	49	37	43	23	21	16	7	
3	1	2						
150:	3	2	1	1	1	2	2	
2	0	1						
160:	0	1	2	0	2	2	0	
1	2	1						
170:	4	2	2	3	3	1	2	
3	4	2						
180:	2	3	4	5	3	2	6	
1	4	2						
190:	9	4	4	4	4	9	5	
3	4	4						
200:	4	5	11	8	14	11	10	
12	11	5						
210:	7	11	18	8	6	8	8	
7	10	5						
220:	3	4	8	12	5	10	1	
1	1	1						
230:	1	1	1	4	2	2	3	
3	1	0						
240:	2	2	2	1	1	1	1	
1	2	0						
250:	0	2	4	3	2	4	2	
1	0	2						
260:	1	2	0	0	1	4	3	
2	2	3						
270:	4	2	0	3	4	0	7	
5	3	3						
280:	5	3	3	8	5	3	6	

290:	10	7	12	7	8	12	10
8	7	10					
300:	10	21	8	12	17	7	18
11	17	20					
310:	16	17	16	25	22	22	21
26	27	21					
320:	20	37	23	24	28	28	34
32	39	58					
330:	37	42	50	40	64	67	68
66	89	71					
340:	91	88	71	120	106	102	132
120	131	139					
350:	145	167	142	164	178	157	184
168	184	204					
360:	197	184	217	207	205	188	198
244	237	240					
370:	238	231	260	258	246	275	253
242	262	246					
380:	211	209	224	178	156	132	101
74	60	51					
390:	40	30	17	13	10	9	2
1	0	0					
400:	0	1	1	2	1	1	0
2	0	0					
410:	0	0	1	0	0	0	0
1	1	1					
420:	0	0	1	0	0	0	0
0	0	0					
430:	0	1	0	1	0	0	0
1	1	1					
440:	1	0	0	0	0	1	1
0	1	0					
450:	0	0	0	0	0	1	0
1	0	0					
460:	1	0	0	0	1	0	0
0	1	0					
470:	0	0	0	0	0	0	1
0	0	0					
480:	0	1	0	0	1	0	0
0	0	0					
490:	1	0	1	0	0	0	0
1	1	2					
500:	0	0	0	0	0	0	0
0	0	0					
510:	2	0	0	1	0	1	0
0	2	0					
520:	0	1	1	1	0	2	1
3	1	1					
530:	2	0	2	4	1	3	2
1	6	4					
540:	6	2	3	6	1	3	2
8	6	7					

550:	3	4	7	10	9	4	5
3	13	3					
560:	6	12	11	10	22	14	11
15	23	21					
570:	22	16	20	20	20	20	26

580:	31	27	32	32	42	40	35
56	49	32					
590:	38	47	35	38	50	54	52
58	46	46					
600:	59	58	61	64	52	57	67
69	61	61					
610:	65	76	82	79	84	70	67
49	49	39					
620:	50	29	24	21	17	13	7
5	6	2					
630:	7	1	2	0	1	1	1
0	1	1					
640:	0	0	0	1	1	1	3
2	1	2					
650:	2	0	1	3	0	2	0
1	1	0					
660:	2	1	0	0	0	1	0
0	1	0					
670:	0	1	0	0	0	0	0
0	0	1					
680:	0	0	0	0	0	0	0
0	0	0					
690:	1	0	0	0	0	0	0
0	0	0					
700:	0	0	0	0	0	0	0
0	0	0					
710:	0	0	0	1	0	0	0
0	0	0					
720:	0	0	0	0	0	0	0
0	0	1					
730:	0	0	0	0	0	1	0
1	0	0					
740:	0	0	0	0	0	1	0
1	2	0					
750:	0	0	0	3	3	0	0
0	2	0					
760:	1	1	2	0	0	1	2
0	4	3					
770:	1	1	0	1	0	0	0
3	1	1					
780:	0	1	0	0	1	0	0
0	1	0					
790:	0	0	0	0	0	0	0
0	0	1					
800:	0	0	0	0	0	0	0
0	0	1					
810:	0	0	0	0	0	0	0
0	2	0					
820:	0	1	0	0	0	0	0
1	0	0					
830:	0	0	0	0	0	0	0
0	0	0					
840:	0	1	0	0	0	0	0
0	0	0					
850:	0	0	0	0	0	0	0
0	1	0					
860:	0	0	0	0	0	0	0
0	0	0					
870:	0	0	0	0	0	0	0
0	0	0					
880:	0	0	0	0	0	0	0
0	0	0					
890:	0	0	0	2	0	0	0
0	0	2					
900:	0	0	1	0	0	0	0

910:	0	0	1	0	1	0	1
0	0	0					
920:	0	0	0	0	0	0	0
0	0	0					
930:	0	1	0	0	0	1	0
0	0	0					
940:	0	0	0	0	1	1	0
1	0	0					
950:	0	0	0	0	0	0	0
0	0	0					
960:	0	0	0	1	0	0	0
0	0	0					
970:	0	0	0	0	0	0	0
0	1	0					
980:	0	0	0	0	0	1	0
0	0	0					
990:	0	0	0	0	0	1	0
0	0	0					
1000:	2	5	1	5	3	4	4
3	5	0					
1010:	3	4	4	3	4	3	4
3	5	4					
1020:	3	3	3	0			

NP420Th.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 70239.2 LT : 70232.4
No detector description was entered
NOPI-420 Th 1/13/95

ROI # 8-1 RANGE : 11 = 393.38keV to 61 = 404.79keV
AREA : Gross = 372 Net = 245 +/- 35
CENTROID : 14.32 = 394.13keV
SHAPE : Fwhm = 0.45keV Fwtm = 1.99keV

ID : No close library match

ROI # 8-2 RANGE : 203 = 437.20keV to 362 = 473.49keV
AREA : Gross = 37139 Net = 36447 +/- 233
CENTROID : 331.24 = 466.47keV
SHAPE : Fwhm = 10.61keV Fwtm = 17.97keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.519 +/- 0.00

ROI # 8-3 RANGE : 555 = 517.54keV to 680 = 546.07keV
AREA : Gross = 4342 Net = 4241 +/- 79
CENTROID : 655.03 = 540.37keV
SHAPE : Fwhm = 6.13keV Fwtm = 17.65keV

ID : No close library match

ROI # 8-4 RANGE : 718 = 554.74keV to 819 = 577.79keV
AREA : Gross = 1063 Net = 858 +/- 64
CENTROID : 770.11 = 566.63keV
SHAPE : Fwhm = 6.32keV Fwtm = 16.85keV

ID : No close library match

NP420TH. CHW

0:	0	0	0	0	0	0	0
0	0	1					0
10:	0	0	3	7	20	17	7
9	10	5					
20:	10	9	4	1	8	14	8
4	17	4					
30:	4	9	3	9	9	7	8
5	10	12					
40:	8	14	6	3	6	5	7
15	7	4					
50:	9	8	6	7	8	3	8
4	6	4					
60:	0	1	7	7	3	7	3
2	5	8					
70:	4	3	3	5	3	7	9
7	5	6					
80:	4	9	7	5	4	4	4
1	5	4					
90:	2	5	5	5	4	5	9
6	8	5					
100:	2	7	4	4	5	3	4
5	1	3					
110:	7	5	3	7	11	9	9
9	5	4					
120:	7	3	4	3	4	6	5
4	5	4					
130:	7	6	5	8	6	5	4
3	9	8					
140:	14	7	9	8	8	8	3
4	6	11					
150:	6	4	5	6	8	8	10
8	6	5					
160:	7	10	7	11	7	10	10
5	2	11					
170:	16	6	7	8	13	11	7
10	9	9					
180:	10	7	8	13	8	17	6
5	15	12					
190:	12	6	11	9	12	9	8
13	8	14					
200:	6	12	6	7	7	9	12
11	14	14					
210:	23	11	11	21	9	21	17
12	13	19					
220:	16	14	17	10	26	14	22
18	30	17					
230:	20	20	18	21	17	12	27
27	16	25					
240:	28	33	23	24	23	34	35
35	28	43					
250:	27	48	41	40	27	51	37
45	50	40					
260:	54	42	32	64	64	60	70
70	71	61					
270:	68	66	84	77	89	81	110
98	115	130					
280:	115	130	117	131	139	165	182
154	210	202					

301	298	347					
300:	387	380	381	406	401	448	425
456	478	498					
310:	491	466	494	482	531	528	543
536	555	521					
320:	559	635	593	624	658	688	727
718	755	746					
330:	788	849	795	834	864	904	890
809	895	872					
340:	758	693	752	610	527	505	402
319	259	216					
350:	151	103	76	49	36	21	15
7	6	3					
360:	0	2	1	1	2	0	0
1	1	0					
370:	0	3	1	3	1	2	2
0	2	0					
380:	2	1	1	1	1	2	0
3	1	0					
390:	1	0	1	0	1	2	2
1	1	3					
400:	0	0	4	2	0	2	1
0	1	1					
410:	4	1	0	1	0	0	1
0	2	0					
420:	0	0	0	2	0	0	0
1	2	0					
430:	1	1	0	2	1	0	1
1	0	0					
440:	1	0	0	1	1	0	0
1	2	1					
450:	4	1	0	2	0	2	0
2	0	1					
460:	3	1	2	3	2	1	2
1	1	1					
470:	1	2	1	1	2	1	1
3	1	0					
480:	0	2	0	1	1	0	2
3	3	3					
490:	1	3	2	1	1	0	0
0	0	2					
500:	1	0	0	1	1	2	0
3	1	4					
510:	1	1	1	1	2	2	3
2	3	0					
520:	0	3	0	1	2	1	1
2	0	3					
530:	1	1	0	5	0	2	2
3	4	3					
540:	1	3	3	1	6	5	1
3	1	4					

550:	4	4	1	1	2	0	2
3	3	9					
560:	2	5	3	4	7	8	6
3	5	4					
570:	6	5	6	9	7	4	7
4	7	6					
580:	5	12	7	7	9	5	7
11	10	4					
590:	5	11	6	6	9	13	14
10	16	16					
600:	12	15	16	26	22	19	17
19	33	25					
610:	25	29	28	39	37	29	37
39	39	46					
620:	49	45	46	61	42	48	47
40	39	53					
630:	39	37	37	50	49	46	43
56	55	57					
640:	53	65	54	61	57	85	71
79	84	83					
650:	94	99	89	108	95	122	105
127	96	84					
660:	98	90	92	79	75	71	64
52	47	43					
670:	37	18	19	13	14	8	8
4	0	0					
680:	0	2	0	3	1	4	0
2	3	2					
690:	2	0	1	1	2	0	0
1	1	4					
700:	0	7	2	1	4	2	3
2	2	4					
710:	3	3	4	1	3	2	0
0	2	2					
720:	2	3	5	0	5	5	8
5	3	5					
730:	2	5	3	7	5	6	5
10	5	8					
740:	9	5	2	12	5	7	11
7	15	9					
750:	13	17	9	10	14	10	16
18	8	11					
760:	15	18	13	16	26	17	16
16	30	18					
770:	23	19	18	18	27	14	27
13	24	25					

10	10	11						
790:	12	6	9	10	8	5	9	
13	9	10						
800:	9	6	8	8	4	7	10	
12	10	5						
810:	5	9	7	10	8	8	4	
5	1	1						
820:	7	4	1	4	0	2	3	
1	0	2						
830:	3	2	1	4	2	4	2	
3	1	2						
840:	5	2	1	3	0	4	6	
1	3	0						
850:	2	2	5	4	4	4	2	
5	0	1						
860:	2	3	0	2	3	4	3	
3	5	2						
870:	5	3	6	2	3	7	3	
6	4	2						
880:	8	1	9	8	2	9	12	
14	6	7						
890:	11	11	13	15	13	11	17	
12	14	10						
900:	14	9	7	19	13	12	20	
15	16	17						
910:	12	16	8	12	15	7	9	
15	14	15						
920:	16	13	13	16	12	14	13	
8	9	9						
930:	13	9	8	9	8	6	7	
3	5	2						
940:	3	2	2	2	2	0	2	
1	1	2						
950:	3	0	1	0	1	3	3	
2	0	1						
960:	1	2	1	1	0	1	1	
0	1	0						
970:	0	0	1	0	1	0	1	
0	1	3						
980:	0	0	3	2	2	0	1	
1	2	1						
990:	2	2	1	4	3	2	5	
2	3	3						
1000:	40	34	25	29	49	30	39	
42	46	45						
1010:	36	28	37	33	34	29	45	
35	35	35						
1020:	38	28	37	8				

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-421

Date 1/3/95

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 = OS-18

2. Record dry weight of sample = .3235 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25 B

Reference Date 1/22/93

Reference Activity ^{232U} 204.88 pCi/g

Spike wt. .2966 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/12/95

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 disappears 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 1/12/95

NP4214.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-421 U 1/13/95

ROI # 9-1 RANGE : 28 = 394.63keV to 159 = 424.91keV
AREA : Gross = 10310 Net = 9650 +/- 153
CENTROID : 127.83 = 417.70keV
SHAPE : Fwhm = 9.00keV Fwtm = 18.06keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.193 +/- 0.00

ROI # 9-2 RANGE : 246 = 445.02keV to 405 = 481.77keV
AREA : Gross = 12036 Net = 11796 +/- 134
CENTROID : 378.28 = 475.59keV
SHAPE : Fwhm = 9.38keV Fwtm = 18.44keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.236 +/- 0.00

ROI # 9-3 RANGE : 519 = 508.11keV to 640 = 536.08keV
AREA : Gross = 2326 Net = 2186 +/- 70
CENTROID : 613.60 = 529.98keV
SHAPE : Fwhm = 8.17keV Fwtm = 18.42keV

ID : No close library match

NP421 u.c HW

0:	0	1	0	0	0	0	0
0	0	2					0
10:	0	1	4	2	4	10	16
12	9	10					
20:	11	7	7	10	5	9	7
2	9	4					
30:	11	10	7	9	10	9	12
6	6	14					
40:	14	11	15	11	21	10	12
13	15	12					
50:	19	22	18	15	16	12	14
21	24	29					
60:	24	38	32	24	20	27	21
25	24	27					
70:	19	26	34	35	36	43	36
41	39	50					
80:	37	49	46	59	50	52	48
58	56	57					
90:	81	66	62	78	77	87	86
66	85	93					
100:	88	94	97	105	84	117	118
119	122	141					
110:	143	128	145	153	181	176	183
169	180	162					
120:	194	195	187	218	200	219	208
215	192	232					
130:	230	249	223	225	233	227	224
194	202	204					
140:	195	155	131	120	110	59	62
48	30	28					
150:	16	11	4	5	4	3	2
2	1	3					
160:	1	3	4	1	1	5	1
2	4	2					
170:	1	2	1	2	2	2	3
0	3	3					
180:	2	3	6	2	3	6	5
4	2	4					
190:	6	8	5	8	4	7	8
10	12	6					
200:	7	4	8	14	9	10	11
11	11	12					
210:	5	15	8	5	10	8	7
9	21	14					
220:	10	7	11	7	4	10	5
1	7	5					
230:	7	8	6	5	3	4	9
4	6	5					
240:	3	2	5	4	2	3	3
1	5	6					
250:	9	10	2	6	5	7	6
5	7	6					
260:	6	7	6	6	6	5	16
6	8	6					
270:	5	12	9	14	11	11	11
11	8	10					
280:	12	12	9	12	13	15	13
13	9	15					

14	18	10					
300:	22	22	29	16	18	18	26
27	33	25					
310:	27	26	25	30	24	22	31
22	27	57					
320:	30	35	41	43	31	50	44
47	51	49					
330:	60	59	57	75	64	72	58
92	76	62					
340:	85	93	85	107	91	83	110
99	118	110					
350:	106	99	162	134	143	143	171
164	185	176					
360:	177	179	207	192	179	184	199
180	187	214					
370:	217	180	218	236	219	229	247
238	253	282					
380:	249	253	247	278	255	253	274
247	199	185					
390:	137	161	114	109	79	61	60
27	20	9					
400:	8	4	2	0	0	0	4
0	0	0					
410:	1	0	1	0	0	0	0
0	0	0					
420:	0	0	0	0	0	0	0
0	0	1					
430:	0	0	0	0	0	0	0
0	0	0					
440:	1	1	0	1	1	1	1
1	0	0					
450:	0	0	0	1	0	0	0
0	0	0					
460:	0	1	0	0	0	0	0
0	0	0					
470:	1	0	0	0	1	0	0
0	1	0					
480:	0	0	0	1	0	0	0
1	2	0					
490:	0	2	0	0	0	1	2
2	0	2					
500:	1	0	2	0	1	2	1
0	1	0					
510:	0	3	2	1	1	2	3
3	1	1					
520:	2	4	2	1	3	4	3
3	5	3					
530:	4	1	4	3	5	0	6
5	2	3					
540:	1	3	2	5	7	5	8
7	5	1					

550:	0	5	1	7	7	12	9
7	10	12					
560:	7	7	7	11	12	8	13
14	15	9					
570:	6	9	23	12	10	11	25
16	10	23					
580:	17	21	23	27	16	25	32
19	32	31					
590:	34	42	24	32	35	43	26
31	33	38					
600:	46	37	44	40	52	44	35
43	46	32					
610:	30	52	49	49	49	52	49
49	45	53					
620:	43	47	43	48	38	49	34
25	21	24					
630:	12	15	8	3	6	3	3
1	0	0					
640:	0	0	2	2	0	2	1
1	0	0					
650:	1	0	1	1	0	0	1
0	2	0					
660:	1	2	3	2	2	3	0
2	0	0					
670:	0	0	2	1	0	0	1
1	0	0					
680:	0	0	0	1	0	0	0
0	0	0					
690:	0	0	1	0	0	0	0
1	0	0					
700:	0	1	0	1	0	0	0
0	0	0					
710:	0	1	0	0	0	0	0
0	0	0					
720:	0	0	0	0	0	0	0
0	0	2					
730:	1	0	0	0	0	0	0
0	0	0					
740:	0	0	1	0	0	1	0
0	0	1					
750:	0	0	1	0	0	0	0
0	0	0					
760:	0	0	1	0	0	1	0
1	1	0					
770:	0	1	2	1	2	0	0
3	1	0					
780:	0	2	0	1	1	0	0
1	0	1					
790:	0	0	1	0	0	0	0
0	1	0					
800:	0	0	0	0	0	0	0
0	0	0					
810:	0	0	0	0	0	0	0
0	0	0					
820:	0	0	0	1	0	0	0
0	0	0					
830:	0	0	0	0	0	0	0
0	0	3					
840:	0	1	0	0	0	0	0
1	0	0					
850:	0	0	0	0	0	0	1

860:	0	0	0	0	0	0	0
0	0	1	0	0	0	0	0
870:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
880:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
890:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
900:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
910:	1	1	0	0	0	0	0
0	0	0	0	0	0	0	0
920:	0	0	0	0	0	0	1
0	0	0	0	0	0	0	0
930:	0	0	0	0	0	1	0
1	0	0	0	0	0	0	0
940:	0	0	0	0	1	1	0
0	0	1	0	0	0	0	0
950:	0	0	0	0	0	1	1
0	1	0	0	0	0	0	0
960:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
970:	0	1	0	0	0	0	0
0	0	0	0	0	0	0	0
980:	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0
990:	0	1	0	2	0	0	0
0	1	0	0	0	0	0	0
1000:	4	4	1	6	4	2	4
6	3	6	0	0	0	0	0
1010:	2	1	3	3	8	6	2
2	4	3	0	0	0	0	0
1020:	4	5	2	0	0	0	0

NP421Th.CHN

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 70376.0 LT : 70369.2
No detector description was entered
NOPI-421 Th 1/13/95

ROI # 10-1 RANGE : 11 = 396.87keV to 64 = 409.14keV
AREA : Gross = 638 Net = 197 +/- 61
CENTROID : 16.31 = 398.10keV
SHAPE : Fwhm = 0.71keV Fwtm = 2.41keV

ID : No close library match

ROI # 10-2 RANGE : 140 = 426.74keV to 355 = 476.53keV
AREA : Gross = 67294 Net = 65602 +/- 353
CENTROID : 322.08 = 468.91keV
SHAPE : Fwhm = 10.32keV Fwtm = 20.18keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.932 +/- 0.01

ROI # 10-3 RANGE : 525 = 515.90keV to 674 = 550.41keV
AREA : Gross = 6303 Net = 5966 +/- 119
CENTROID : 644.44 = 543.56keV
SHAPE : Fwhm = 6.02keV Fwtm = 17.32keV

ID : No close library match

ROI # 10-4 RANGE : 695 = 555.27keV to 815 = 583.06keV
AREA : Gross = 1883 Net = 1441 +/- 100
CENTROID : 763.00 = 571.02keV
SHAPE : Fwhm = 5.98keV Fwtm = 18.30keV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NO PI-422

Date 1/3/95

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 = 05-18.

2. Record dry weight of sample = .3056 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232u} 204.88 pCi/g

Spike wt. .2990 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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:

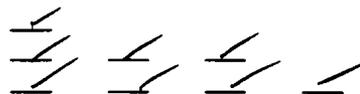
7. Add 1 ml of perchloric acid (HClO_4) to the sample solution in the teflon beaker. Evaporate to fumes of HClO_4 . Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).
8. Transfer solution to a 50 ml PP centrifuge tube. Add ~10 mg Fe carrier and coprecipitate 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. Dissolve 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 # 496 #3A)

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

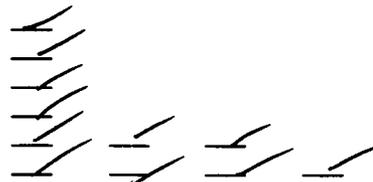


Separation Date 11/10/95

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

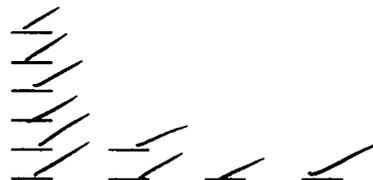
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/12/95

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 disappears 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 1/12/95

NP4ZZU.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-422 U 1/13/95

ROI # 11-1 RANGE : 30 = 394.56keV to 164 = 425.43keV
AREA : Gross = 9120 Net = 8985 +/- 109
CENTROID : 132.48 = 418.16keV
SHAPE : Fwhm = 9.03keV Fwtm = 17.63keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.180 +/- 0.00

ROI # 11-2 RANGE : 279 = 451.92keV to 415 = 483.25keV
AREA : Gross = 9640 Net = 9531 +/- 109
CENTROID : 380.40 = 475.28keV
SHAPE : Fwhm = 8.99keV Fwtm = 17.97keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.191 +/- 0.00

ROI # 11-3 RANGE : 531 = 509.98keV to 648 = 536.93keV
AREA : Gross = 3036 Net = 2977 +/- 64
CENTROID : 618.69 = 530.18keV
SHAPE : Fwhm = 9.59keV Fwtm = 16.98keV

ID : No close library match

MCB # 1 ACQ 01-12-95 AT 02:39:28 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-422 U 1/13/95

ROI # 11-1 RANGE : 23 = 392.94keV to 167 = 426.12keV
AREA : Gross = 9139 Net = 8921 +/- 118
CENTROID : 132.48 = 418.16keV
SHAPE : Fwhm = 9.03keV Fwtm = 17.58keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.178 +/- 0.00

ROI # 11-2 RANGE : 276 = 451.23keV to 414 = 483.02keV
AREA : Gross = 9644 Net = 9527 +/- 110
CENTROID : 380.40 = 475.28keV
SHAPE : Fwhm = 9.03keV Fwtm = 17.98keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.191 +/- 0.00

NP422TH.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:29 RT : 70480.5 LT : 70473.6
No detector description was entered
NOPI-422 Th 1/13/95

ROI # 12-1 RANGE : 12 = 390.26keV to 66 = 402.76keV
AREA : Gross = 559 Net = 163 +/- 58
CENTROID : 28.77 = 394.14keV
SHAPE : Fwhm = 0.28keV Fwtm = 0.66keV

ID : No close library match

ROI # 12-2 RANGE : 165 = 425.68keV to 371 = 473.38keV
AREA : Gross = 39949 Net = 38741 +/- 281
CENTROID : 336.22 = 465.33keV
SHAPE : Fwhm = 11.33keV Fwtm = 21.52keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.550 +/- 0.00

ROI # 12-3 RANGE : 530 = 510.19keV to 688 = 546.77keV
AREA : Gross = 6897 Net = 6579 +/- 121
CENTROID : 660.89 = 540.50keV
SHAPE : Fwhm = 7.21keV Fwtm = 19.13keV

ID : No close library match

ROI # 12-4 RANGE : 698 = 549.09keV to 834 = 580.58keV
AREA : Gross = 1588 Net = 1178 +/- 101
CENTROID : 782.08 = 568.55keV
SHAPE : Fwhm = 1.96keV Fwtm = 14.92keV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-423

Date 1/3/95

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 = 05-18

2. Record dry weight of sample = .3347 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 204.88 pCi/g

Spike wt. .2981 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49043A)

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 11/10/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/12/95

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 disappears 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 1/12/95

NP423 U.C.H.W

MCB # 1 ACQ 01-12-95 AT 02:39:29 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-423 U 1/13/95

ROI # 13-1 RANGE : 11 = 392.99keV to 157 = 426.73keV
AREA : Gross = 5654 Net = 5268 +/- 120
CENTROID : 115.84 = 417.22keV
SHAPE : Fwhm = 8.70keV Fwtm = 17.63keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.105 +/- 0.00

ROI # 13-2 RANGE : 235 = 444.75keV to 398 = 482.41keV
AREA : Gross = 6252 Net = 5891 +/- 124
CENTROID : 362.06 = 474.11keV
SHAPE : Fwhm = 9.31keV Fwtm = 19.61keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.118 +/- 0.00

ROI # 13-3 RANGE : 487 = 502.98keV to 634 = 536.94keV
AREA : Gross = 6402 Net = 6199 +/- 105
CENTROID : 597.37 = 528.48keV
SHAPE : Fwhm = 10.14keV Fwtm = 20.25keV

ID : No close library match

NP423Th.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:29 RT : 70550.9 LT : 70544.0
No detector description was entered
NOPI-423 Th 1/13/95

ROI # 14-1 RANGE : 11 = 391.78keV to 68 = 404.90keV
AREA : Gross = 306 Net = 190 +/- 35
CENTROID : 49.11 = 400.55keV
SHAPE : Fwhm = 0.33keV Fwtm = 4.80keV

ID : No close library match

ROI # 14-2 RANGE : 162 = 426.53keV to 362 = 472.57keV
AREA : Gross = 11090 Net = 10890 +/- 132
CENTROID : 332.91 = 465.88keV
SHAPE : Fwhm = 10.38keV Fwtm = 19.53keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.154 +/- 0.00

ROI # 14-3 RANGE : 540 = 513.54keV to 681 = 546.00keV
AREA : Gross = 5218 Net = 5105 +/- 88
CENTROID : 656.07 = 540.26keV
SHAPE : Fwhm = 5.53keV Fwtm = 17.87keV

ID : No close library match

ROI # 14-4 RANGE : 702 = 550.83keV to 820 = 577.99keV
AREA : Gross = 768 Net = 649 +/- 54
CENTROID : 765.57 = 565.47keV
SHAPE : Fwhm = 5.55keV Fwtm = 10.97keV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-425

Date 1/3/95

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 = OS-18

2. Record dry weight of sample = .2607 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232}U 204.88 pCi/g

Spike wt. .2978 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>14.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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:

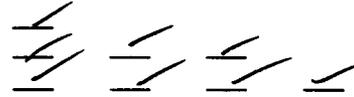
7. Add 1 ml of perchloric acid (HClO_4) to the sample solution in the teflon beaker. Evaporate to fumes of HClO_4 . Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).
8. Transfer solution to a 50 ml PP centrifuge tube. Add ~10 mg Fe carrier and coprecipitate 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. Dissolve 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 # 49643A)

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

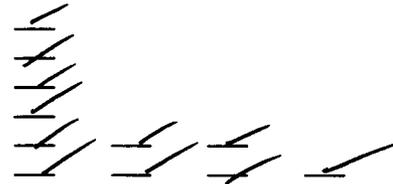


Separation Date 1/4/95 ⁷⁰
1/10/95

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

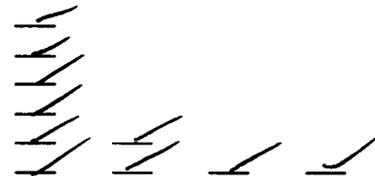
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/12/95

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 1/12/95

NP425 U.CHW

MCB # 1 ACQ 01-12-95 AT 02:39:29 RT : 50005.5 LT : 50000.0
No detector description was entered
NOPI-425 U 1/13/95

ROI # 15-1 RANGE : 27 = 396.82keV to 154 = 425.96keV
AREA : Gross = 5675 Net = 5674 +/- 75
CENTROID : 119.89 = 418.14keV
SHAPE : Fwhm = 8.12keV Fwtm = 15.06keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.113 +/- 0.00

ROI # 15-2 RANGE : 273 = 453.27keV to 401 = 482.64keV
AREA : Gross = 6334 Net = 6269 +/- 87
CENTROID : 370.80 = 475.71keV
SHAPE : Fwhm = 8.72keV Fwtm = 15.26keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.125 +/- 0.00

ROI # 15-3 RANGE : 497 = 504.67keV to 639 = 537.26keV
AREA : Gross = 8736 Net = 8664 +/- 102
CENTROID : 607.04 = 529.93keV
SHAPE : Fwhm = 9.16keV Fwtm = 15.73keV

ID : No close library match

NP 425 Th. c HW

MCB # 1 ACQ 01-12-95 AT 02:39:29 RT : 70601.5 LT : 70594.7
No detector description was entered
NOPI-425 Th 1/13/95

ROI # 16-1 RANGE : 14 = 391.54keV to 67 = 403.64keV
AREA : Gross = 157 Net = 66 +/- 28
CENTROID : 53.89 = 400.65keV
SHAPE : Fwhm = 0.29keV Fwtm = 5.09keV

ID : No close library match

ROI # 16-2 RANGE : 193 = 432.41keV to 372 = 473.29keV
AREA : Gross = 7737 Net = 7736 +/- 88
CENTROID : 340.08 = 466.00keV
SHAPE : Fwhm = 8.80keV Fwtm = 19.68keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.110 +/- 0.00

ROI # 16-3 RANGE : 532 = 509.83keV to 692 = 546.36keV
AREA : Gross = 4527 Net = 4446 +/- 81
CENTROID : 664.90 = 540.17keV
SHAPE : Fwhm = 5.63keV Fwtm = 18.13keV

ID : No close library match

ROI # 16-4 RANGE : 721 = 552.98keV to 811 = 573.54keV
AREA : Gross = 619 Net = 529 +/- 42
CENTROID : 769.52 = 564.06keV
SHAPE : Fwhm = 1.88keV Fwtm = 11.98keV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-424

Date 1/19/95

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 = OS-4

2. Record dry weight of sample = .5122 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232U} 204.88 pCi/g

Spike wt. .9995 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49043A)

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 1/23/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/25/95

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 disappears 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 1/25/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-209

Date 1/19/95

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 = DS-4

2. Record dry weight of sample = .4403 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{204.88} _{^{232}U} pCi/g

Spike wt. 1.0036 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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

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— ✓ — —
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Separation Date 1/23/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/25/95

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 disappears 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 1/25/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-113

Date 1/20/95

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 = 05-4.

2. Record dry weight of sample = .4982 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}U

Reference Date 1/22/93

Reference Activity 204.78 pCi/g

Spike wt. .9914 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 19750A)

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

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Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 4/3/95

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 disappears 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 4/3/95
2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-114

Date 1/19/95

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 = 03-4

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 $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513

Reference Date 1/22/93

Reference Activity ^{232}U 204.88 pCi/g

Spike wt. 1.0041 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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

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Separation Date 1/23/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/25/95

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 disappears 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 1/25/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-115

Date 1/20/97

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 = 05-4

2. Record dry weight of sample = .4982 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25 B

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. .9900 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/3/95

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 disappears 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-116

Date 1/20/95

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 = 054.

2. Record dry weight of sample = .5051 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 _{^{232}U}
Reference Activity 204.88 pCi/g

Reference Date 1/22/93

Spike wt. .9876 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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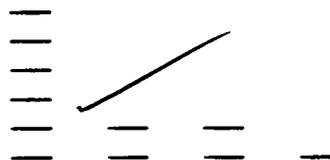
Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

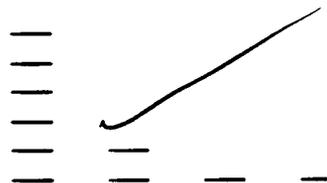
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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaporate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/3/95

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 disappears 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-117

Date 1/20/95

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 = 05-4

2. Record dry weight of sample = 5071 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. .9938 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750 B)

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 1/30/95

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

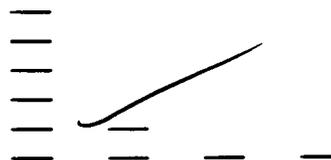
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/3/95

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 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-118

Date 1/20/95

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 = 05-4

2. Record dry weight of sample = .5027 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 253
_{232u}

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. .9974 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH 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. Dissolve 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 # 49750A)

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

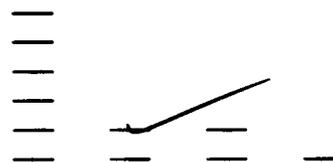


Separation Date 1/30/95

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

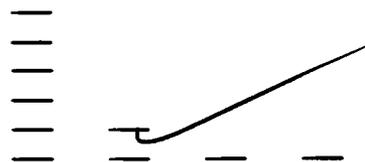
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaporate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/3/95

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 disappears 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-119

Date 1/19/95

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 = 05-4.

2. Record dry weight of sample = .5060 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513
 _{^{232}U}

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0020 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 45643A)

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

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Separation Date 1/23/05

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/25/95

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 disappears 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 1/25/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-120

Date 1/19/95

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 = 05-4

2. Record dry weight of sample = .5216 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25 B
232u

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0061 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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

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Separation Date 1/23/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/26/95

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 disappears 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 1/25/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-121

Date 1/19/95

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 = 05-4.

2. Record dry weight of sample = .5080 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93
Spike wt. 1.0012 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750K)

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

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Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/3/95

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 disappears 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-122

Date 1/19/95

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 = DS-4.

2. Record dry weight of sample = .5110 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253
204.80
Reference Activity ^{232}U pCi/g

Reference Date 1/22/93
Spike wt. 1.0018 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/3/95

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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-123

Date 1/19/95

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 = 05-4.

2. Record dry weight of sample = .5130 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513
 ^{232}U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93

Spike wt. 1.0045 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 1/30/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaporate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/3/95

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 disappears 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 2/1/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-307

Date 1/20/95

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 = 054.

2. Record dry weight of sample = .5032 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity 204.80 pCi/g

Spike wt. .9973 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/9/95

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 disappears 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-397

Date 1/20/95

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 = 05-4.

2. Record dry weight of sample = .2934 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 25B
232u
Reference Activity 204.88 pCi/g

Reference Date 1/22/93

Spike wt. .9995 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH 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. Dissolve 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 # 49750*)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-398

Date 1/20/95

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 = 05-4.

2. Record dry weight of sample = .5054 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93
Spike wt. .9959 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 47750A)

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

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Separation Date 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl

Add ~5 ml conc HNO₃ to dissolve residue

Add equal vol (~5 ml) DI water so soln 8M HNO₃

Load onto column in 8M HNO₃

Wash 3-4 column vols 8M HNO₃

Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaporate U fraction to near dryness

Pick up in about 2 ml conc HNO₃

Dilute with about 2 ml DI water to approx 8M HNO₃

Load onto column in 8M HNO₃

Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe

Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/9/95

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 disappears 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPJ-399

Date 1/20/95

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 = 65-4.

2. Record dry weight of sample = .5059 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. .9976 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl —
Add ~5 ml conc HNO₃ to dissolve residue —
Add equal vol (~5 ml) DI water so soln 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 3-4 column vols 8M HNO₃ — — —
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 HNO₃)

Evaoprate U fraction to near dryness —
Pick up in about 2 ml conc HNO₃ —
Dilute with about 2 ml DI water to approx 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NDPI-400

Date 1/20/95

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 = 05-4.

2. Record dry weight of sample = .4939 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232U

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. .9969 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprte U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-401

Date 1/20/95

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 = 05-4.

2. Record dry weight of sample = .5033 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}U
Reference Activity 704.88 pCi/g

Reference Date 1/22/93

Spike wt. .9958 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 disappears 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-402

Date 1/26/95

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 = 05-4.

2. Record dry weight of sample = .5014 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. 1.0014 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprte U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 disappears 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-403

Date 1/26/95

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 = 05-4.

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 $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232U

Reference Date 1/22/93

Reference Activity 204.82 pCi/g

Spike wt. .9985 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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 2/2/95

***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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaporate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/9/95

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 disappears 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 2/8/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-404

Date 1/26/95

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 = 03-4

2. Record dry weight of sample = .5068 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. 1.0001 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750 K)

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

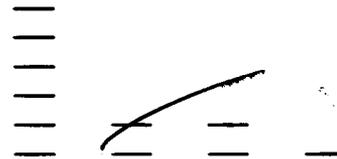


Separation Date 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprte U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/13/95

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 disappears 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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NQPI-405

Date 1/26/95

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 = 05-4

2. Record dry weight of sample = .5020 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513
232U

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0012 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 2/8/92

***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 HNO₃)

Heat Th fraction to evaporate HCl

Add ~5 ml conc HNO₃ to dissolve residue

Add equal vol (~5 ml) DI water so soln 8M HNO₃

Load onto column in 8M HNO₃

Wash 3-4 column vols 8M HNO₃

Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaporate U fraction to near dryness

Pick up in about 2 ml conc HNO₃

Dilute with about 2 ml DI water to approx 8M HNO₃

Load onto column in 8M HNO₃

Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe

Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 disappears 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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-406

Date 1/26/95

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 = 05-4.

2. Record dry weight of sample = .4989 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 201.88 pCi/g

Spike wt. 1.0026 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49758A)

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

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Separation Date 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl

Add ~5 ml conc HNO₃ to dissolve residue

Add equal vol (~5 ml) DI water so soln 8M HNO₃

Load onto column in 8M HNO₃

Wash 3-4 column vols 8M HNO₃

Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness

Pick up in about 2 ml conc HNO₃

Dilute with about 2 ml DI water to approx 8M HNO₃

Load onto column in 8M HNO₃

Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe

Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 disappears 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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-407

Date 1/26/95

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 = 05-4

2. Record dry weight of sample = .5098 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232u} 204.88 pCi/g

Spike wt. 1.0009 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>H₂O₂</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl

Add ~5 ml conc HNO₃ to dissolve residue

Add equal vol (~5 ml) DI water so soln 8M HNO₃

Load onto column in 8M HNO₃

Wash 3-4 column vols 8M HNO₃

Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness

Pick up in about 2 ml conc HNO₃

Dilute with about 2 ml DI water to approx 8M HNO₃

Load onto column in 8M HNO₃

Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe

Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # N@PI-408

Date 1/26/95

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 = 05-4

2. Record dry weight of sample = .5007 g and place in a teflon PFA vessel.

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

$^{232}\text{U}/^{228}\text{Th}$ spike # 258

Reference Date 1/22/93

^{232}U
Reference Activity 204.82 pCi/g

Spike wt. .9987 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 disappears 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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-409

Date 1/26/95

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 = 05.4

2. Record dry weight of sample = .5018 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0016 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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. Dissolve 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 # 49750A)

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

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Separation Date 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPE-410

Date 1/26/95

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 = 05-4.

2. Record dry weight of sample = .5067 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}u

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0030 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/13/95

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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-411

Date 1/26/95

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 = 05-4.

2. Record dry weight of sample = .5059 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 2513

Reference Date 1/22/93

Reference Activity ^{232u} 204.88 pCi/g

Spike wt. .9994 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH 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. Dissolve 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 # 49750A)

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 2/8/95

***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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/13/95

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 2/14/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-212

Date 2/1/95

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 = 03-4

2. Record dry weight of sample = .5356 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
 ^{232}U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93
Spike wt. .4980 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>H₂O₂</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-214

Date 2/1/95

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 = 05-4

2. Record dry weight of sample = .5085 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 2513
 ^{232}U

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. .4975 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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

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Separation Date 2/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaporate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-215

Date 2/1/95

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 = 054

2. Record dry weight of sample = .5176 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232u

Reference Date 1/22/93

Reference Activity 704-88 pCi/g

Spike wt. .4991 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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 2/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl —
Add ~5 ml conc HNO₃ to dissolve residue —
Add equal vol (~5 ml) DI water so soln 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 3-4 column vols 8M HNO₃ — — —
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 HNO₃)

Evaporate U fraction to near dryness —
Pick up in about 2 ml conc HNO₃ —
Dilute with about 2 ml DI water to approx 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-216

Date 2/1/95

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 = OS-4

2. Record dry weight of sample = .5183 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253
 ^{232}U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93
Spike wt. .4977 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49750A)

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 2/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl —
Add ~5 ml conc HNO₃ to dissolve residue —
Add equal vol (~5 ml) DI water so soln 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 3-4 column vols 8M HNO₃ — — —
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 HNO₃)

Evaoprate U fraction to near dryness —
Pick up in about 2 ml conc HNO₃ —
Dilute with about 2 ml DI water to approx 8M HNO₃ —
Load onto column in 8M HNO₃ —
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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.

4/3/95
Th Counting Date 2/22/95

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 disappears 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.

4/3/95
U Counting Date 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-217

Date 2/1/95

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 = OS-4

2. Record dry weight of sample = .5166 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232u

Reference Date 1/22/93

Reference Activity 204.82 pCi/g

Spike wt. .4949 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-218

Date 2/1/95

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 = 05-4

2. Record dry weight of sample = .5283 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232U
Reference Activity 204.88 pCi/g

Reference Date 1/22/93
Spike wt. .4943 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-219

Date 2/1/95

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 = 054.

2. Record dry weight of sample = .5246 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232u
Reference Activity 204.68 pCi/g

Reference Date 1/22/93
Spike wt. .4953 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-221

Date 2/1/95

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 = 054

2. Record dry weight of sample = 0.5129 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253
 ^{228}u
 Reference Activity 204.88 pCi/g

Reference Date 1/22/93
 Spike wt. 4953 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>7</u>
<u>HCl</u>	<u>3</u>
<u>HNO₃</u>	<u>1</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve the precipitate in ~3 ml conc HCl. Add 1 ml ultrapure water to dilute to ~9M.

Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 2/22/95

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 disappears 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 2/21/95

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-113

Date 1/9/95

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 = OS 11 + 21.

2. Record dry weight of sample = .9965^{7.98 1.945} g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 25B

Reference Date 1/22/93

Reference Activity ²³²U 204.88 pCi/g

Spike wt. 1.0004 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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH to pH = 7.
**Note: if sample already contains significant Fe, then Fe carrier does not need to be added.
9. Separate the Fe scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-114

Date 1/9/95

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 = 0311+21.

2. Record dry weight of sample = .9957 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 204.88 pCi/g

Spike wt. .9972 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. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # N O P E - 1 1 5

Date 1/9/95

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 = 0511 + 21.

2. Record dry weight of sample = .9812 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 2.04.88 pCi/g

Spike wt. 1.0022 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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-116

Date 1/9/95

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 = 0511+21.

2. Record dry weight of sample = .9976 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253
232u

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0017 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOP-I-117

Date 1/9/95

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 = 0511 + 21.

2. Record dry weight of sample = .9841 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232U

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0014 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-118

Date 1/9/95

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 = 0511+21.

2. Record dry weight of sample = .9901 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

²³²U Reference Activity 204.88 pCi/g

Spike wt. 1.0027 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-119

Date 1/9/95

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 = 0511+21.

2. Record dry weight of sample = .9985 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. .9984 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. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprte U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-120

Date 1/9/95

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 = 0511+21

2. Record dry weight of sample = .9932 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. 1.0040 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-121

Date 1/9/95

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 = 0511+21.

2. Record dry weight of sample = .9902 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232}U
Reference Activity 204.88 pCi/g

Spike wt. 1.0005 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. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-122

Date 1/9/95

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 = 0511 + 21.

2. Record dry weight of sample = .9991 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{232U} 204.88 pCi/g

Spike wt. 1.0016 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-123

Date 1/9/95

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 = 0511 + 21.

2. Record dry weight of sample = 1.0006 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # Z5B
_{232U}

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0004 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 (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. 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 NH₄OH to pH = 7.
**Note: if sample already contains significant Fe, then Fe carrier does not need to be added.
9. Separate the Fe scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-424

Date 1/3/95

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 = 05-18

2. Record dry weight of sample = .5147 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity ^{^{232}U} 204.87 pCi/g

Spike wt. .2976 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HCl</u>	<u>10.0</u>
<u>HF</u>	<u>0.5</u>
_____	_____
_____	_____

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-1M

Date 1/12/95

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 = .9350 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 253
 _{^{232}U}

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0037 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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 HNO₃)

Heat Th fraction to evaporate HCl _____
Add ~5 ml conc HNO₃ to dissolve residue _____
Add equal vol (~5 ml) DI water so soln 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 3-4 column vols 8M HNO₃ _____
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 HNO₃)

Evaoprate U fraction to near dryness _____
Pick up in about 2 ml conc HNO₃ _____
Dilute with about 2 ml DI water to approx 8M HNO₃ _____
Load onto column in 8M HNO₃ _____
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-113

Date 1/12/95

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 = OS-4 + 11

2. Record dry weight of sample = .9102 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

^{232u}
Reference Activity 204.88 pCi/g

Spike wt. 1.0000 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>10</u>
<u>HCl</u>	<u>2</u>
<u>H₃BO₃</u>	<u>50</u>

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 scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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 1/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-115

Date 1/12/95

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 = 054+11.

2. Record dry weight of sample = .9976 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of ²³²U/²²⁸Th spike to the PFA vessel.

²³²U/²²⁸Th spike # 25B
232u

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0023 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>10</u>
<u>HCl</u>	<u>2</u>
<u>H₃BO₃</u>	<u>50</u>

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:

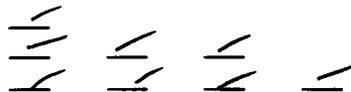
7. Add 1 ml of perchloric acid (HClO₄) to the sample solution in the teflon beaker. Evaporate to fumes of HClO₄. Pick up in a small amount (~2 ml) of conc. HCl and dilute to approximately 2M (total ~10 ml).
8. Transfer solution to a 50 ml PP centrifuge tube. Add ~10 mg Fe carrier and coprecipitate actinides by addition of NH₄OH to pH = 7.
**Note: if sample already contains significant Fe, then Fe carrier does not need to be added.
9. Separate the Fe scavage by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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

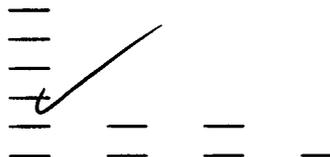


Separation Date 1/16/95

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

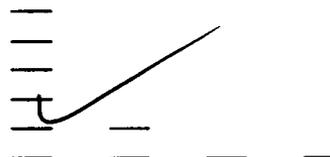
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-116

Date 1/12/95

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 = 054 + 11.

2. Record dry weight of sample = .9840 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B
232u

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0016 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>10</u>
<u>HCl</u>	<u>2</u>
<u>H₃BO₃</u>	<u>50</u>

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. Dissolve 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 # 49043A)

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

✓
✓
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Separation Date 1/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 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 disappears 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 _____

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-117

Date 1/12/95

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 = 054+11

2. Record dry weight of sample = .9707 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity 204.88 pCi/g

Spike wt. 1.0044 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>10</u>
<u>HCl</u>	<u>2</u>
<u>H₃BO₃</u>	<u>50</u>

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. Dissolve 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 # 49643K)

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

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Separation Date 1/16/95

***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 HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
Elute 4-5 column vols 9M HCl --> Th

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Uranium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> Fe
Elute 4-5 vols 0.1M HCl --> U

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Source Preparation:

Thorium - OH⁻ precipitation onto filter

1. Evaporate solution containing Th to near dryness, add ~ 2 ml conc HNO₃ 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 1/18/95

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 disappears 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 1/17/95

NPI17U.CHN

MCB # 1 ACQ 01-17-95 AT 05:12:01 RT : 56787.5 LT : 56778.7
No detector description was entered
NOPI-117 U 1/18/95

ROI # 15-1 RANGE : 33 = 398.20keV to 152 = 425.50keV
AREA : Gross = 7194 Net = 6894 +/- 112
CENTROID : 123.06 = 418.86keV
SHAPE : Fwhm = 7.02keV Fwtm = 13.00keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.121 +/- 0.00

ROI # 15-2 RANGE : 269 = 452.35keV to 404 = 483.33keV
AREA : Gross = 7430 Net = 7294 +/- 101
CENTROID : 371.31 = 475.83keV
SHAPE : Fwhm = 9.03keV Fwtm = 15.39keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.128 +/- 0.00

ROI # 15-3 RANGE : 518 = 509.49keV to 636 = 536.57keV
AREA : Gross = 3474 Net = 3355 +/- 75
CENTROID : 609.35 = 530.46keV
SHAPE : Fwhm = 5.69keV Fwtm = 13.24keV

ID : No close library match

Handwritten calculation:
$$\frac{6894}{40000} = 1.057$$

Handwritten calculation:
$$\frac{6894}{41869}$$

NP117Th.c#W

MCB # 1 ACQ 01-18-95 AT 10:31:52 RT : 25001.6 LT : 25000.0
No detector description was entered
NOPI-117 Th 1/19/95

ROI # 5-1 RANGE : 33 = 3.83keV to 97 = 4.00keV
AREA : Gross = 361 Net = 360 +/- 19
CENTROID : 76.37 = 3.95keV
SHAPE : Fwhm = 0.02keV Fwtm = 0.13keV

ID : No close library match

ROI # 5-2 RANGE : 191 = 4.26keV to 354 = 4.69keV
AREA : Gross = 34565 Net = 34524 +/- 189
CENTROID : 325.95 = 4.62keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.15keV

ID : No close library match

ROI # 5-3 RANGE : 480 = 5.03keV to 628 = 5.43keV
AREA : Gross = 7821 Net = 7820 +/- 89
CENTROID : 604.47 = 5.37keV
SHAPE : Fwhm = 0.05keV Fwtm = 0.16keV

ID : No close library match

ROI # 5-4 RANGE : 640 = 5.46keV to 757 = 5.77keV
AREA : Gross = 1401 Net = 1283 +/- 59
CENTROID : 703.02 = 5.63keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.17keV

ID : No close library match

U/Th Digestion (Nopal rock samples)

Sample I. D. # NOPI-118

Date 1/22/95

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 = 024210.

2. Record dry weight of sample = .9805 g and place in a teflon PFA vessel.
3. Using a weighing boat, quantitatively add a known amount of $^{232}\text{U}/^{228}\text{Th}$ spike to the PFA vessel.

$^{232}\text{U}/^{228}\text{Th}$ spike # 25B

Reference Date 1/22/93

Reference Activity 204.80 pCi/g

Spike wt. 1.0021 g

3. Add reagents to vessel and record volumes:

Reagents	Volume (ml)
<u>HF</u>	<u>10</u>
<u>HCl</u>	<u>2</u>
<u>H₃BO₃</u>	<u>50</u>

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 scavenge by centrifugation and decant. Wash the precipitate with ultrapure water, centrifuge, and decant. Repeat washing.
10. Dissolve 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 # 49643A)

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 1/16/95

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

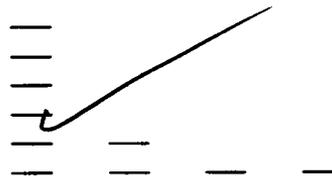
Thorium Column (Biorad 0.7 cm diameter column with 10 cm resin; prewash with 4-5 column vols 8M HNO₃)

Heat Th fraction to evaporate HCl
Add ~5 ml conc HNO₃ to dissolve residue
Add equal vol (~5 ml) DI water so soln 8M HNO₃
Load onto column in 8M HNO₃
Wash 3-4 column vols 8M HNO₃
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 HNO₃)

Evaoprate U fraction to near dryness
Pick up in about 2 ml conc HNO₃
Dilute with about 2 ml DI water to approx 8M HNO₃
Load onto column in 8M HNO₃
Wash 2 vols (8-10 ml) 8M HNO₃ --> 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 HNO₃ 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 1/18/95

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 disappears 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 1/17/95

MCB # 1 ACQ 01-17-95 AT 05:12:01 RT : 56847.7 LT : 56839.0
No detector description was entered
NOPI-118 U 1/18/95

ROI # 16-1 RANGE : 40 = 397.48keV to 162 = 425.34keV
AREA : Gross = 7413 Net = 7352 +/- 92
CENTROID : 130.42 = 418.12keV
SHAPE : Fwhm = 7.08keV Fwtm = 13.01keV

ID : SB-125 at 427.89keV
Corrected Rate = 0.129 +/- 0.00

ROI # 16-2 RANGE : 275 = 451.14keV to 411 = 482.19keV
AREA : Gross = 9984 Net = 9898 +/- 109
CENTROID : 382.82 = 475.76keV
SHAPE : Fwhm = 8.29keV Fwtm = 16.65keV

ID : SB-125 at 463.38keV
Corrected Rate = 0.174 +/- 0.00

ROI # 16-3 RANGE : 526 = 508.46keV to 645 = 535.63keV
AREA : Gross = 5043 Net = 4841 +/- 93
CENTROID : 622.77 = 530.55keV
SHAPE : Fwhm = 4.73keV Fwtm = 12.97keV

ID : No close library match

NP118Th.CHH

MCB # 1 ACQ 01-18-95 AT 10:31:52 RT : 25001.6 LT : 25000.0
No detector description was entered
NOPI-118 Th 1/19/95

ROI # 6-1 RANGE : 29 = 3.91keV to 82 = 4.05keV
AREA : Gross = 187 Net = 187 +/- 14
CENTROID : 63.78 = 4.00keV
SHAPE : Fwhm = 0.01keV Fwtm = 0.08keV

ID : No close library match

ROI # 6-2 RANGE : 175 = 4.29keV to 347 = 4.75keV
AREA : Gross = 16332 Net = 16332 +/- 128
CENTROID : 319.10 = 4.67keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.15keV

ID : No close library match

ROI # 6-3 RANGE : 480 = 5.10keV to 622 = 5.47keV
AREA : Gross = 4951 Net = 4951 +/- 70
CENTROID : 599.78 = 5.42keV
SHAPE : Fwhm = 0.04keV Fwtm = 0.16keV

ID : No close library match

ROI # 6-4 RANGE : 646 = 5.54keV to 760 = 5.84keV
AREA : Gross = 804 Net = 775 +/- 36
CENTROID : 693.90 = 5.66keV
SHAPE : Fwhm = 0.06keV Fwtm = 0.19keV

ID : No close library match